Type of presentation: Oral

MS-9-O-2168 In-situ TEM study of Zn_2SiO_4 nanotube formation by Kirkendall effect

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Hollow Zn_2SiO_4 has evoked significant interest as a multifunctional material with applications in various domains ranging from cathode ray tubes, electrocatalysis, luminescent devices and selective metal ion absorption. Among various methods for fabrication of hollow structures, the classic one is Kirkendall process, exploiting differences in the diffusion rates of different species. In this study, we synthesize Zn_2SiO_4 by exploiting Kirkendall effect involving the reaction between ZnO nanorods and a chemically-synthesized SiO_2 shell. Using in-situ heating in the TEM, we have monitored the nucleation and coalescence of the Kirkendall voids leading to tubular structure in the SiO_2-coated ZnO nanorods. We compare the mechanism of formation of the nanotubes with results from in-situ heating in the TEM and samples heated in air as well as under reducing conditions. While heating the samples in air leads to microstructural evolution that is similar to the in-situ experiments, there is a significant difference in the samples heated under reducing conditions. Heating the ZnO@SiO_2 nanostructures in a reducing atmosphere (95% Ar + 5% H_2) leads to the formation of amorphous silica nanotubes owing to etching of ZnO in H_2 atmosphere. Thus, this understanding of the relevant diffusion processes for the diffusion couple at nanoscale presents a general conceptual platform to fabricate different multifunctional one-dimensional hollow nanostructures. The fabricated Zn_2SiO_4 nanotube show excellent toxic metal ions (titanium and uranium) adsorption property and also serve as good cathode materials for Li-ion batteries.

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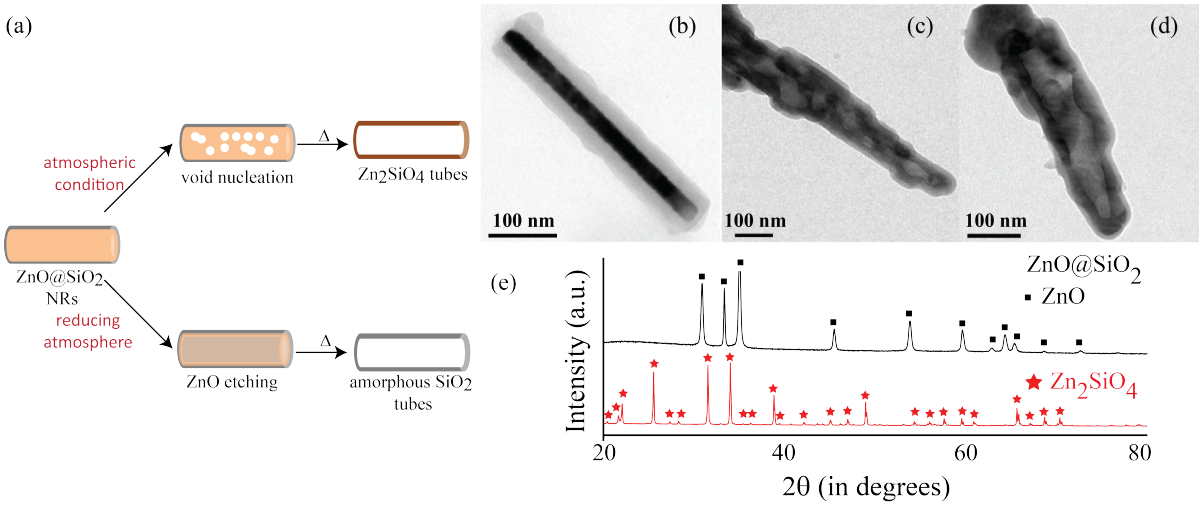

Fig. 1: Fig. 1. (a) schematic of microstructure evolution under different conditions; (b), (c) and (d) are TEM images showing nanotube formation from ZnO@SiO_2 on annealing in air; (e) Comparison of the XRD patterns of the ZnO@SiO_2 core@shell structure, as synthesized and after heating the same in air at 900°C, thus resulting into rhombohedral Zn_2SiO_4 phase

