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SYNTHESIS OF As_2Se_3 IN ORGANIC MEDIUMСИНТЕЗ As_2Se_3 В ОРГАНИЧЕСКОЙ СРЕДЕ

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Abstract. Arsenic (III) selenide has been synthesized in organic medium by using metaarsenite and amorphous selenium. The synthesis was carried out at the temperature range of 433–443 °K, pH ≥ 10 , during 2–8 hours. Resulting sediment is filtered, washed at first by 0.1 M of acetate acid solution, then by ethyl alcohol and dried at 353 °K. The chemical analysis of the sediment is made, thermal, X–ray phase analyses are carried out, and electron microscope images of microparticles are taken.

Аннотация. Селенид мышьяка (III) синтезировали в органической среде с использованием метаарсенита и аморфного селена. Синтез проводили в температурном интервале 433–443 °К, при pH ≥ 10 , в течение 2–8 часов. Образовавшийся осадок отфильтровывали, промывали сначала с помощью 0,1 М раствора уксусной кислоты, потом этиловым спиртом и сушили при температуре 353 °К. Проводился химический, термический, рентгенофазовый анализ осадка. Изображения микрочастиц получены с помощью электронного микроскопа.

Keywords: arsenic (III) selenide, sodium metaarsenite, selenium, thermal analysis, morphology, X–ray analysis.

Ключевые слова: селенид мышьяка (III), метаарсенит натрия, селенид, термический анализ, морфология, рентгенофазовый анализ.

Introduction

Thin films of glass semiconductor chalcogenides found wide application in holography, electrography and photosensitive films, photothermo–plastic records [1]. The physical properties of glass semiconductor chalcogenides, as well as the simplicity of sample preparation technology make it a perspective material for fiber and integrated optics [2]. Such glasses have high beam refraction indices, it makes more important their application in optical fiber brunch. Selenium forms stable covalent bonds basically with arsenic ions and that is the most widely studied dual system. Arsenic (III) selenide is promising material for large number of applications in xerography, photocells, switching and memory devices, semiconductor and optical material. Arsenic (III) selenide is synthesized mainly by the ampoule method. Currently, extensive research work is carried out in the direction of production of arsenic (III) selenide thin films, nano– and of micro–particles. According to information obtained, in most cases, nano– and micro– particles can be produced solvo or hydrothermal synthesis conditions.

Experimental Details

To synthesize arsenic (III) selenide compound in the organic medium a sodium metaarsenite has been used with amorphous selenium in the ethylene glycol medium. Reagents were taken as samples in accordance with the stoichiometric composition of arsenic (III) selenide compound. After mixing reagents have been placed in experience flask to a teflon cuvette and have been put to a microwave oven. The reaction carried out in the temperature range of 433–443 °K, pH >10 limits, during 2–8 hours. Obtained sediment is filtered, at first washed with 0.1 M of CH₃COOH solution and then ethanol and dried in vacuum at 353 °K.

Physicochemical properties of arsenic (III) selenide compound produced in the organic solvent medium were studied by means of chemical analysis, differential thermal analysis in Thermoskan–2 device, X–ray phase analysis in D8 ADVANCE powder diffractometer, of morphology in microstructure analysis (Hitachi TM3000), optical absorption spectrum with spectrophotometer Spekol–1500.

Result and Discussion

The resulting precipitate was investigated by microstructure analysis. It has been ascertained that the nanoparticles (Figure 1) are formed at 443 °K. In practice it has been ascertained that sizes of nanoparticles vary depending on the temperature, pH and heat treatment time.

