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# OBTAINING OF AgCuS EPITAXIAL LAYERS WITH MOLECULAR BEAMS CONDENSATION METHOD

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**Abstract.** Thin layers of AgCuS compound were obtained with condensation method from molecular beam. The optimal mode for obtaining an isotropic medium was determined, in order to keep the chemical composition maintenance, conditions were created to include of additional chalcogen atoms into the system. The surface structure and micro composition analyzes of the obtaining epitaxial layers were studied per Scanning Electron Microscope (SEM). The obtaining results for the thin layers were compared with the obtaining results for the AgCuS crystal.

***Keywords:*** *AgCuS, SEM, surface structure, chemical analysis.*

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## 1. Introduction

Physico-chemical properties of functional materials are formed of depending on their synthesis methods and structural features. That's why the acquisition of these materials and the phase formation processes should be studied. As research methods develope, these studies have begun to be carried out with higher accuracy (Jabarov *et al*., 2021; Normuradov *et al*., 2023a; Alekperov *et al*., 2021; Davranov *et al*., 2024). Chalcogenide semiconductors have a special place among functional materials. So that different structures can be formed in these compounds depending on the bonds created by metal and chalcogen atoms and the valence of metal atoms. Therefore the phase generation processes in these compounds are widely being studied recently. The valence changeability observing in metal atoms causes the formation of two-phase systems in some cases. Because of the variable valency of copper atoms, such cases were observed during the structural studies were carried out Cu – X (X = S, Se, Te) systems (Aliyev *et al*., 2019; Yamamoto & Kashida, 1991; Kashida & Yamamoto, 1991). Therefore, studying is important the structure, phase transitions and phase formation processes of these materials.

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It was determined that in the case of thin layers certain differences appears in many properties of materials. But the characteristics of thin films should also be studied for the preparation of multifunctional systems in small sizes. Therefore, extensive researches are being conducted in the direction of obtaining and studying thin layers of many materials recently (Normuradov *et al*., 2022; Bekpulatov *et al*., 2023; Mursakulov *et al*., 2022). On the base of thin layers of chalcogenide semiconductors, it is possible to obtain heterojunctions with different purposes (Normuradov *et al*., 2023b; Madatov *et al*., 2024; Dovranov *et al*., 2024). In order to ensure the functionality of thin layers, special attention should be paid to the formation of phases during the obtaining of these layers. Especially the properties of the structure should be maintained during obtaining thin layers of materials with different components. It appears from previous researches that the Ag-Cu-S system carries enough complex character. It was determined that this compound keeps its properties under the influence of high temperature and ionizing rays. For that reason, this compound have a special place among chalcogenide semiconductors (Guin *et al*., 2016; Jabarov *et al*., 2021; Trots *et al*., 2007).

It is known that obtaining thin layers of complex systems creates some technological difficulties. Therefore, thin layers of systems with the highest symmetry elements should be obtained. It was known that the highest symmetry elements in the Ag-Cu-S system are the AgCuS compound obtaining with equal concentrations of Ag and Cu elements during results obtaining from the structural researches. It is also appropriate to study of AgCuS epitaxial layers during the studying thin layers of the Ag-Cu-S system. For this purpose, thin layers of the silver copper sulfur system were obtained and their surface structure and micro-composition analysis were conducted.

## 2. Experiments

### *2.1. Synthesis process*

AgCuS polycrystal has been used for the production of thin layers. The synthesis process of polycrystals has been used the typical standard method for chalcogenide semiconductors. B5 silver and copper metals, masses of high-purity sulfur elements measured on an electronic scale and was taken in appropriate stoichiometric quantity and was mixed. In order to obtain a homogeneous system, 33.3333% Ag, 33.3333% Cu and 33.3333% S were taken according to the stoichiometric amount. A high vacuum ($P = 10^{-3}$ Pa) was created in the quartz ampoule and the obtaining mixture was filled into this ampoule. The ampoule was placed in a constant temperature furnace. The temperature of a sample was increased up to the melting temperature of sulfur ($T_S$ = 395 K) and was kept at this temperature for 4-5 hours. After the reaction of sulfur with metals (Ag and Cu), the temperature was raised to their melting temperature ($T$ = 1410 K). It was kept at this temperature for 4-5 hours. After ending these stages, the furnace temperature was cooled at a slow rate of 60 K/h. In order to homogenization of sample, it was kept at temperature $T$ = 450 K for 7 hours.

### *2.2. Technology of obtaining epitaxial layers*

Thin layers of AgCuS compound were obtained with a special technology (Mehrabova *et al*., 2024; Alekperov *et al*., 2023). The use of evaporation sources with special purity is one of the main conditions in the technology of obtaining epitaxial layers. Polycrystal of AgCuS compound was used for obtaining thin films of the Ag-Cu-S system. The crystal was used as an evaporation source for obtaining epitaxial layers. Epitaxial layers were obtained with molecular beam condensation (MDK) method in a UVN-71P-3 device in a vacuum at $(1\text{-}2)\cdot10^{-4}$ Pa. The scheme of the device was given in Figure 1. In a vacuum, the molecules which coming out of the vaporization source heated at high temperatures spread out according to the laws of optical geometry fall to the bottom and there their condensation occurs. At this time, the length of molecules free path is many times greater than the dimensions of the evaporation device chamber.

