IOSR Journal Of Applied Physics (IOSR-JAP)
e-ISSN: 2278-4861.Volume 12, Issue 5 Ser. II (Sep. – Oct 2020), PP 23-27
www.Iosrjournals.Org

Indium (III) Selenide Production in Organic and Inorganic Mediums

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Abstract
Conditions of indium (III) selenide synthesis in organic and inorganic mediums are studied in this work. Select the area volume yellow precipitate. To a solution of indium tartrate complex selenerizing added sodium borohydride at pH 10-11. It is ascertained that the nano- and micro particles of In_2Se_3 compound produced in organic medium (ethylene glycol) form in the shape of flakes at 453 – 473 K for 10 – 12 hours. Indium selenide output is 95 – 97 % in organic medium, 98 – 99 % in inorganic medium. Chemical composition of the sediment and its release rate from the solution has been determined. The rate of the sediment of deposition from the solution was determined. Thermal and X-ray analyses of resulting indium selenide have been made.

Keywords: *Indium (III)selenide, nanoparticles. thermographic analysis, hydrothermal method, X-ray analysis, deposition rate, chemical analysis.*

Date of Submission: 10-09-2020 Date of Acceptance: 25-09-2020

I. Introduction

As comparison of In_2Se_3 compound production both in organic and inorganic medium isn't found in technical publications, we have combined our experiments and should like to specify their similar and differing characteristics in this paper.

Various methods of indium selenide production have been published during last years. Let us cite the main essence of some of them.

The work [1] reports that In_2Se_3 single-crystal thin layer preparation has been carried out at the first time by means of mechanical peeling and investigation of $(\alpha \to \beta)$ crystal phase transitions, and corresponding changes of electrical properties in these thin layers are also given in this work. As opposed to bulk single-crystals, β-phases can remain in single-crystal thin layers at the room temperature. The single-crystal nature of the layers before and after a phase transition ensures unique determination of changes in electrical resistance. In particular, the β-phase has resistivity in 1 – 2 orders less than the $\alpha-$phase. In addition, we find that the temperature of $(\alpha \to \beta)$ phase transitions increases as much as 130 K if the layer thickness decreases from ~87 nm to ~ 4 nm. These single-crystal thin layers are ideal for research of phase transitions scaling behavior and electrical properties changing in connection to this process.

The work [2] determines boundary conditions and formation zone of indium sulfide and (III)selenide at the deposition by thiocarbamide and selenocarbamide. Potentiometer of indium chloride $InCl_3$ has been carried out in the hydroxide solution concentration range of 0.0001 – 0.100 mole/l.

Substitution solid solution films containing up to 7.5% indium are produced at the first time by chemical deposition from the water mediums in the system $Cu_2Se-In_2Se_3$. The composition, structure and morphology of these films are studied. It is ascertained by the X-ray phase analysis and X-ray photoelectron spectroscopy method that in a solid solution composition copper is in the univalent state. It is shown that layers have the global structure and are nanostructures. Heightened interest to substitution solid solutions in the system $Cu_2Se-In_2Se_3$ is caused by availability of their application as materials for the solar power engineering, in consideration of about 20% radiation transformation efficiency factor, high absorption coefficient and resistance to radiation damage. Various methods of $Cu_2Se-In_2Se_3$ films production are known: thermal vacuum evaporation, pulverization of water solutions, molecular beam epitaxial, electrochemical deposition, high-frequency ion sputtering and selenization of separate $Cu-In$ layers. But all these methods need complex processing equipment; high temperatures, high vacuum, and don't ensure required functional characteristics and also considerable reduction in commercial value. A serious prospect has $Cu_2Se-In_2Se_3$ solid solutions films hydrochemical deposition method, which is featured by simple hardware implementation and low-temperature conditions of processing.

Structural properties depending on In_2Se_3 powder pressure have been studied at the room temperature [4]. In_2Se_3 is transformed into the β-phase at 0.7 GPa, that's in the order lower that critical pressure phase transition in typical semiconductors. The β-phase continues from decompression up to ambient pressure. Spectroscopic experiments confirm this result. The core module and s/a ratio for the β-phase have shown strong nonlinear dependence on pressure.

Structural and electronic properties of amorphous and single-phase polycrystalline films $\gamma-$and $\kappa-In_2Se_3$ are determined [5]. Stable $\gamma-$phases origin uniformly in a film volume and have high specific resistance, while meta-stable $\kappa-$phases origin at the film surface and have moderate resistance. Films considerably differ, but the electronic properties are similar. Resistance increase in the amorphous In_2Se_3 film is interpreted at annealing in respect to substance replacement at crystallization. At In_2Se_3 film preparation great attention should be paid to electrical measurement, as the presence of chalcogen excess or surface oxidation can considerably influence the film's properties.

