

Electronic Supplementary Information

## **Extension of the Benzyl Alcohol Route to Metal Sulfides: “Nonhydrolytic” Thio Sol-Gel Synthesis of ZnS and SnS<sub>2</sub>**

**Bettina Ludi, Inga Olliges-Stadler, Marta D. Rossell, and Markus Niederberger\***

### *Chemicals*

Zinc(II) acetate (99.99%, Aldrich), diethylzinc (purum, 1 M in hexane, Fluka) and tin(IV) chloride (99%, Aldrich) were used as precursors for ZnS and SnS<sub>2</sub>. Benzyl mercaptan (99%, Aldrich) was used as reaction medium. All chemicals were used without further purification.

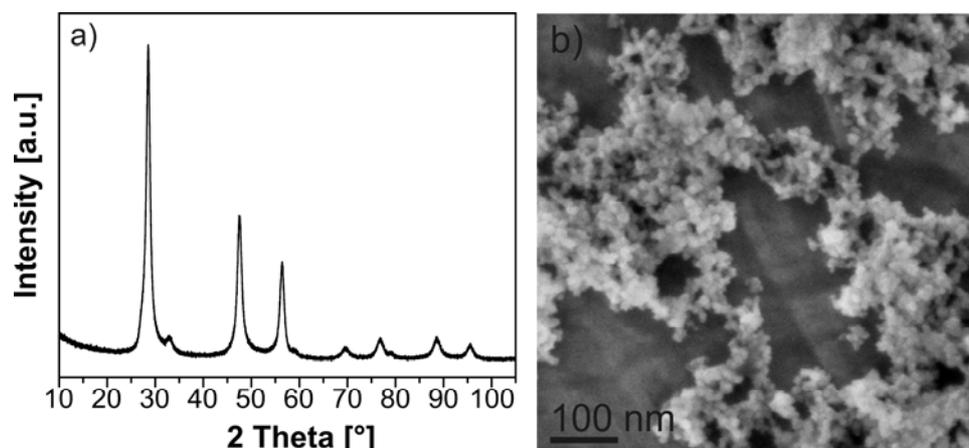
### *Synthesis*

In a typical synthesis 2 mmol of the metal sulfide precursor were reacted in 11.4 ml of benzyl mercaptan in a stainless steel autoclave equipped with a Teflon liner of a total volume of 50 ml. After 48 h of reaction time at 200°C the reaction mixture was cooled to room temperature naturally, centrifuged and the resulting precipitate was thoroughly washed with acetone and diethylether. The product was dried at room temperature and ground into a fine powder. SEM and TEM samples were prepared by directly dropping the ultrasonicated particle suspension on a standard SEM aluminum holder or on a carbon coated copper grid.

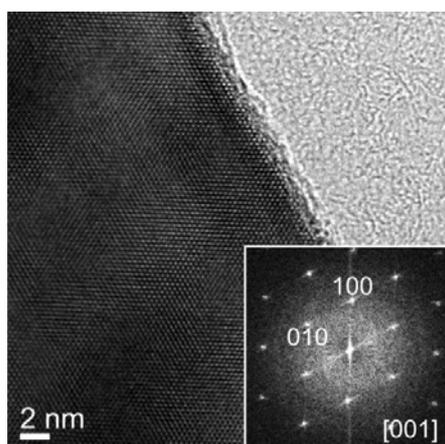
A slight variation of the procedure was necessary when diethylzinc was used as precursor. The diethylzinc solution was added slowly to benzyl mercaptan at room temperature under vigorous stirring and inert atmosphere in a round bottom flask. The resulting mixture was heated up to 140°C in an oil bath for 1 h until a suspension which could be stirred again was obtained. This suspension was then transferred to the autoclave and standard treatment followed.

### *Instruments and Characterization*

X-ray diffraction (XRD) patterns of the metal sulfide powders were measured in reflection mode (Cu K $\alpha$  irradiation) on an X'pert PRO PANalytical diffractometer. Scanning electron microscopy (SEM) images were performed on a Carl Zeiss Leo 1530. TEM, HRTEM, SAED as well as EDX investigations were performed on a JEOL 2200 TEM/STEM microscope operated at 200 kV.



**Figure ESI-1.** XRD pattern and SEM image of ZnS nanoparticles synthesized using diethylzinc as precursor.



**Figure ESI-2.** Representative HRTEM image of tin disulfide; inset: power spectrum.