Synthesis and characterization of $AgInS_2$ nanoparticles by microwave assisted chemical precipitation

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Abstract: In this work, a novel method of microwave assisted chemical precipitation procedure was used to synthesize $AgInS_2$ nanoparticles. Ag, In, and S precursors with a stoichiometric ratio of 1:1:2 were dissolved in ethylene glycol as solvent and complexing agent. The resultant precipitate was placed under microwave irradiation for 5 min to obtain product. X-ray diffraction pattern (XRD) and scanning electron microscopy (SEM) were employed to characterize the final product.

Keywords: Microwave irradiation, Nanoparticles, Characterization, Ethylene glycol.

Introduction

Ternary I-III-VI_2 semiconductors can be the promising technological compounds in the fields of light-emitting diodes, lasers, solar cells and photocatalysis. These materials, such as $CuInS_2$ (CIS) and $AgInS_2$ (AIS), usually have the direct-band-gap with high extinction coefficients in the visible to near-infrared region [1]. Currently, the Ag–In–S system has been known as an interesting candidate owing to the particular and unique semiconductive properties. $AgInS_2$ is unique compound among the I-III-VI_2 semiconductors. The crystalline phases of this material are tetragonal (chalcopyrite type) and orthorhombic phase. The orthorhombic form is stable above 620˚C and the chalcopyrite form is stable at below 620˚C [2, 3]. Tetragonal and orthorhombic phases have two direct band gaps (1.86 and 2.02 eV) and (1.96and 2.04 eV), respectively [4-7]. $AgInS_2$ shows n-type electrical conductivity, which can be doped with Sb[8]and Sn[9] atoms and changes to p-type conductivity.

In recent years, the various methods are used to fabricate $AgInS_2$ such as spray pyrolysis, hot-press, hydrothermal and solvothermal treatment [10, 11]. Thermolysis processes require to the high temperatures and long times in a non-uniform thermal condition, leading to non-homogeneous nucleation and particle growth [11]. As an outstanding thermolysis way, microwave irradiation can be used to synthesize different nanomaterials in nanoscale. Decreasing the reaction time, improving the purity of product and high regeneracy are considerable cases to control the reaction in this method [12]. Hence, we report a microwave assisted chemical precipitation method to prepare $AgInS_2$ nanoparticles.

Experimental section

All of the chemicals used in this work were purchased from Merck Co. and used without further purification. Silver nitrate, indium chloride hydrate, and thioacetamide were employed as the starting materials to synthesize $AgInS_2$. Ethylene glycol and SDS were used as solvent and surfactant, respectively. In this study, the stoichiometric weights of reactants were employed so that the atomic ratio of Ag:In:S (1:1:2) was maintained. The experimental procedure was arranged in brief: Firstly, Silver and indium precursors were mixed with each other. An appropriate amount of SDS (In:SDS atomic/molecular ratio of 1:5) was used to provide high dilution and also, prevent further agglomeration. Thioacetamide as the sulfur source was added to above mixture to synthesize $AgInS_2$ compound. Secondly, resulting suspension was put into a suitable container and placed in a domestic microwave device with the power of 900 W for 5 min. The reaction proceeding was controlled continually. Finally, the obtained precipitation was filtered, washed several times with ethanol and distilled water and dried at 70 °C for 4 h in a vacuum oven. The product was characterized by using XRD and SEM.

Results and Discussion

Figure 1 shows XRD pattern of synthesized product with orthorhombic crystalline phase. This pattern is in a close agreement with JCPDS Card No. 025-1328. Obviously, the major peaks at 2θ values of 24.99°, 25.42°, 26.58°, 28.38°, 28.75°, 36.80°, 43.69°, 44.53°, 48.05°, 48.26°, 51.91°, 52.23°, 52.63° and 53.14° matching with the 120, 200, 002, 121, 201, 122, 040, 320, 203, 400, 322 and 331 planes of orthorhombic $AgInS_2$phase belong to prepared sample. This pattern reveals a good crystalinity of $AgInS_2$ with a minor impurity of Ag_2S phase, which can be a result of insufficient washing during the product processing.

