



Influence of EDTA²⁻ on the hydrothermal synthesis of CdTe nanocrystallites

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ABSTRACT

Transformation from Te nanorods to CdTe nanoparticles was achieved with the assistance of EDTA as a ligand under hydrothermal conditions. Experimental results showed that at the beginning of reaction Te nucleated and grew into nanorods. With the proceeding of reaction, CdTe nucleus began to emerge on the surface, especially on the tips of Te nanorods. Finally, nearly monodispersed hexagonal CdTe nanoparticles with diameters of about 200 nm were obtained. The effects of EDTA on the morphology and formation of CdTe nanoparticles were discussed in consideration of the strong ligand-effect of EDTA, which greatly decreased the concentration of Cd²⁺. Furthermore, the possible formation process of CdTe nanoparticles from Te nanorods was further proposed. The crystal structure and morphology of the products were investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM).

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1. Introduction

Recently, considerable attentions have been paid to II–VI semiconductor materials because of their technical importance as electrical and optoelectronic components [1–3], light-emitting devices [4–6], nonlinear optical materials [7,8] and biological labels [9–11]. As an important II–VI semiconductor material, CdTe nanocrystals have been investigated as possible candidates for integration into light-emitting devices and for luminescent doping of photonic colloidal crystals. After a typical organometallic approach was introduced in the early 1990s [12], this synthetic scheme of CdTe nanocrystals was considered to be an inspiring example and model system in this field. Although the organometallic synthesis has been ameliorated to eliminate its disadvantages such as poor reproducibility and the use of extremely toxic, expensive and explosive precursors, the selenium and tellurium precursors being used are closely related to organophosphorus compounds that are not very “green” at all [13]. In comparison, an aqueous synthesis of CdTe nanocrystals is simpler, greener and highly reproducible [14]. There are numerous reports regarding the synthesis of II–VI nanomaterials in aqueous phase by using inorganic cadmium salts as the cadmium precursor [15–17]. And we also researched on the preparation of CdTe nanocrystals under hydrothermal conditions [18]. CdTe nanocrystals prepared in aqueous solution are usually stable and have acceptable photoluminescence quantum yield at room temperature. However, their formation mechanism in aqueous solution especially in

hydrothermal condition has rarely been reported in the open literature. In this study, we investigated the transformation process of CdTe crystallite from Te nanorods in the presence of EDTA, a strong ligand with Cd²⁺.

2. Material and methods

All reagents used in this work, including cadmium nitrate (Cd(NO₃)₂·4H₂O), sodium tellurite (Na₂TeO₃), ethylenediaminetetraacetic acid disodium salt (Na₂EDTA), ammonia solution (NH₃·H₂O) and hydrazine hydrate (N₂H₄·H₂O), were AR grade reagents obtained from China National Medicines Corporation Ltd. and were used directly without any treatment. All solutions were prepared with Milli-Q water (18.2 MΩ) as a solvent.

In a typical synthesis, 0.77 g Cd(NO₃)₂·4H₂O was dissolved in 10 ml saturated Na₂EDTA solution and the solution turned out to be a little turbid. After stirring for 5 min, 1 ml NH₃·H₂O was added and the white precipitation disappeared. 2.5 ml Na₂TeO₃ (0.5 M) solution and 2.5 ml N₂H₄·H₂O were added into the solution in sequence under continuous stirring until the solution became colorless and transparent. The obtained solution was then transferred to a 25 ml Teflon-lined stainless steel autoclave. The autoclave was filled with deionized water up to 90% of the total volume, sealed, and heated in an oven at 150 °C for an appropriate duration. After reaction, the autoclave was allowed to cool to room temperature naturally. Black product was obtained and collected by filtration, washed repeatedly with distilled water and absolute ethanol, and then dried in vacuum drier at 60 °C for 4 h.

X-ray diffraction (XRD) analysis was carried out on a Bruker D8 Advance X-ray diffractometer. The topographies of Te nanorods

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and CdTe nanoparticles were examined through a HITACHI S-4800 field emission scanning electron microscope (SEM). Element mappings of CdTe along with Te were performed under HORIBA EMAX Energy EX-350 energy dispersive spectrometer attached to S-4800 SEM. The excitation and emission spectra were recorded on an Edinburgh PL S920 fluorescence spectrophotometer at room temperature.

3. Results and discussion

3.1. Crystal structure analysis

The XRD patterns of products subjected to different reacting durations are shown in Fig. 1. All the diffraction peaks of pattern *a* can be indexed to hexagonal Te, which is in good agreement with the standard values in the literature (JCPDS no. 86-2269). From the pattern, the diffractions of planes (111) (220) (311) are well observed (marked with ■). No other impurities are detected in the sample. In contrast, pattern *d* is indexed to phase-pure and zinc-blended CdTe with lattice constant of $a=6.482 \text{ \AA}$ in accord with JCPDS card no. 15-0770. The main diffraction planes such as (100) (101) (102) (110) are marked with ▲. However, the patterns of *b* and *c* have all the characteristic diffraction peaks of Te and CdTe, which confirm the coexistence of Te and CdTe.

