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MnS spheres: Shape-controlled synthesis and its magnetic properties

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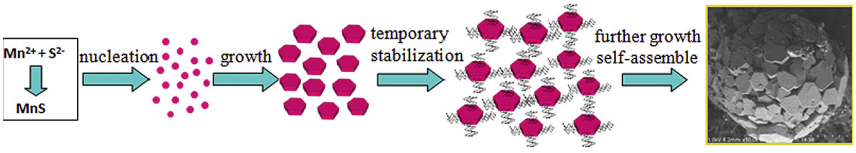
HIGHLIGHTS

- Sphere-like MnS hierarchical microstructures were synthesized and characterized.
- The size and morphology of MnS crystals can be turned by the concentration of L-Cystein molecules.
- The morphology of MnS hierarchitectures exerts a remarkable effect on their magnetic property.

GRAPHICAL ABSTRACT



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ABSTRACT

Sphere-like MnS hierarchical microstructures were successfully synthesized by a simple hydrothermal approach, which are composed of the size tunable and self-assembled nanoparticles. These hierarchical microspheres are γ-MnS phase, which is confirmed by X-ray diffraction (XRD) results, and the stoichiometry of MnS microspheres is checked by XPS measurement. Morphological studies performed by scanning electron microscopy (SEM) method show that the as-prepared γ-MnS samples are hierarchical microspheres. The size and morphology of composed nanoparticles can be turned by the concentration of L-Cystein molecules. Here, L-Cystein not only plays a role of sulfur source but also capping agent. Furthermore, a rational mechanism about the formation and evolution of the products is proposed. The present work shows that the origin of the observed difference of magnetic properties is due to the morphology difference of MnS crystals.

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1. Introduction

In the field of nanotechnology, many efforts have been placed on the self-assemble of nanoscale building blocks into well-defined three-dimension (3D) hierarchical nano/microstrutures, which

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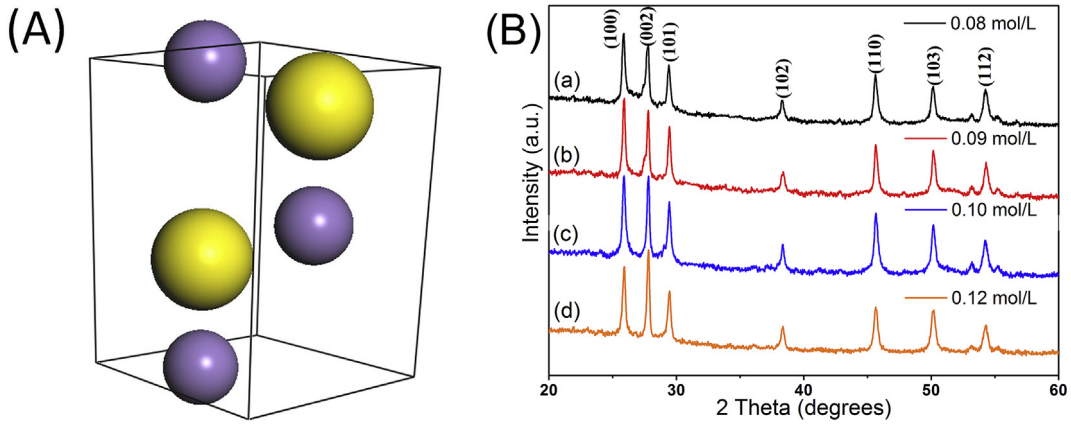

Fig. 1. (A) crystal structure of γ-MnS (Mn: purple ball; S: yellow ball). (B) XRD patterns of the MnS samples prepared at different concentrations of L-Cysetine: (a) 0.08 mol/L; (b) 0.09 mol/L; (c) 0.10 mol/L; (d) 0.12 mol/L. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