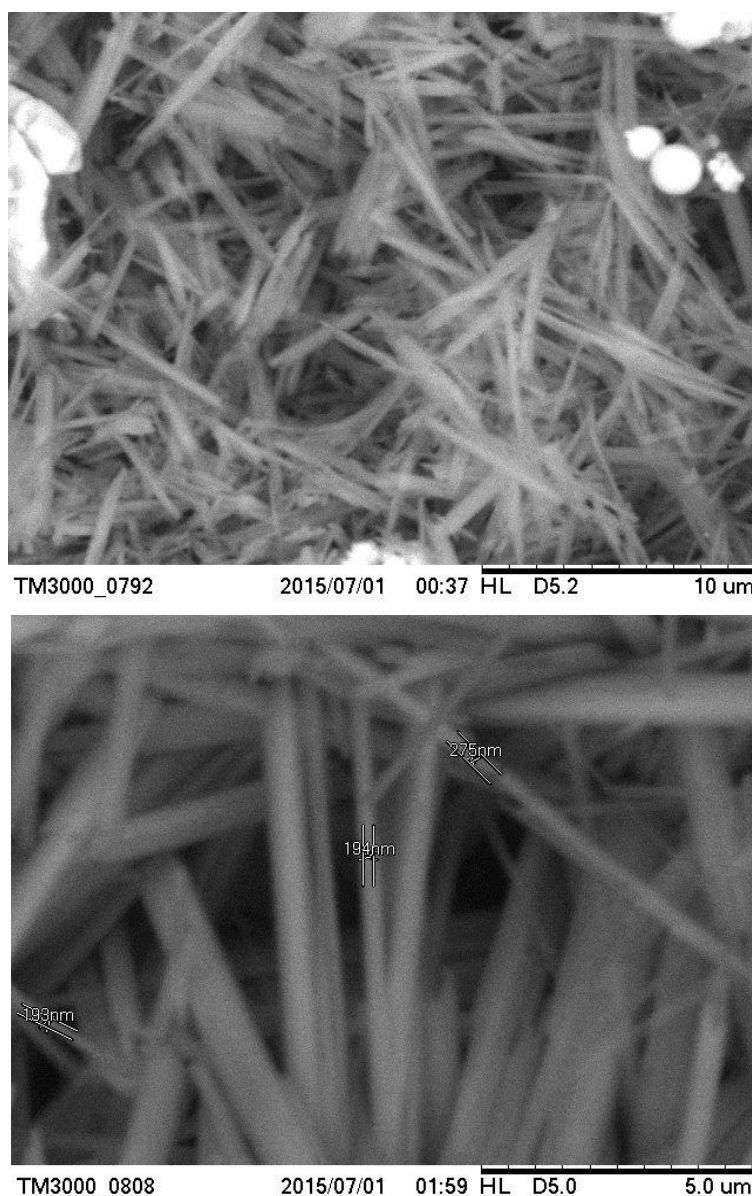


Figure 1. Nanostructure of arsenic (III) selenide at 443 °K.

As a result of analysis of micrographs taken on a glass substrate it has been revealed that the compound produced in the form of rod-shaped nano-particles. The size of particles is in the range of 190–280 nm.

X-ray phase analysis (D8 ADVANCE in powder diffractometer, $\text{CuK}\alpha$, $\lambda=1,5406\text{\AA}$, $0 < 2\theta < 80^\circ$) of the compound has been carried out. As a result of XPA the identity of arsenic (III) selenide compound has been confirmed (Figure 2).

