Study of synthesis condition of silver antimony selenide by hydrothermal method

B. Z. Rzayev, A. M. Garayev, A. B. Rzayeva
Nakhchivan Branch of NAS of Azerbaijan Institute of Natural Resources, Nakhchivan, Azerbaijan,
e-mail: aliye.rzaeva@mail.ru

Analysis of literary material shows that for $AgSbSe_2$ only thin films by vacuum thermal evaporation have been obtained. With regard to the production of nano- and microparticles of $AgSbSe_2$ data are not available in the literature. The influence of the deposition conditions of amorphous-crystalline nano- and microparticles of $AgSbSe_2$ from ethylene glycol solutions on their morphology is investigated in the present work.

The mixture of potassium antimonyl tartrate with silver chloride (1) was mixed with ethylene glycol; then sodium selenosulfate was added to the mixture as a selenizing reactant. Experimental glassware in a teflon cell was placed in a microwave electric oven Speedwave four BERGHOF (Germany). The sample was stored in the oven for 10 h at a temperature of 433 K. The resulting precipitate was filtered through a glass filter, washed with diluted solution of hydrochloric acid, ultrapure water, finally, with ethyl alcohol, dried at 333–343 K in vacuum. The output was 90–92 %. At the temperature of 453–473 K resulting precipitate of $AgSbSe_2$ was partially dissolved in ethylene glycol. Experiments have been carried out with fine chemical reagents. Composition of the obtained compound (ratio Ag : Sb : Se) was determined by derivatograph NETZSCH STA 449F349F3 (Germany) and by chemical analysis. Phase analysis of nano- and microparticles of $AgSbSe_2$ was studied using the X-diffractometer D2 PHASER "Bruker" (CuK_α radiation 2θ, (λ = 1.54056 Å), 10–70 degrees) and chemical analysis. Morphological investigations have been performed using scanning electron microscopy TM 3000 (Hitachi, Japan). Chemical, thermographic, X-ray and morphological analyses were performed. Optical absorption was measured using a spectrophotometer U-5100 (Hitachi), the value of the band gap on the basis of the spectrum obtained from the dispersed $AgSbSe_2$ solution in ethyl alcohol was calculated.

Analyses of the samples synthesized by hydrothermal method (silver antimony selenide), were carried out by thermogravimetric and differential colorimetric methods. The analysis results have shown that when the sample was heated to the temperature of 20–750 ºC the weight loss of 10–11 % occurred. Calculations according the graph (the melting point) have shown that the ratio by mass (weight) of silver and antimony to selenium corresponds to 53.42 : 45.57. The data obtained correspond to the formula of $AgSbSe_2$. Differential calorimetric analysis of the sample showed that at the melting point (862 K) the peak area was 9,5781 μVs/mg.

The results of X-ray analysis of silver antimony selenide by the intensities and positions of the exhibiting peaks fully correspond to the standard data (PDF 00:012:0379). The effect of temperature on the production, growth and formation of nano- and microparticles synthesized by solvotermal method was investigated; the images of these particles using an electron microscope were recorded. Composition of $AgSbSe_2$ compound was also confirmed by chemical methods.