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The Patented Project Design for Preparing $SnSe_2$ Nanosheets

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Abstract. The patent design provides one method for preparing photoelectricity thin film used in solar cell. It belongs to the field of photoelectric film preparation technology. The $SnSe_2$ nanosheet was prepared from the solid powders of $SnCl_22H_2O$ and SeO_2. The hydrazine hydrate, NaOH and ammonia are used as reducing agents and assistant agent respectively in the patent. It includes the steps of reacting, heating, washing, drying to prepared Tin diselenide. The obtained $SnSe_2$ with high purity has good dispersivity, and regular and uniform shape. The method has a wide application prospect because of safety process and the pollution-free and manageability aided solution.

Introduction

Due to good photoelectric properties, semiconductor materials of IV-VI group tin selenium (SnSe, $SnSe_2$) are widely used in the optoelectronic fields such as optical recording, solar battery, laser and so on [1,2]]

$SnSe_2$ is an n-type semiconductor with band gap of about 0.9 eV and hexagonal close-packed (HCP) structure and often described as sandwich structure of Se-Sn-Se layers combined with Se and Sn atoms by van der Waals force [3]. The band structure and elastic constants of $SnSe_2$ were calculated by density-functional theory (DFT) and it is identified as indirect band gap [4].

Existing methods of preparation of Tin and selenium compounds mainly consist of solvothermal method [5], direct vapour transport (DVT) technique [6], electron beam irradiation method [7] and chemical vapor deposition method [3]. In addition, D. Passeri deposited the ultrathin SnSe film on fused quartz by thermal evaporation technology [8]. The tin selenium film of nanoscale thickness was prepared by electrodeposition [9].

For lower cost material, tin selenium is a very promising photoelectric material. The research mainly concentrated on tin selenium film. It is necessary to explore the low cost preparation method of tin selenium, because the existing manufacturing process used now is complex and high cost. The unique physical properties such as light, heat, electricity, magnetism, nanometer semiconductor materials give the foundation to prepare semiconductor devices with superior performance [10,11]. At present the methods of preparing nanometer materials are mainly liquid methods such as solvent thermal method [12], chemical bath deposition [13], sol-gel [14] etc. but it is important to find a simple process to prepare nanometer material which has good dispersivity, in regular shape and uniform size.

Among existing methods there are some disadvantages in preparing nanomaterials. Some method gains nanometer materials which are easy to aggregate and with poor dispersity, the morphology and dimension homogeneity are not easy to control. The shape of Tin selenide flakes prepared by some method was not regular and impure in phase and composition. Different shapes of nanomaterials affect the specific surface area, it also indicates that the growth orientations for the grains of nanomaterials with different shapes may also be different, therefore it may affect the properties of nanomaterials. Due to nanometer materials with poor purity, since the mixed impurity phase is another matter, its performance will differ from the single phase materials, there are large differences in optical properties and electrical conductivity. These physical properties of semiconductor materials are very important. The existing technology closest to this work is published in Journal of Qingdao University of Science & Technology, twenty-seventh volumes fifth, 2006 October [5]. This article investigated preparation and structure characterization of $SnSe_2$ nanoflakes and given a method preparing $SnSe_2$ nanoflakes. Although the product is similar, but the there are obvious differences in the raw material and technical process. The existing technology adopts raw materials of $SnCl_2 \cdot 2H_2O$ and $NaSeO_3 \cdot 5H_2O$, the raw materials adopted in this invention are $SnCl_2 \cdot 2H_2O$ and SeO_2, while different raw materials may need different technical process to prepare the same products.

The $SnSe_2$ hexagonal nanoflakes were synthesized is single-phase powder by hydrothermal in patent. The $SnSe_2$ powder has uniform and homogenous hexagonal nanoflakes with side length of about 600 ~ 700 nm and thickness about 30 ~ 40 nm. It will be used in many fields by using of the shape and size of hexagonal nanoflakes. The $SnSe_2$ powder will have broad application in electrode material of lithium ion batteries, solar battery materials, sensors, laser device.