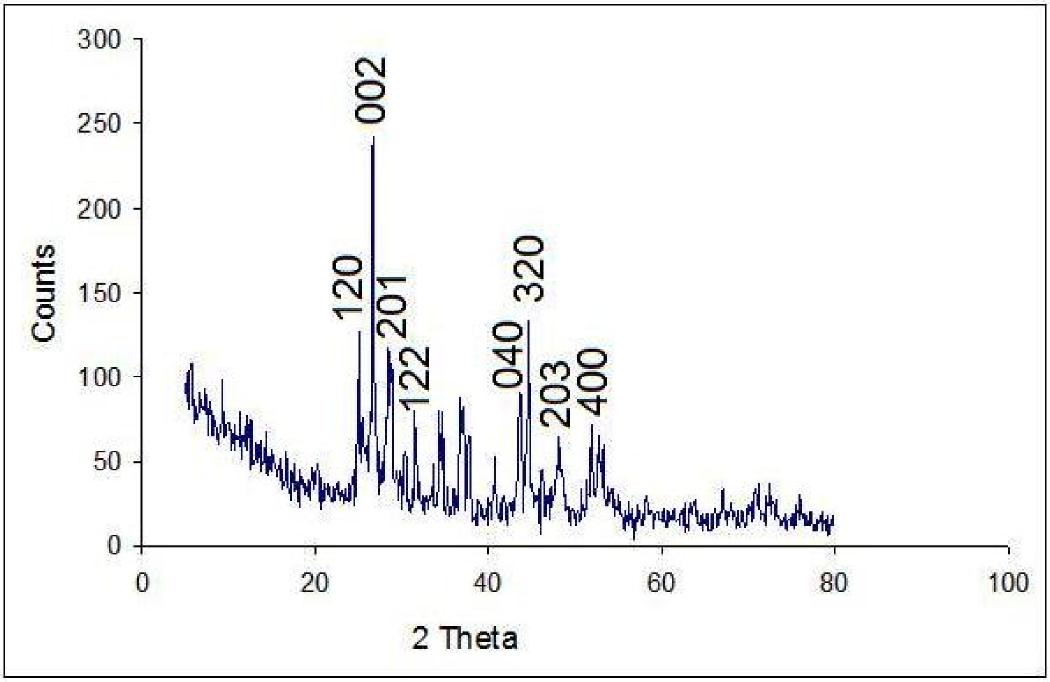

Fig. 1. XRD pattern of obtained $AgInS_2$ nanoparticles.

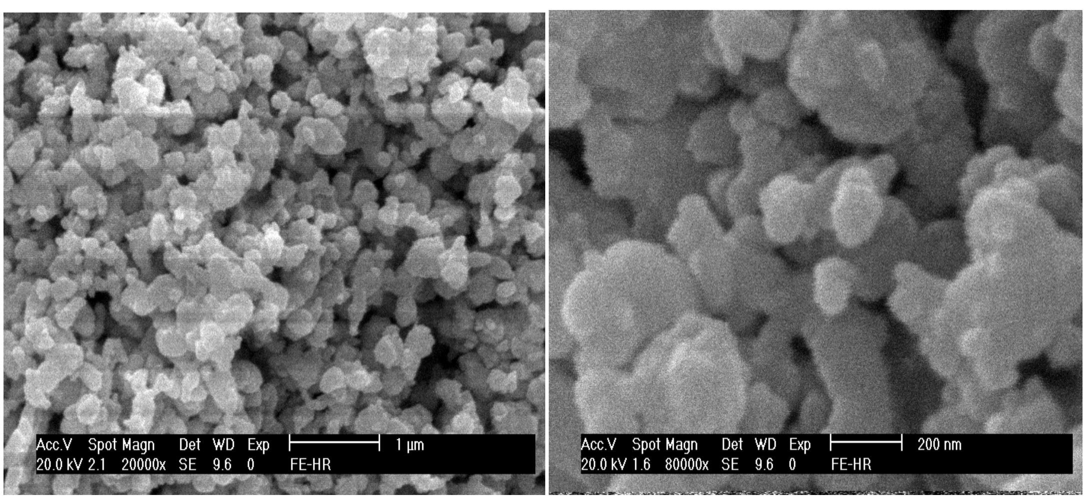

Fig. 2. SEM images of $AgInS_2$ nanoparticles.

SEM images of prepared sample shown in Figure 2 reveal the particle morphology with the average size of 68 nm. A minor agglomeration is observed in these images, which usually refers to presence of high conducting surface for obtained $AgInS_2$ particles in nanoscale.

Conclusion

In a summary, $AgInS_2$ nanoparticles were synthesized by using microwave assisted precipitation reaction between $AgNO_3$, $InCl_3.4H_2O$ and thioacetamide. SDS was used as surfactant to prevent high agglomeration and supply desired dilution. This method is a novel and efficient process to synthesize nanomaterials with various morphologies. High reproducibility, improved yields, small particle size and reach to product at the short time can be considered as advantages of this method.

Acknowledgments

The financial support of this study, by Iran University of Science and Technology and Iranian Nanotechnology Initiative, is gratefully acknowledged.

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