Microwave Assisted Preparation and Characterization of Monodispersed PbSe Nanoparticles in Solar Cell

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Abstract: PbSe nanoparticles were used in solar cells, and the microwave method was applied to prepare monodispersed nanocrystalline PbSe in the solvent of ethylene glycol. The nanocrystals were analyzed by X-ray diffraction (XRD), transmission electron microscopy (TEM), differential scanning calorimetry (DSC), atomic force microscope (AFM), scanning electron microscope (SEM), and energy-dispersed spectroscopy (EDS). It is found that ethylene glycol both as reducing agent and dispersant plays a key role in the formation of PbSe nanocrystals.

Key word: nanoparticle; microwave; PbSe; semiconducting material
CLC number: O614.43^{+}3 **Document code:** A **Article ID:** 1002-185X(2010)S2-103-04

Recently, the semiconductor nanocrystals have attracted considerable attention in the field of solar cell, because they have no harming effects on the environment. Some organic materials have some advantages, such as low cost, large processing, which has been researched. However, the solar energy conversion efficiencies of devices including organic materials are very low due to their low charge carrier mobility and short diffusion length. Some inorganic materials were introduced for the purpose of improving their efficiencies[1]. Nozik firstly put forwards that semiconductor quantum dots have the potential to increase the maximum attainable thermodynamic conversion efficiency of the solar photo conversion up to about 66% by utilizing hot photogenerated carriers to produce higher photovoltages or higher photocurrents[2]. Quantum yields of 300% for exciton formation in PbSe quantum dots at photo energies 3.8 times of the HOMO-LUMO transition energy[3] proposed. Several methods have been known for the preparation of PbSe. In these synthesis ways, the solvothermal synthesis including much milder conditions and softer chemistry can be conducted at a relatively lower temperature. For exploring the solvothermal methods[4] as a route for the prepa- ration of metal chalcogenides, it can be found that different morphology exists in PbSe, but it needs long time, even to 72 h.

In this work, we choose a microwave method, by which shorter time is needed to complete the experiment. In most cases, 30 min to 3 h are necessary. In our experiment, ethylene glycol was chosen as the solvent. The glycol also worked as reducing agent for the preparation of submicrometer particles of the transition metals. On the other hand, reactions were conducted using microwave heating, because metallic particle were produced as intermediate in the polyol reaction.[5] However, there are many microwave phenomena, such as nonthermal effects and the superheating effect[6], which still need further investigation. In this paper, we report the synthesis of PbSe nanoparticles by microwave irradiation.

1 Experimental

All the reagents used in the experiment were of the analytical grade. $Pb(Ac)_2 \cdot 3H_2O$ was purchased from Tianjin Tianda Chemical Experiment Factory (Tianjin), Se powders from Xingang, Guangzhou (China), and ethylene glycol from Tianjin (China).

The XRD patterns were recorded on an X-ray diffraction (D8 ADVANCE, Germany) using Cu Kα λ= 0.154 18 nm radiation. Energy dispersion spectrum (EDS) measurements were conducted using an EMXA

Received date: 2010-04-22
Foundation item: Zhejiang Province Key Scientific and Technology Project (2008C01005-1); Shenzhen Science and Technology Plan Project (08df-03)
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(HoRiBa) instrument. The SEM was performed on S-3400N (II) instrument. The TEM images were obtained with a JEOL-JEM-1230 (Japan) instrument, working at 200 kV accelerating voltage. Differential scanning calorimetry (DSC) was conducted by a instrument, Netszch STA 409PC (Germany), using nitrogen as a purging gas while the scanning rate was 10 °C/min. AFM images of the nanocrystals were recorded with a CSPM5000 instru- ment in the tapping mode with a silica probe (Budget- Sensors Tapping300Al). The scan dimension and the roughness analysis were performed on 5 μm×5 μm.

In the experiment, lead acetate was added into a 150 mL round-flask. Then 100ml ethylene glycol was poured into this flask, and the solution was put into the microwave oven for approximately 2 min. Stoichiometric quantities of Se powder was then added. The reactions were conducted in a microwave oven for 2 h, and then cooled naturally to room temperature. The product was washed with distilled water and absolute ethanol in sequence for several times and then filtered. Finally, the dark product was dried in vacuum at 70 °C for 12 h.

