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Fe_7S_8 nanorods and nanosheets

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Abstract

Fe_7S_8 nanorods and nanosheets were synthesized in large scale by a novel flux method. The starting materials are Fe, S powder and KI flux. XRD patterns demonstrated the as-prepared products were Fe_7S_8. The morphology could be controlled by the synthetic temperature easily. Nanorods and nanosheets were synthesized at 750 and 850 °C, respectively. The thickness of synthesized nanosheets was smaller than 100 nm. The nanorod diameter was about 200 nm, its length was about 2 μm, and the growth direction of nanorod was [0 1 2]. The growth mechanism for Fe_7S_8 nanorods and nanosheets are discussed in detail.

Keywords: Transition metal compounds; Fe_7S_8; Nanorods; Nanosheets; Flux method

1. Introduction

One-dimensional (1D) crystal structures, such as wire-like, ribbon-like, and tube-like structures are currently the focus of much attention due to their special properties [1]. These fascinating systems are expected to exhibit remarkable mechanical properties, and electrical, optical, and magnetic properties, which are quite different from those of their corresponding polycrystal powder materials. During the past decades, 1D nano-materials with unexpected properties have been prepared in various ways including template-directed synthesis [2], vapor-phase synthesis [3], solvothermal synthesis [4], vapor–liquid–solid method [5], solution–liquid–solid methods [6], etc., such as well-aligned ZnO nanowire arrays may serve as room-temperature ultraviolet nanolasers [7], and CdS single nanowire can be used in electrically driven lasers [8]. However, in most of these methods, template, catalyst, strict experimental conditions or long reaction time are usually necessary, which may bring difficultly to large-scale synthesis and future applications. Now a very important issue in the study and application of 1D nanomaterials is how to assemble individual atoms into 1D nanostructure in an effective and controllable way.

The pyrrhotite Fe_7S_8, with monoclinic, trigonal, and hexagonal structures, has been extensively studied due to its interesting ferromagnetic properties [9] and complex crystalline structure [10]. Traditionally metal chalcogenides are synthesized by the elemental reaction in evacuated tubes at elevated temperature [11–13], or by reaction of aqueous metal salt solutions with toxic and malodorous gas H_2E (E = S, Se, or Te) [14]. Iron chalcogenides are also obtained by the method of rapidly quenched ternary or the hydrothermal-reduction route [15,16]. However, there are few reports on the preparation of Fe_7S_8 with 1D nanostructures. Conventionally, the iron chalcogenide bulk single crystals have mainly been grown by vapor-phase methods, such as the chemical vapor transport technique using $AlBr_3$ and I_2 as transporting agents [17] and the closed-tube vapor transport technique [18]. The above-mentioned methods generally require special, complicated devices, or sophisticated techniques. Moreover, the temperature gradient has to be carefully controlled. Our group has developed a series of chemical synthetic method to prepare 1D nanostructure metal chalcogenides and

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oxides such as CdE (E = S, Se, Te), WS_2, MnO_2, ZnO, and SnO_2, etc. [19–23]. Recently we developed a flux method for the synthesis of 1D materials [24,25]. Here Fe_7S_8 nanorods and nanosheets were synthesized in large scale by the simple flux method. The morphology could be controlled by the synthetic temperature.

2. Experimental

Our growth apparatus was a furnace with a horizontal quartz tube, same as the apparatus used in preparing W nanowires [26]. The starting materials are Fe, S powder and KI flux. Metal Fe powders (0.2 g) were mixed with KI (1 g) in a mortar and ground for 5 min. Then the products were placed in a crucible and transferred to the center of the furnace. The S powders were placed in another crucible and transferred to the place upstream of the furnace. The furnace temperature was increased to 750–850 °C for 1 h and was kept at 750–850 °C for 2 h under a constant flow of argon at rates of 30 standard cubic centimeters per minute (sccm). After the furnace was cooled to room temperature, the products were washed several times with distilled water to remove KI flux and dried in an oven at 60 °C for 5 h.

The samples were characterized by a Bruker D8 Advance X-ray diffractometer (XRD) with Cu Kα radiation (λ = 1.5418 Å). The size and morphology of the as-prepared samples were obtained by a Hitachi Model H-800 transmission electron microscope (TEM) (with a tungsten filament at an accelerating voltage of 200 kV), and a LEO 1530 scanning electron microscope (SEM). EDAX was also measured by the SEM.