Patent Claims

Patent claim 1: The invention gives a preparing method of kind of high purity of solar cells with $SnSe_2$ photoelectric film nanosheets, comprising the following steps:

(1) Firstly, put 0.8~1.2 $SnCl_2 \cdot 2H_2O$, 0.787 ~ 1.18 SeO_2, 0.200 ~ 0.450 NaOH, this three kinds of solid powders mixed evenly, then add 12 ~16 copies of the deionized water mixing;

(2) Then the mixture solution is poured into a high-pressure reaction kettle, add 0.95 ~ 1.9 copies and 0.80 ~ 1.6 copies of ammonia;

(3) The high-pressure reaction kettle is heated to 160 ~ 200 ºC, holding time is 16 ~ 24 hours, and then cooled to room temperature and take it out;

(4) Filtrate the reacted solution through water millipore film, successively with deionized water and ethanol, repeated washing, filtering, removing impurities, washing to neutral;

(5) Tin selenide powder was obtained after washing and drying.

Patent claim 2: According to the first patent claim, the mixture powder was uniformly mixed in deionized water and dissolved more fully using ultrasonic vibration.

Patent claim 3: According to the first patent claim, the purity of the reducing agent is 85% or 80%.

Patent claim 4: According to the first patent claim, the ammonia concentration of auxiliary agents: 15%, 17% or 20%.

Patent claim 5: According to the first patent claim, Teflon was used as lining in the high pressure reactor.

Patent claim 6: According to the first patent claim, the products obtained after washing was drying at 50 ~ 80 ºC.

Patent claim 7: According to the first patent claim, drying was carried out in vacuum oven.

In application of invention the one or more detail project should be given. The design of projects should have differences in details as follows.

Project 1

Firstly mix three kinds of solid powders 1.0 $SnCl_2·2H_2O$, 0.984 SeO_2 and 0.210 NaOH evenly, and then add 12 copies of deionized water mixed evenly by ultrasonic vibration. That mixture solution was poured into high pressure reactor with Teflon line, then add 0.95 copies of the 85% hydrazine and 1.2 copies of 17% ammonia water. The high pressure reactor is heated to 180 ºC and held for 20 hours, and then cooled to room temperature and take it out. The reacted solution was filtrated through water millipore filtration film and successively washed with deionized water and ethanol, repeated washing, filtering, removing impurities, washing to neutral. The obtained product was dried in vacuum oven at 70 ºC, the $SnSe_2$ powder was gotten after washing and drying. After testing, the purity of $SnSe_2$ powder was 100%.

Project 2

Similarly, mix three kinds of solid powders 0.8 $SnCl_2·2H_2O$, 0.787 SeO_2, 0.280 NaOH evenly, adding with 14 copies of deionized water mixed evenly by ultrasonic vibration. That mixture solution was poured into high pressure reactor lined with Teflon, then add 1.9 copies of the 80% hydrazine and 0.8 copies of the 20% ammonia water. The high pressure reactor is heated to 160 ºC, held for 24 hours, and then cooled to room temperature and take it out. The reacted solution was filtrated through the water millipore filtration, successively washed with deionized water and ethanol, repeated washing, filtering, removing impurities, washing up to neutral. The product power obtained was dried in vacuum oven at 50 ºC after washing and drying. After testing, the tin selenide powder was 99.5% in purity.

Summary

In this work, based on experimental data, the invention projects were designed. The invention adopts raw materials of solid powders of $SnCl_2·2H_2O$, $2H_2O$, SeO_2, NaOH and reacting agents of hydrazine through reacting, heating, washing, drying and other steps to prepare Tin diselenide nanosheets. The product tin selenide nanoparticles are single-phase powder with hexgonal sheets which are uniform and regular in shape with side length about 600~700nm and thickness about 30~40nm. Compared with similar technology, the tin selenide nanosheets in this invention have high purity, good dispersion and uniform regular shape. Moreover, the technological process was safe, the aided solutions were easy treated, non-pollution. Therefore this method in the invention has very broad application prospects.

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