2 Results and Discussions

2.1 XRD studies

The XRD pattern of the as-prepared PbSe nanocrystals is shown in Fig.1. The pattern matches well with the literature (JCPDS card No. 6-0354). The strong and sharp peaks reveal good crystallinity and relatively large particle size. No obvious impurity peaks were detected for PbO and Se, indicating that the product was pure enough. The average crystalline size was determined from Debye-Scherer equation.

2.2 DSC measurements

DSC was carried out using purging gas of argon at a scanning rate of 10 °C/min. There is one obvious peak at 225.1 °C corresponding to the eutectic reaction PbSe+Se →PbSe+liquid.[7] The peak disappeared when the sample was cooled and reheated again because of the small amount of Se and its evaporation. XRD measurements did not detect the unreacted Se due to its relative low sensitivity.

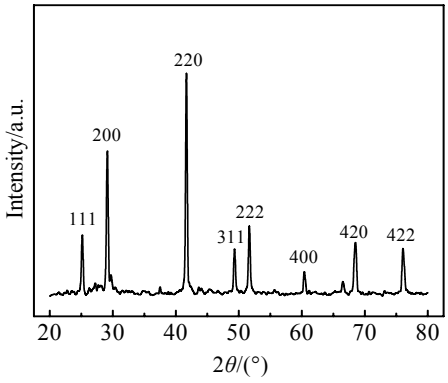

Fig.1　XRD pattern of PbSe nanocrystals

2.3 SEM measurements

SEM image shows that the products are uniform nanorods with regular morphology (Fig.3). The atomic ratio of Se:Pb was about 1:1.

2.4 EDS measurements

Fig.4 depicts the EDS spectrum of the product. Two major elements were detected as Pb and Se. The quantitative EDS analysis reveals that the atomic ratio of Se:Pb is 48:51, close to the nominal composition.

2.5 TEM measurements

Fig.5 shows the TEM images of the PbSe powder.

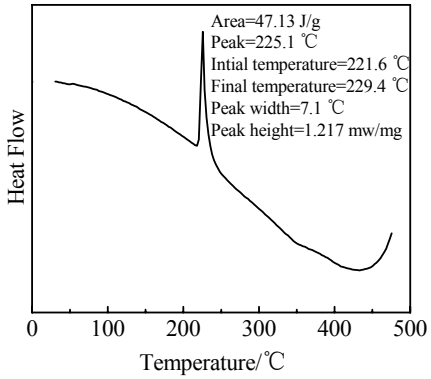

Fig.2　DSC curve of as-prepared PbSe nanocrystals

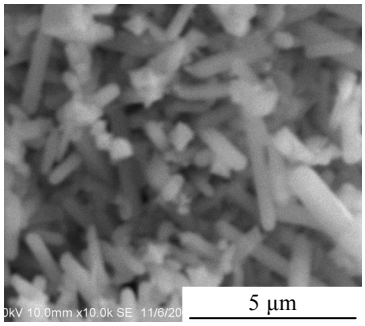

Fig.3　SEM image of the PbSe products with reaction time of 2 h

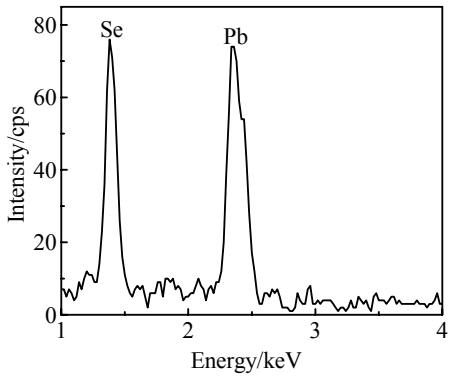

Fig.4　EDS spectrum of the particle

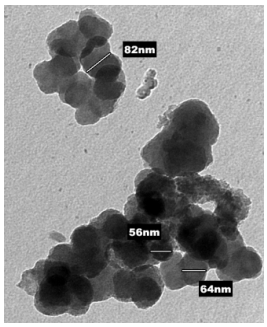

Fig.5　TEM image of PbSe powder

The shape of the crystals is irregular and they are aggregated, but most of them are close to cubic, with diameter in the range of 50-100 nm, consistent with the XRD results.