II. Experimental Procedure

10 ml is taken from the indium chloride solution (0.1 M) and tartaric acid solution is added, whereupon indium transits in a firm complex. The complex is so firm that indium hydrate isn't formed at the action of ammonia solution at pH 1 – 12. Without tartaric acid indium and ammonia form hydrate of the composition $In(OH)_3$ at pH > 3.5. Indium is conversed into the firm complex with the aim to carry out reactions in alkaline medium. Selenizing reagent is prepared by dilution of elementary selenium (amorphous or melted) in natrium borhydride solution. During the reaction indium solution pH is regulated within the limits of 10 – 11, and selenizing reagent is added in stoichiometric quantity. Bulky sediment of black color is formed. The qualitative analysis of this sediment has shown the presence of indium and selenium. In various experiments are produced sediments of different colors when medium pH changes. The set of experiments has been carried out to specify color change and hydrogen ions concentration impact. The results are given in the table 1.

Table 1: Dependence of indium selenide color and deposition completeness on medium pH
[In] = 0.1 M, [Se] = 0.1 M, temp. 323 – 333 K

№	$InCl_3$, ml	Solution Se	pH	Sediment, mg	Sediment color	Sediment formula
1	10	15	8	178.1	black	In_2Se_3 + Se
2	–	–	9	231.2	brown	In_2Se_3
3	–	–	10	233.7	yellow	In_2Se_3
4	–	–	11	227.5	yellow	In_2Se_3

It is evident of the table data that selenium in the first experiment is partly hydrolyzed and mixed with the sediment; therefore complete deposition of indium selenide doesn't occur (separate experiments are carried out to ascertain selenium solution behavior at various pH values; it is ascertained that selenium is partly deposited from the solution at pH lower than 9). At the rest of experiments resulting sediments consist of In_2Se_3. Sediments are washed by distilled water and dried at 33 K.

III. Results And Discussion

The chemical analysis of produced sediments is carried out. The sediments are decomposed by nitric acid, evaporated up to isolation of salt mixture. Then distilled water and some drops of hydrochloric acid are added to the mixture, and all that is moved to a graduated flask. Indium is deposited by ammonia in the form of hydrate. The sediment is filtered, washed by distilled water and decomposed in a muffle furnace at 350 ºC. Resulted In_2O_3 is weighed. Selenium in filtrate is determined by the hydroxylamine method. The results are given in the table 2.

Table 2: Chemical analysis of indium (III) selenide sample

In_2Se_3 sample, g	Components, g			
	indium		selenium	
	theoretic.	pract.	theoretic.	pract.
0.4322	0.2548	0.2561	0.1747	0.1761

Notice: The results are average values of four experiments.

As evident from the table results, the number of elements found in experiments is in good agreement with theoretical calculations. And it shows that produced indium selenide corresponds by its composition to the formula In_2Se_3.

The main technological parameter of sediment is its ex-solution rate. Taking this into account, ex-solution rate of In_2Se_3 has been determined (10.2 g of sediment used for experiments).

Table 3: Indium (III) selenide ex-solution rate

V, ml	T, min.	V, ml	T, min.
1000 – 900	0.20	500 – 400	0.45
900 – 800	0.25	400 – 300	0.50
800 – 700	0.30	300 – 200	0.60
700 – 600	0.35	200 – 100	0.70
600 – 500	0.40	100 – 15	1.40

The data in the table 3 shows that within 6 minutes 10.2 g of the sediment (up to 15 ml in volume) is deposited of 1000 ml solution. This is a very good indicator.

The thermal analysis of the produced In_2Se_3 sediment is carried out in the presence of air by the derivatograph NETZSCH STA 449F3. The results are shown in the figure 1.