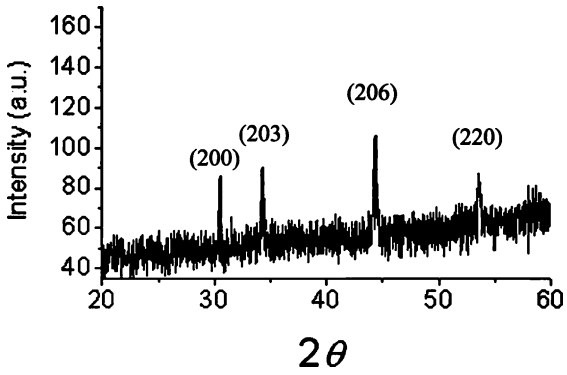

Fig. 1. XRD pattern of sample prepared at 750 °C.

3. Results and discussion

The products synthesized at 750 °C were characterized and analyzed first. XRD patterns (shown in Fig. 1) were used to determine the chemical composition and crystal structure of the synthesized samples. All of the reflections could be

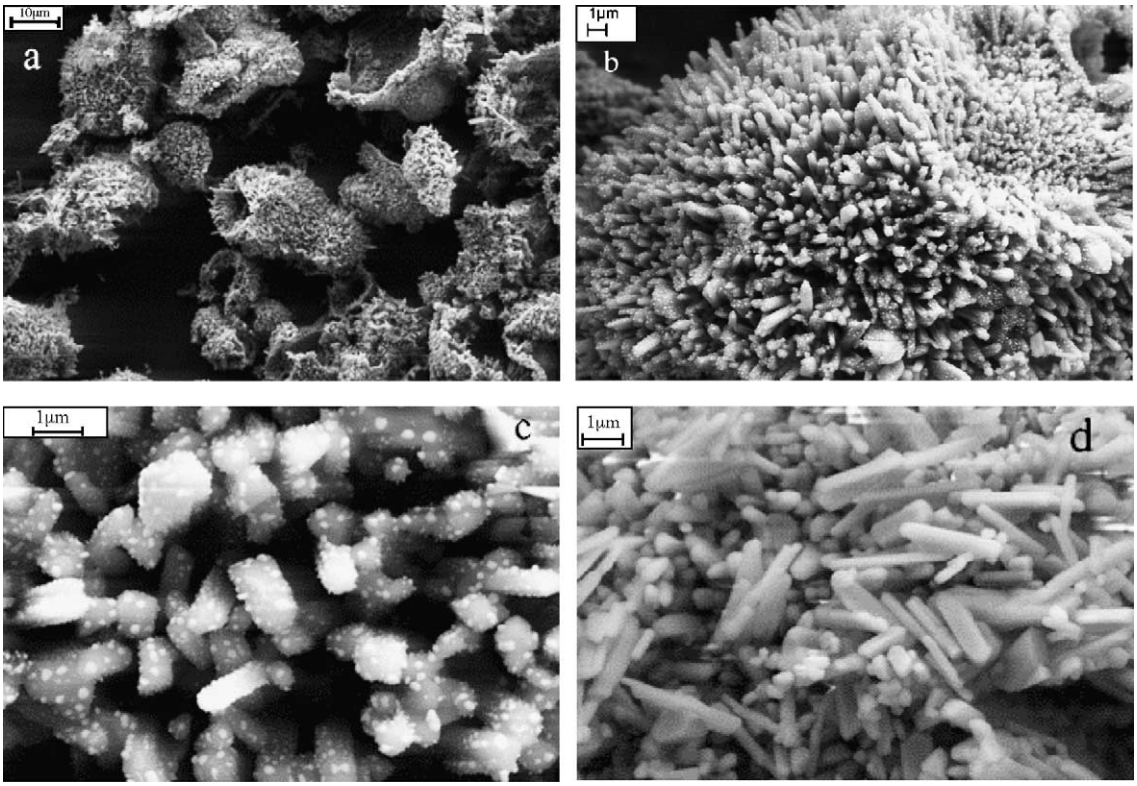

Fig. 2. Typical SEM images of samples prepared at 750 °C: (a) the low-magnification SEM image; (b) the high-magnification SEM image; (c) the front-end of the Fe_7S_8 nanorod; (d) the length of the Fe_7S_8 nanorods.