2.6　AFM Measurements

The surface morphology of the particles was characterized by AFM. Fig. 6a displays the morphology from size face. The profile can be also seen in Fig.6b. Fig.6c has parameters as following: surface distance is 70.7 nm, horizontal distance is 68.6 nm, vertical distance is 0.371 nm, angle is –0.31°, roughness is 1.82 nm, height 1 is 8.1 nm, height 2 is 8.47 nm, size is 5000 nm ×5000 nm, and image height is 17.90 nm. It indicates that the size of the crystals in some parts was about from 6 nm to 20 nm. This result is probably attributed to the intense microwave radiation. Nevertheless, some phenomena, such as some nonthermal and superheating effect, are still not fully understood. Further research in this area is suggested in the future.

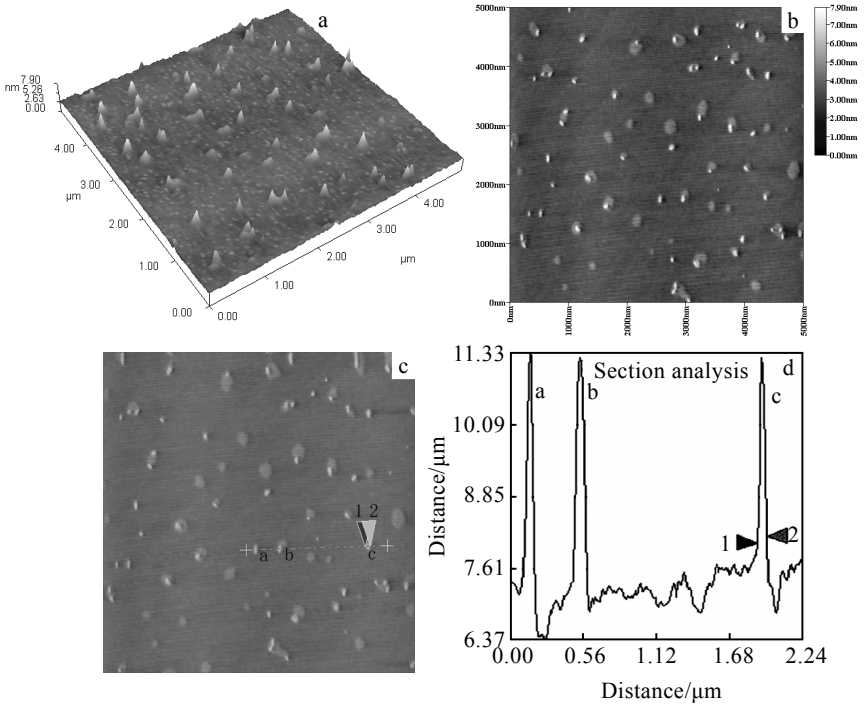

Fig.6　AFM morphology (a), 5000 nm×5000 nm profile (b), and surface analysis of some special sections (c, d) of the product

3　Conclusions

In summary, PbSe nanoparticles are prepared by the microwave method. The advantage of this process is that it is simple and efficient in producing nanoparticles. Some preparation conditions are proposed for this synthesis procedure. The details in mechanism of forming the particles still need further investigation.

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太阳能电池中单分散性 PbSe 量子点的微波法合成和表征

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摘　要：量子点 PbSe 被提出能应用于太阳能电池中，采用微波方法，用乙二醇作为溶剂来合成单分散性的纳米晶体 PbSe，并用 X 射线衍射、红外光谱、透射电镜、同步热分析仪、原子力显微镜、扫描电镜，X 射线能谱仪来表征。实验证明，在 PbSe 的合成过程中，乙二醇是良好的还原剂和分散剂。

关键词：量子点；微波；PbSe；半导体材料

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