could be expected to exhibit some novel properties [1–12]. Manganese sulfide (MnS), among paramagnetic material, has been used in many fields owing to its unique electric [13], optical [14] and magnetic properties [15]. There are three phases for MnS crystals, including the stable α-MnS, and the two metastable phases β-MnS and γ-MnS. Comparing with α-MnS, β-MnS and γ-MnS may provide a great deal of opportunities for exploring their excellent properties. In this regard, many efforts have been devoted to the control of the crystalline phases of MnS in the past few years [16–19]. However, the controlled-synthesis metastable phases of MnS is still very difficult, since they are not thermally stable and the transformation easily occurs from metastable phases into the stable phase at the condition of high-temperature or high-pressure [20]. Moreover, since the physical and chemical properties of nanomaterials not only depend on the phase, but also on the size, shape and organization [21–25], tuning growth of MnS crystals with controlled morphology has been received much attention and urgently needed. So far, MnS micro/nanocrystals with various sizes and shapes have been successfully prepared, such as rods [26], cubes [27], stars [28], corals [29], flowers [30], bipods [20], boxes [31], dandelion [32], and so on. Among these morphologies, 3D hierarchical nano/microstrutures may offer a novel way to explore their new properties (especially, magnetic properties), comparing with their solid counterparts. For instance, MnS superstructures show a large coercive field (e.g., 1265 Oe for the 40 nm nanocube assemble), due to the small size and self-assembly properties of the samples [27]. Nevertheless, there are only a few reports on the synthesis of MnS superstructures self-assembled from nanoparticles [33–35]. It is still a big challenge to develop a facile and rapid method for preparing nanoparticle-assembled MnS superstructures with controlled morphology. Herein, we develop a facile hydrothermal method to synthesize nanoparticle-assembled MnS hierarchical microstructures with controlled morphology. Moreover, it is found that the morphology of MnS exerts a remarkable effect on their magnetic property.

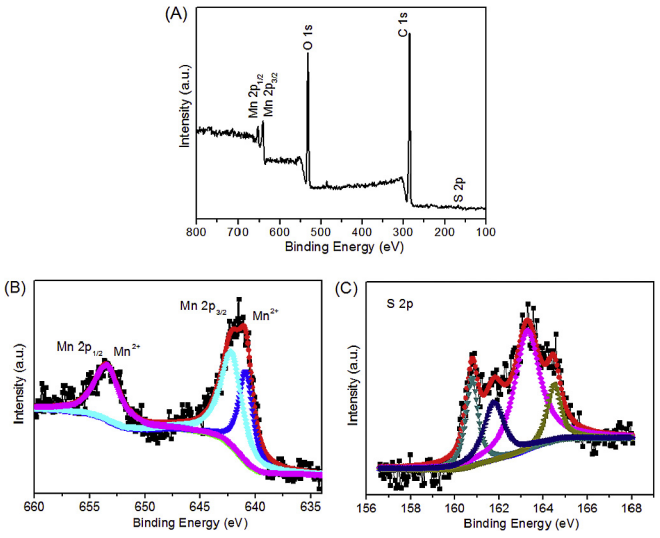

Fig. 2. XPS analysis of the sphere-like MnS hierarchitectures obtained at the high concentration of L-Cysetine (eg. 0.10 mol L^{-1}): (A) survey spectrum, (B) Mn 2p and (C) S 2p binding-energy spectrum.

2. Experiment

The MnS hierarchical microstructures were synthesized by a simple hydrothermal method. First, 1.0 mmol $Mn(CH_3COO)_2 \cdot 4H_2O$ and 0.2 mmol urea were dissolved in 15 mL water solution with different concentration of L-Cysetine (0.08, 0.09, 0.10 and 0.12 mol/L) under vigorous magnetic stirring at room temperature for 15 min. The above reaction solution was then transferred to a 30 mL Teflon-lined stainless steel autoclave and hydrothermally treated at 180 °C for 10 h. The autoclave was cooled to room temperature naturally. The as-prepared product was collected by centrifugation, washed with distilled water and ethanol for three times, respectively, and finally dried at 60 °C for 10 h to get the powder.

The powder X-ray diffraction (XRD) patterns of the as prepared samples were collected with a Rigaku D/max 2500V/PC X-ray diffractometer with Cu-K_α radiation (λ = 1.5406 Å), using a scanning rate of 0.017 °s^{-1}. The morphology of the synthesized MnS spheres was investigated by scanning electron microscope (SEM, Hitachi S-4800). X-ray photoelectron spectroscopy (XPS) was measured by a Sigma Probe (ThermoVG, U. K.) with a source of Al Kα radiation (1.486 eV). Magnetic studies were conducted on the MnS microspheres by using a SQUID magnetometer (MPMS, Quantum Design).

3. Results and discussion

The MnS hierarchical microstructures were synthesized as mentioned above and the powder X-ray diffraction (XRD) patterns of the as-prepared samples can be indexed to a hexagonal phase of γ-MnS (JCPDS No. 40-1289), as shown in Fig. 1. No peaks from other

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