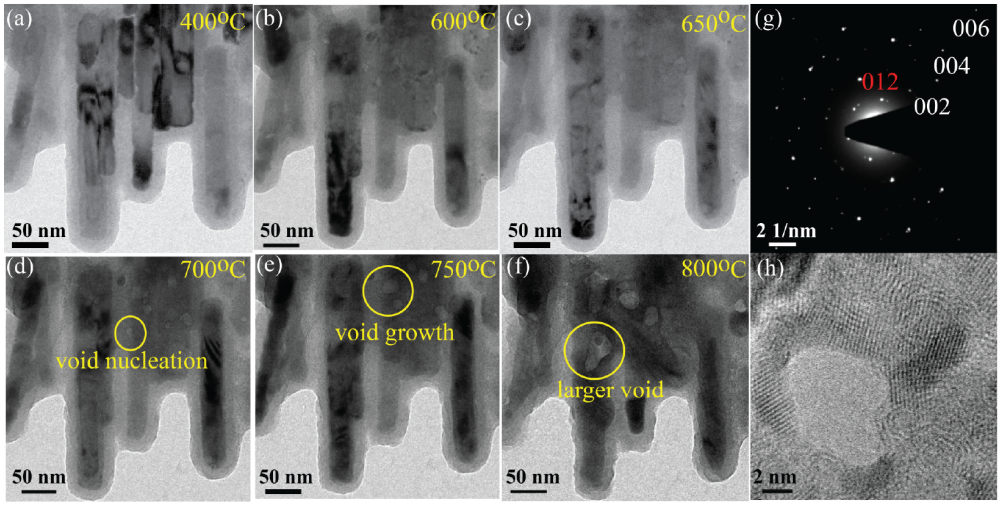

Fig. 2: Fig. 2. (a-f) are results of in-situ heating showing the hollow structure formation; (g) is SAED pattern of intermediate showing mixed phase of silicate (marked in red) and ZnO; (h) HRTEM showing Kirkendall void

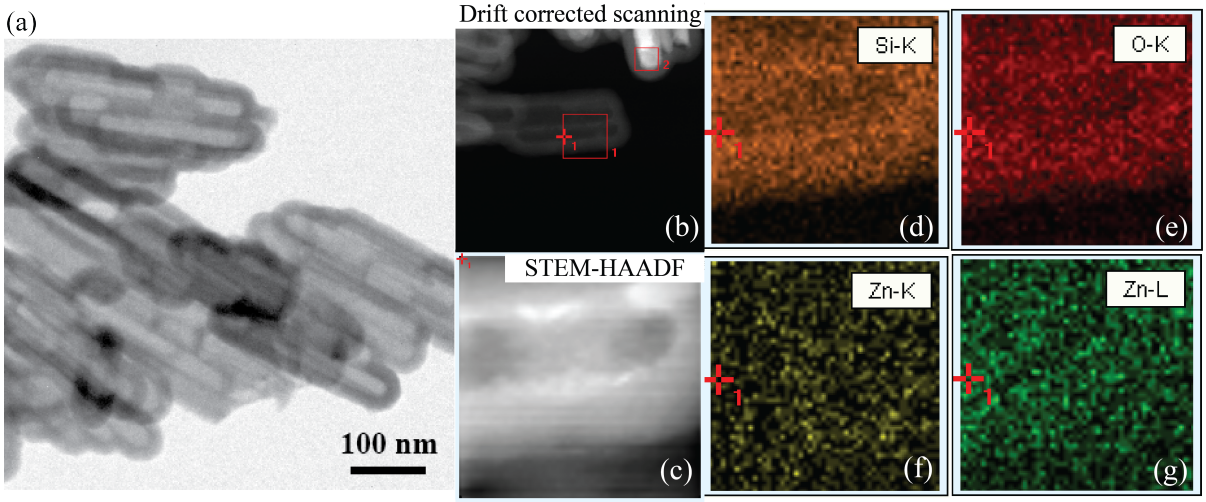

Fig. 3: Fig. 3. (a) BF TEM image showing SiO_2 nanotubes formed on heating ZnO@SiO_2 in reducing atmosphere; (d-g) are STEM elemental mapping results from the region (b-c) showing no presence of Zn, further confirming etching of ZnO in H_2 atmosphere