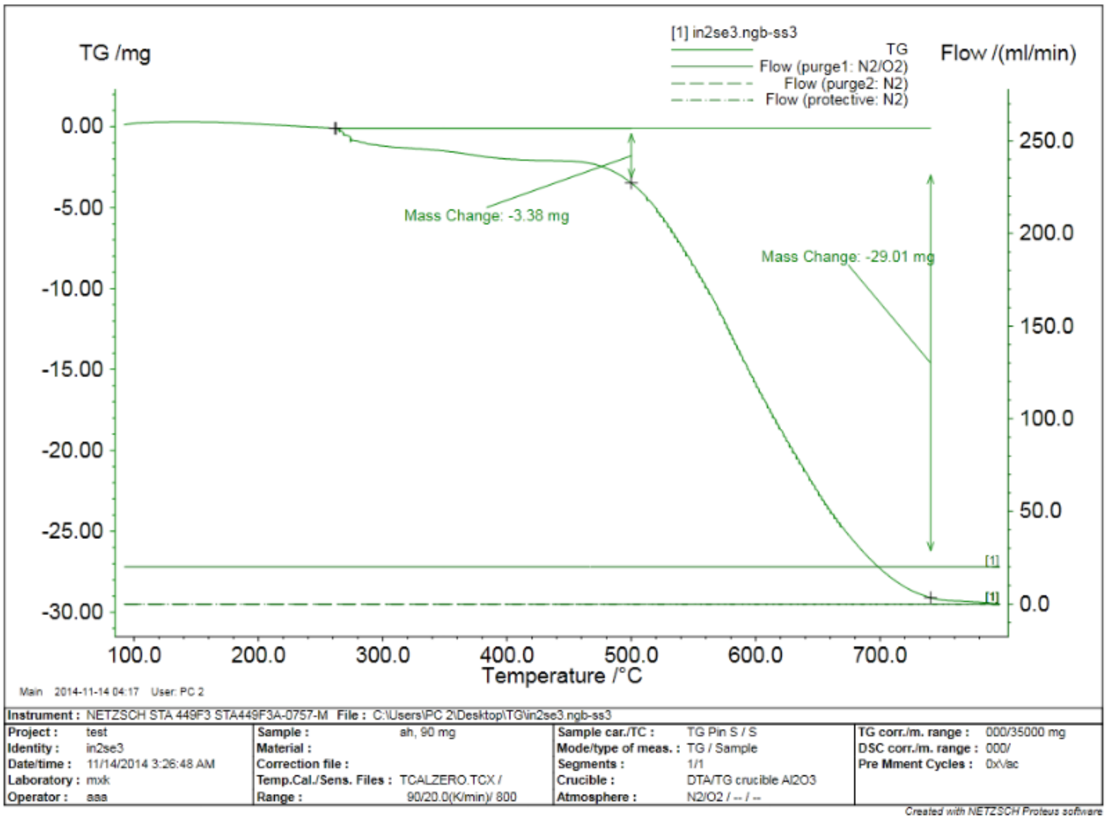

Fig. 1: The thermal analysis of In_2Se_3

As evident from the fig. 2, the composition of In_2Se_3 sample with the mass 90 g doesn't change up to the temperature 300 ºC. The mass loss at 300 – 500 ºC is 3.38 mg. This is likely due to the presence of elemental selenium in the sample's composition. The mass loss at 500 – 740 ºC is 29.01 mg. This shows oxidation of In_2Se_3. The composition of the sample contains 44.32 mg of indium, and this corresponds to 54.39 mg of indium oxide. In_2O_3 in amount of 57.61 mg has been found in the experiment. Here the difference is only 3.22 mg. The difference can be attributed to incomplete sublimation of selenium oxide. All this shows that the composition of resulting indium selenide corresponds to the formula In_2Se_3. Also result of X-ray of indium selenide (fig. 2).

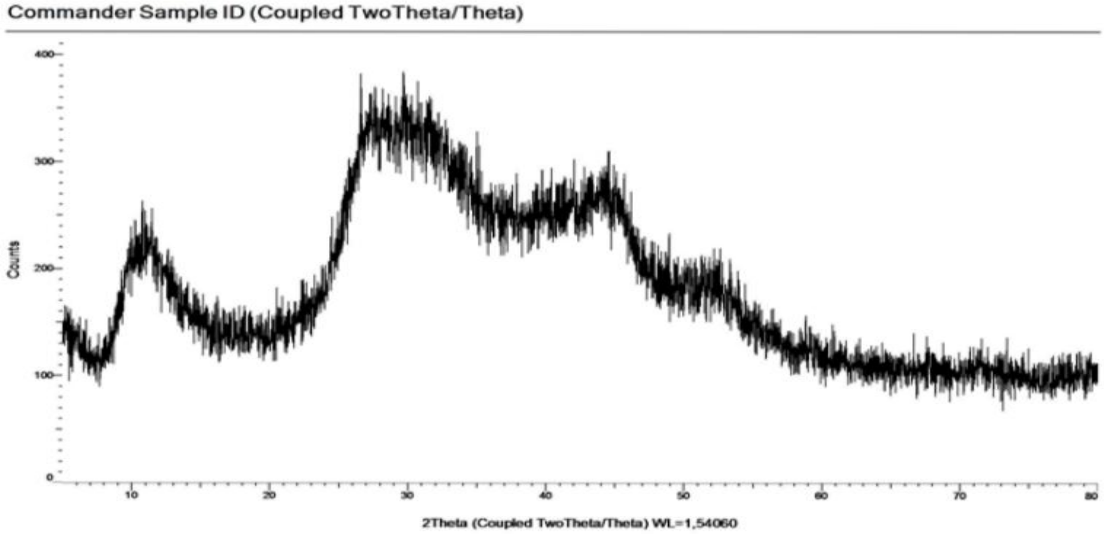

Fig. 2: X-ray of In_2Se_3

The elemental analysis of the compound composition is made by the method of electron probe microanalysis (on the device Launch Trion XL dilution refrigerator-OXFORD). The results are shown in the following table 4 (fig.3).

