Poster Presentation

MS122.P08

Structure and microstructure of primary particles in mesoporous SnO_2 beads

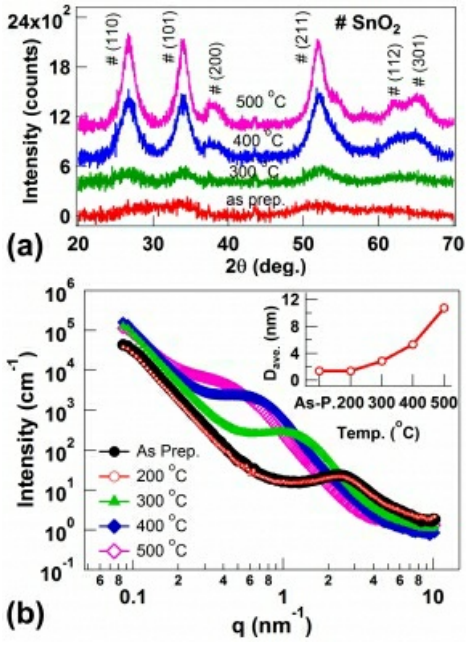
Suresh Koppoju[1], Easwaramoorthi Ramasamy[1]
[1]*International Advanced Research Centre For Powder Metallurgy & New Materials, Hyderabad, India*
E-mail: sureshkoppoju@arci.res.in

Sum-micron size mesoporous metal oxide beads such as TiO2 and SnO2 possess unique microstructure by tightly packed random network of primary crystalline nanoparticles (5 to 10 nm). Such microstructure leads to interconnected pore structure with high internal surface area. These mesoporous beads have already attracted widely owing to their potential applications in various energy conversion and storage devices. For example, sub-micron size SnO2 beads have recently achieved power conversion efficiency of more than 10% in dye-sensitized solar cells (DSSCs).

Hydrothermal (HT) technique is one of the prominent methods to synthesise mesoporous SnO2 metal oxide beads. The Size of the mesoporous beads as well as primary crystalline particles are determined by the temperature during HT process. Heat treatment of as-synthesized beads around 500oC in air leads to the removal of trapped organic compound which in turn creates mesoporous structure in beads. The formation of crystalline primary particles and their assembly is presently known mechanism. However, no systematic work has been carried out to investigate the microstructure of the primary particles at various stages during synthesis of mesoporous SnO2 beads. Usually, transmission electron microscopy is employed to investigate the microstructure at the length scale of a few nanometers. As the size of the beads is too thick for electron transmission, TEM could not be applied effectively to study the microstructure of the primary particles in these beads. Therefore, it is very important to understand size of the primary particles, which determines the effective surface area and mesoporous structure of SnO2 beads. Therefore, we have applied the small angle X-ray scattering (SAXS) technique to investigate the microstructure of the primary particles and x-ray diffraction (XRD) to study their crystallinity and crystal structure in mesoporous SnO2 beads at different stages of synthesis and post heat treatment.

XRD studies (Fig.1a) reveal that the as-prepared SnO2 beads by the HT method at 160oC exhibit amorphous nature. Further, SAXS investigation of the as-prepared micro-beads showed a strong scattering above q=1 nm-1 (Fig.1b) and the estimated size of the scatterers is 1.3+-0.4nm. Combining XRD and SAXS studies, these scatterers are found to be amorphous Sn-O nanoclusters. Upon heat treatment at 400oC for more than 1 hour, amorphous Sn-O nanoclusters crystallize and also grow to larger particles (inset to Fig. 1b). By employing SAXS, we have overcome the limitations of TEM to investigate the microstructure of primary particles in mesoporous SnO2 beads from nucleation to growth, owing to the high transmission of X-rays. Nucleation and growth mechanism of the primary SnO2 particles and also their microstructure and crystal structure as a function of HT temperature and post heat treatment will be discussed in the presentation.

Fig.1: (a) XRD and (b) SAXS profiles of the SnO2 beads as prepared at 160oC and heat treated at 200 - 500oC. Inset to Fig.1b shows average primary particle diameter with heat treatment temperature.



Keywords: SnO2, SAXS, XRD