The series of four patterns indicate that the initial stage was dominated by the nucleation and growth of Te crystallites. CdTe crystallites appeared gradually with the advance of reaction and their amount increased accordingly as evident from the intensities

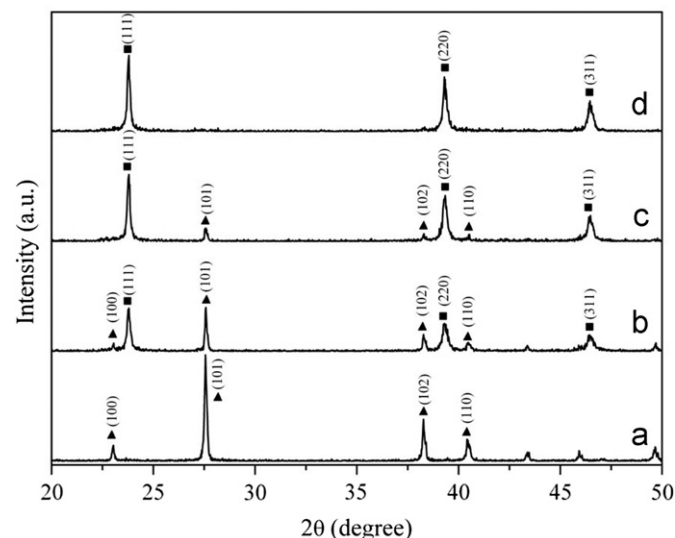


Fig. 1. XRD patterns of samples acquired at different reacting stages ((a)–(d) represent the different reaction durations 2 h, 6 h, 12 h and 36 h, respectively). The diffraction peaks of CdTe and Te are marked with ■ and ▲, respectively.

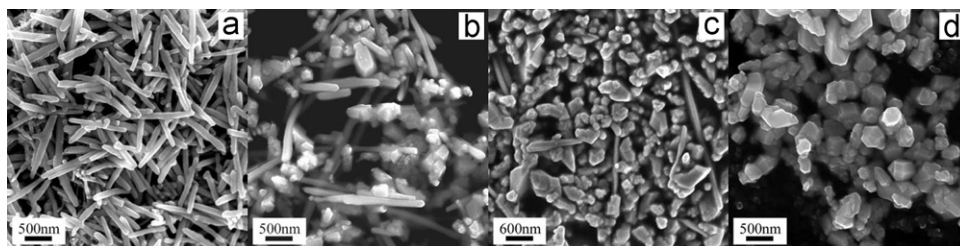


Fig. 2. SEM images of samples from Te nanorods to CdTe nanoparticles at different growth stages ((a) 2 h; (b) 6 h; (c) 12 h; and (d) 36 h).

of diffraction peaks. It is certain that the nucleation and growth of CdTe will consume some Te, which results in the decrease of Te content. When the reaction completed, only CdTe remained and Te was exhausted. Our previous experiments without the presence of EDTA showed that there was only CdTe even in very short reaction duration [18]. This suggested that EDTA played an important role in the process of reaction and it impetuses us to perform this study.

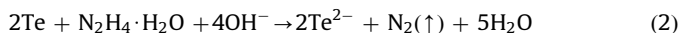
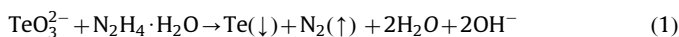
3.2. Morphology observation at different stages

Fig. 2 shows typical SEM images of these four samples for different reacting durations. Firstly, the products were only nanorods with diameters of about 100 nm with lengths up to 1–2 μm (an aspect ratio of approximately 10–20). Many researchers have reported their synthesis of one dimensional (1D) Te nanomaterials [19–21]. The reasons for the formation of 1D Te nanomaterials will be discussed in the following text. With the progress of reaction, there were nanoparticles formed with diameters of 200–500 nm and their amount increased gradually. In the meaning while the originally formed nanorods reduced in number and so both nanorods and nanoparticles coexisted in the system as shown in Fig. 2b and c. Finally, the nanorods disappeared completely and only nanoparticles left.

In order to obtain insightful information on the composition of both nanorods and nanoparticles, we performed element mapping with energy disperse spectrum (EDS). The element-mapping function attached on EDS analysis provides us a useful tool to distinguish one element from another. The position of every element exist in one sample was marked with bright points and brighter position represent more element content. A nanorod with nanoparticles on it was chosen as the object and the results were shown in Fig. 3. It can be seen that Te elements exist not only in the nanorod but also in the nanoparticles as revealed by the “Te map” picture, but the Cd mapping (“Cd map” picture) shows that Cd element only exist in the particles. Therefore, it can be concluded that the nanorods consist of Te while the nanoparticles are CdTe.

3.3. Growth mechanism analysis from Te to CdTe

The reaction to form CdTe nanocrystallites can be described as follows:



In these reactions, Na_2TeO_3 serves as an excellent water-soluble Te source and can be readily reduced to Te by hydrazine hydrate in hydrothermal conditions. The newly produced Te with high reactivity will nucleate and grow into nanorods (Eq. (1)).

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