Table 4: The results of the compound elemental analysis

An element	Weight %	Atomic %
Se	56.08	64.99
In	43.92	35.01
Totals	100.00	

The results in this table correspond to stoichiometric composition of the compound. The view of nanoparticles elementary spectrum is presented in the figure stated below.

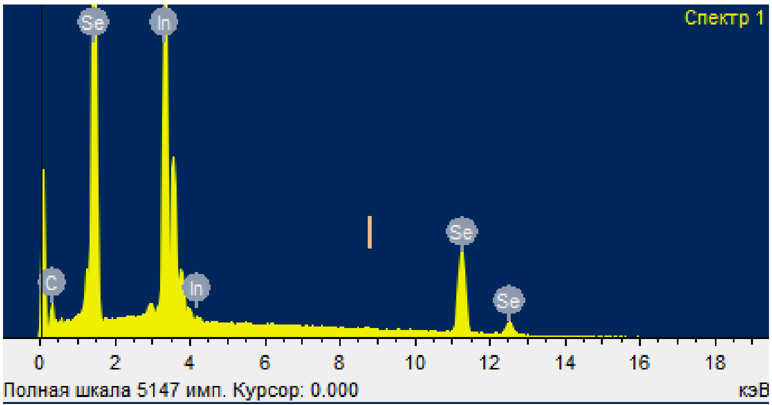

Fig. 3: In_2Se_3 compound elementary composition spectrum

Effect of temperatures (140, 160, 180 ºC) on emerging, growing and forming of nanorods is studied at the solvothermal method of In_2Se_3 producing, forms of nanorods are taken (fig. 4). As evident from the figures, their width changes in the range 1.09 μ – 230 nm. The compound obtained in water medium doesn't form at the room temperature (fig. 5). Morphology of the obtained compounds was studied at the electronic microscope TM-3000 Hitachi.

a)

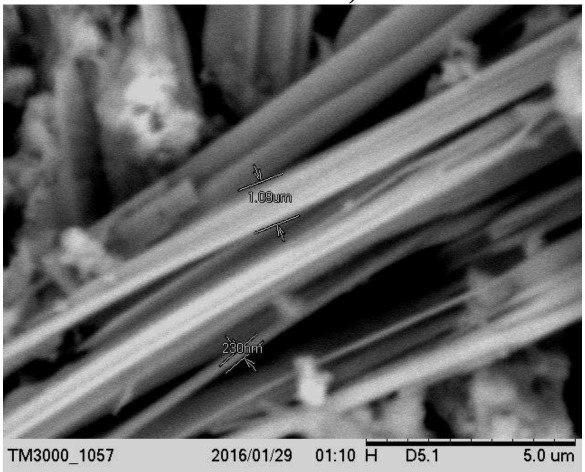

Fig. 4: In organic medium, at 150 ºC during 6 hours

b)

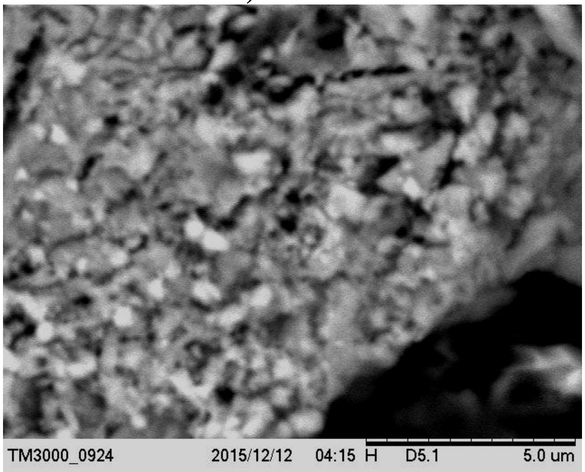

Fig. 5: In inorganic medium, at the room temperature,

IV. Conclusion

Indium tartaric acid is translated into a stable complex. Then, the solution was added selenerizing (selenium borohydride solution). The voluminous yellow precipitate is formed. The composition of sediment to determine the chemical and electronic – rentgen spectral analysis methods. It determines the rate of the sediment discharge from the solution. Results of the experiments showed that both in the organic and inorganic in composition environments is the same - In_2Se_3 of sediment. Complete difference is that micro and nano sticks In_2Se_3 are formed only in an organic medium (in ethylene glycol used by as the organic medium).

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G.I. Isakov, et. al. "Indium (III) Selenide Production in Organic and Inorganic Mediums." *IOSR Journal of Applied Physics (IOSR-JAP)*, 12(5), 2020, pp. 23-27.