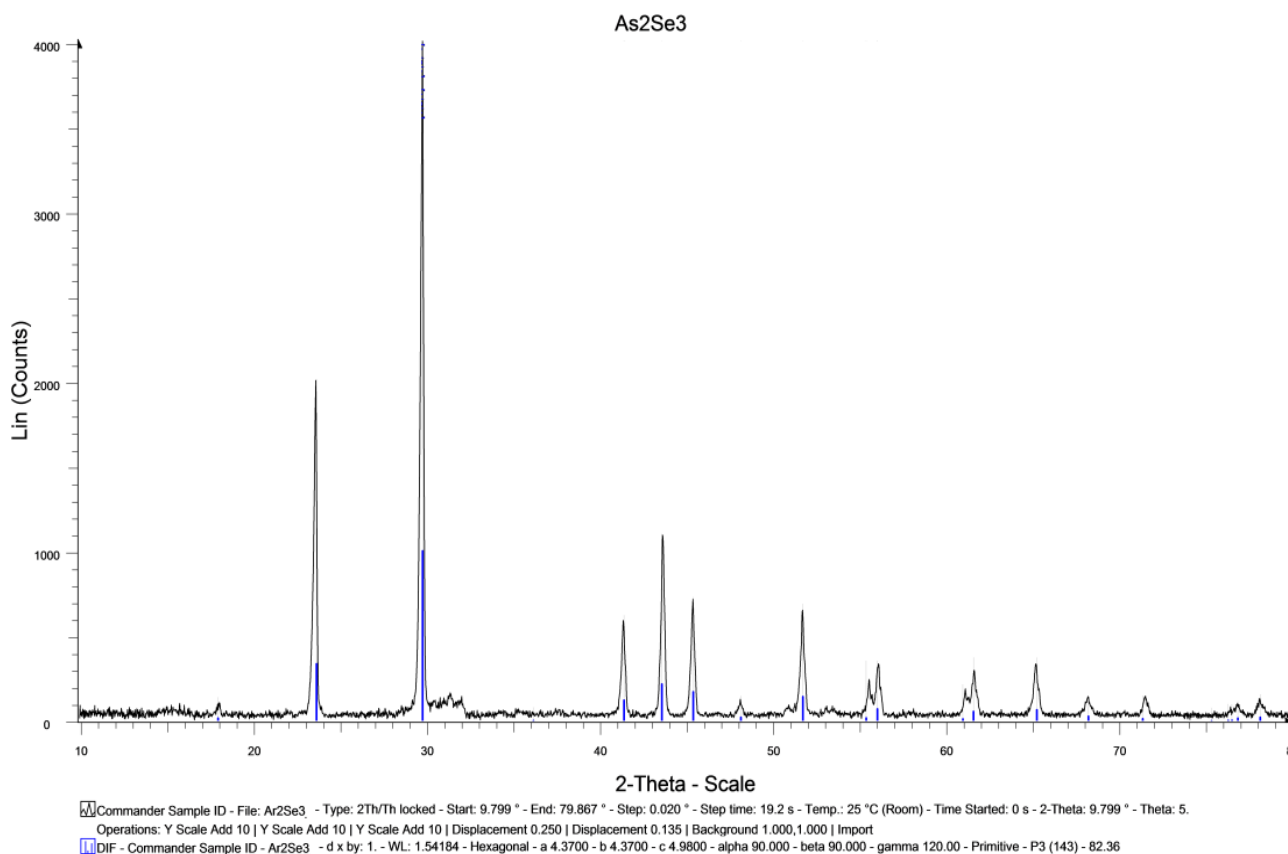


Figure 2. X-ray structure of arsenic (III) selenide compound.

The melting temperature of arsenic (III) selenide has been determined by Termoscan–2 devices. It is ascertained that the melting temperature of As_2Se_3 is 380°C , the crystallization temperature — 200°C , the softening and glass-transition temperature $T_g = 185^\circ\text{C}$. DTA curve is shown in the Figure 3.

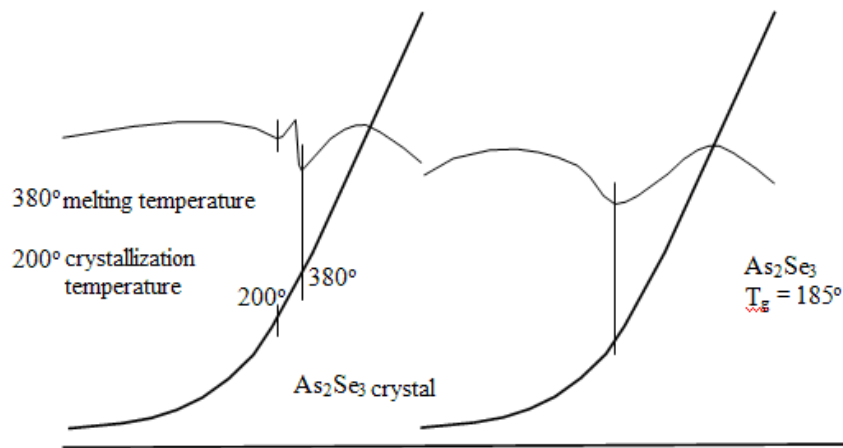


Figure 3. DTA of As_2Se_3 .

Optical transmission coefficient of arsenic (III) selenide semiconductor is determined in the wave-length range of 190–1100 nm by the method of single-beam spectroscopy. Spectroscopic measurements were carried out at compactly designed Specol–1500 spectrophotometer working in single-beam mode. Energy-gap width was calculated on the basis of spectrum produced by this spectrophotometer. Determined energy-gap width changes about the value of $E_g \approx 1.68$ eV.

Conclusion

In the course of this reaction it has been determined that during the hydrothermal synthesis an arsenic (III) selenide is obtained in the form of nano- and microrods, at the same time spherical particles also can be observed.

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