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Phase Investigation of Micro Crystals of ZTO

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Abstract: The synthesis and characterisation of ternary semiconducting oxide, zinc stannate or zinc tin oxide (ZTO) has been made with a view to understand the structural and morphological variations with temperature. The entire group of metal oxide semiconductors includes structures such as ABO_3 perovskites, A_2BO_4 spinels and $AB(OH)_6$ hydroxyl metal oxides, with immense application potentials in environmental, electronic as well as opto-electronic fields. ZTO also exists in the various phases as $ZnSnO_3$, Zn_2SnO_4 and $ZnSn(OH)_6$. In this work, a detailed investigation has been made on the phase variations and structural behaviour of ZTO ceramics with annealing temperature. The synthesis method adopted was one- step low temperature hydrothermal technique, with the use of CTAB as surfactant. The crystallite phases were investigated using X-ray Diffraction (XRD) and Differential Scanning Calorimetry (DSC) was employed in the thermal analysis of the as synthesised samples.

No appreciable change was observed in the diffraction pattern when the sample was annealed upto 200^o C. On further increasing the temperature, a sharp endothermic peak was observed in the DSC curve and the variations in XRD pattern showed a vivid change in crystallinity, indicating a transition to amorphous phase. The perfect cubic shape of the crystals was observed from Scanning Electron Microscopy (SEM) images at room temperature.

Keywords: Hydrothermal method, Zinc stannate, Differential Scanning Calorimetry.

Introduction and Experimental

With the active developments in the field of material science, there has been an urge to synthesise novel functional materials for specific applications. Zinc oxide being a wide band gap semiconductor, occupies a significant position among all metal oxides, since it can be used in a wide variety of applications such as photo catalysis, sunscreens, transparent conducting oxides etc [1,2]. Another important binary oxide is tin oxide, wide band gap n-type semiconductor with similar applications. The objective of this work is to investigate the physical and structural behaviour of a combination of these two - Zinc tin oxide or zinc stannate, commonly known as ZTO. This ternary oxide also finds applications in various fields such as transparent conducting oxides, photo catalysis, gas sensors for the detection of combustible gases etc [3,4]. It combines the properties of both ZnO and SnO_2. It occurs in two important forms such as $ZnSnO_3$ perovskite structure and Zn_2SnO_4 spinel structure. This work reports the synthesis of ZTO micro crystals via a one-step hydrothermal method, carried out at low temperature, and the structural and crystallographic variations with increase in temperature.

The synthesis of ZTO powder is carried out by low temperature hydrothermal reaction technique using cetyl trimethyl ammonium bromide (CTAB) as the surfactant.0.1M of Zinc acetate was dissolved in doubly distilled water to obtain a clear solution. To this, 40ml of tin chloride pentahydrate was added under continuous stirring, to obtain a milky white solution. Under continuous stirring, required amount of 1M KOH stock solution was added to limit the pH to 10. A precipitate is formed, which is then transferred to a Teflon lined stainless steel autoclave, and kept in furnace at 150^{o} C for 3 hours. It is then allowed to cool naturally to room temperature and the resulting solution is washed and centrifuged several times to obtain slurry. The slurry is dried at 100^{o} C to obtain white powder of Zinc Tin oxide microcrystals. The crystallinity of the sample is verified by X ray Diffraction (XRD) using Cu-K α of wavelength 1.54443 Å with Rigaku Miniflex 600 machine. The phase transition of the sample is monitored with the increase in temperature using the thermodynamic tool of Differential Scanning Calorimetry (DSC). The sample is heated in steps of 10 ^{o} C per minute using DSC 4000, Perkin Elmer. The surface morphology is analysed using Hioki Scanning Electron Microscope.

Results and Discussions

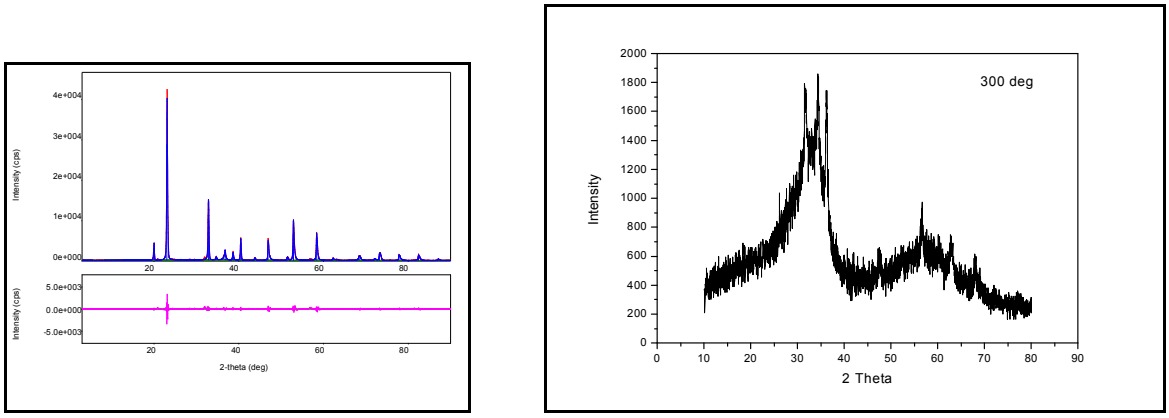

Fig. 1,2. XRD pattern of sample calcined at 150^{o}C and 300^{o}C

The room temperature powder XRD image of the sample in Fig 1 shows the high crystallinity of ZTO micro crystals calcined at 150^{o}C. Fig 1. corresponds to face-centered perovskite ZnSnO3, JCPDS data file 11-0274. Fig 2 shows the sample calcined at 300^{o} C in which the degree of crystallinity is lower.

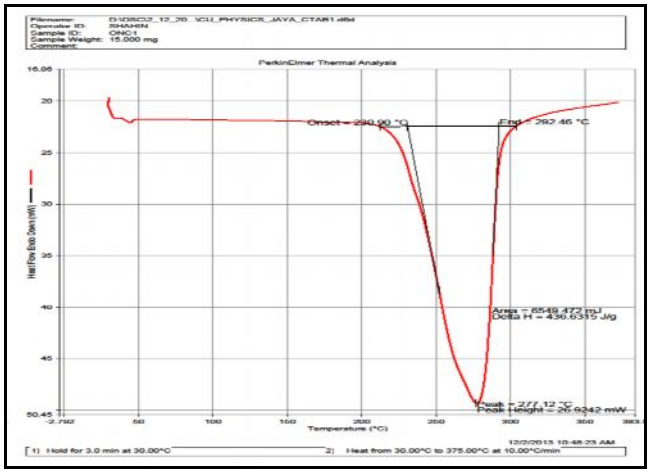

Fig.3 DSC spectrum of ZTO sample

The DSC curve Fig.3 shows a large endothermic peak at 277.12^{o} C, showing the absorption of energy by the sample. Due to this absorption, the crystallinity of the sample gets reduced, indicating a phase transition of the sample. The onset of transition is seen at a temperature of 230.9 ^{o} C and the enthalpy of heating (ΔH) is 436.63 J/g. This is clearly verified by the X ray Diffraction pattern of the sample, showing a vivid change in crystallinity of the sample above 230.9 ^{o}C.

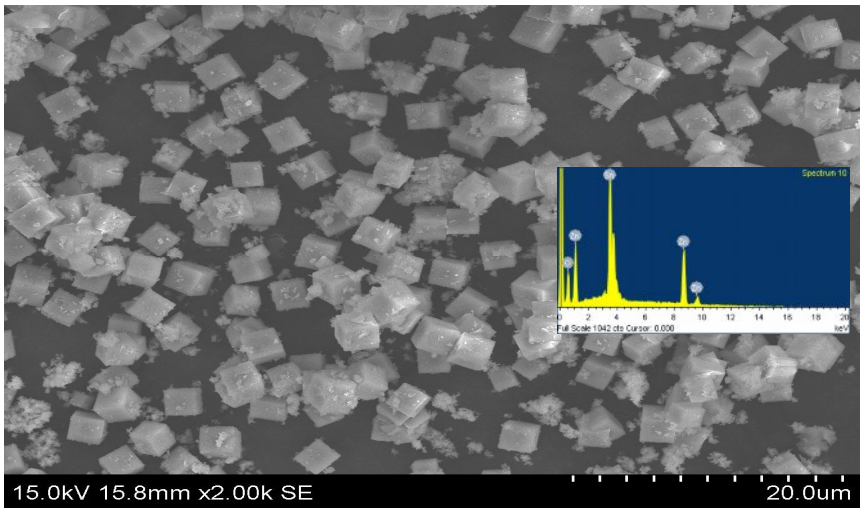

Fig.4 Scanning Electron Microscope images of the ZTO sample. EDS spectra is shown in the inset

The SEM images, Fig.4 show a uniform distribution of cubic shaped micro crystals, without any agglomeration. The use of CTAB as surfactant inhibits the formation of clusters by reducing the surface tension of the precursor solution, thereby reducing agglomeration. Also, hydrothermal synthesis of ceramics offers the synthesis of wide variety of shapes and forms of particles, which can be controlled with thermodynamic variables, such as reaction temperature, types and concentrations of the reactants, in addition to non-thermo dynamic variables, such as stirring speed and time [5]. The EDS spectrum of the sample shows the presence of tin, zinc and oxygen and hence confirms the chemical composition of the sample.

Conclusion

The synthesis of perovskite structured zinc stannate micro crystals were carried out using low temperature hydrothermal method and the phase transition with different calcinations temperatures are investigated. XRD patterns show a vivid change in crystallinity above a temperature of 230.9^{o}C and DSC curve confirms the transition from crystalline phase to amorphous. SEM images show perfectly cubic, non-agglomerated samples and EDS confirms the chemical composition. The synthesised sample can be proposed to be good candidates for transparent conductors and as photo catalysts.

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