It is known that losting of chalcogen components (S, Se, Te) happens during evaporation of chalcogenide compounds and their solid solutions in vacuum. Therefore, a compensatory additional chalcogen source was used in the growing process of epitaxial layers. The vaporizer of the main source was made from graphite of the branded MPQ-6. In addition to graphite vaporizers, vaporizers that made of tungsten and tantalum were used for obtaining epitaxial layers of these materials. The crystal structure, properties of the obtaining epitaxial layers don't differ much and carbon (C) atoms in composition weren't observed. Before using the graphite vaporizers were cleaned with benzene, boiled in 3:1 $HCl:HNO_3$ solution for several hours, washed in distilled water and finally thermally treated in vacuum at $T = 1300$ K for several days. The evaporator of main source consists of a two-plug Knudsen cavity which made of graphite in the form of a cylinder, heated by current. The diameter of the hole opened in the groove is 0.6 mm. The temperature of evaporator was measured through V7-21A digital universal millivoltmeter with a platinum-rhodium (PR 30/668) thermocouple.

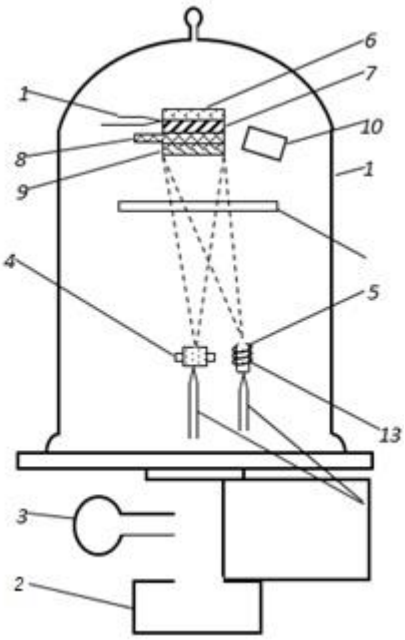


**Figure 1.** Scheme of UVN-71P3 vacuum device: 1 - lid, 2 - high vacuum pump, 3 - ion lamp, 4 - Knudsen groove, 5 - effusion groove, 6 - substrate heater, 7 - substrate, 8 - displaceable mask; 9 - mask holder; 10 - quartz thickness gauge; 11 - thermocouple; 12 - partition; 13 - the heater of the deffusion groove

A mover partition was placed between the evaporator and the bottom, in the path of the molecular beam for precisely control the acquisition time of epitaxial layers. Using from partition was created an opportunity to conduct the growth process of the epitaxial layer at a constant speed. Considering that the partial decomposition of the vaporized material, the Knudsen cavity was filled with new material each time for obtaining a layer with a stable chemical composition. In the experiments which conducted to control the crystal perfection, type and concentration of charge carriers in the obtaining epitaxial layers used an additional compensating sulfur evaporation source. The source of sulfur vaporization consists of an effusion cavity made from quartz. The temperature of the cavity was controlled using a RIF-101 device with an accuracy of ±0.05 °C. The effusion cavity was made whole for preventing contamination of the vacuum system and loss of volatile S component during heating. The insertion of the outlet pipe into the effusion groove directed the outgoing vapor bundle correctly onto the substrate. $h \sim 1$ μm thick epitaxial layers were obtained.

### *2.3. Study of surface structure*

A Scanning Electron Microscope is used for conducting the surface structure, size effects and micro composition analyzes of materials (Normuradov *et al*., 2020; Shchur *et al*., 2022). AgCuS crystal and thin films were also studied in SEM, ZEISS, ΣIGMA VP SEM microscope. Information about crystal and thin layers were comparatively analyzed based on the obtaining images. The amount of chemical elements in a sample was determined, amount of Cu, Ag and S elements with percentage were shown.

## 3. Discussion of results

The surface structure of AgCuS compound polycrystal and thin films obtaining on a glass substrate has been studied with Scanning Electron Microscope. The researches were fulfilled under normal conditions and at room temperature. The obtaining surface structure for polycrystals was given in Figure 2.

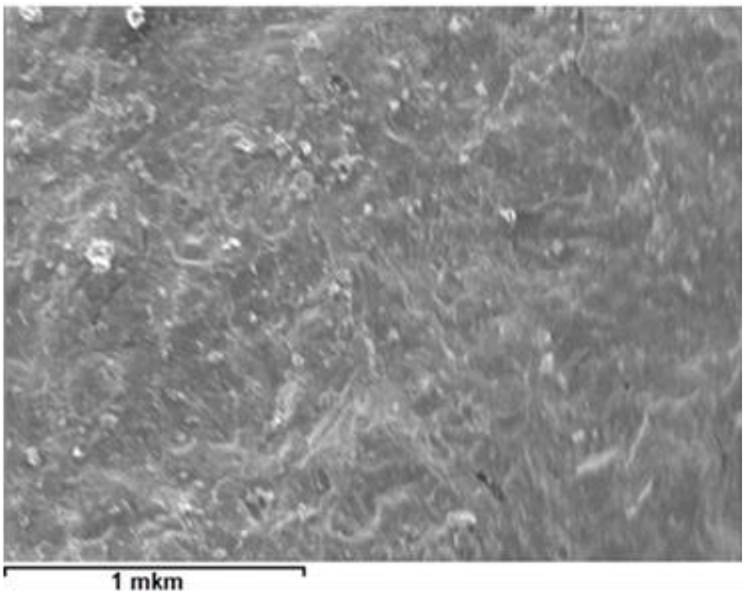


**Figure 2.** Surface structure of AgCuS compound polycrystal

As can be seen from Figure 2, the AgCuS compound was synthesized in a form of belonging to chalcogenide semiconductors. Sulfur atoms which appropriate stoichiometric amount was completely was solved and therefore wasn't detected as a residue. In order to see more clearly resolving of the chemical elements included in the compound, maps of the chemical elements were also obtained separately (Figure 3).

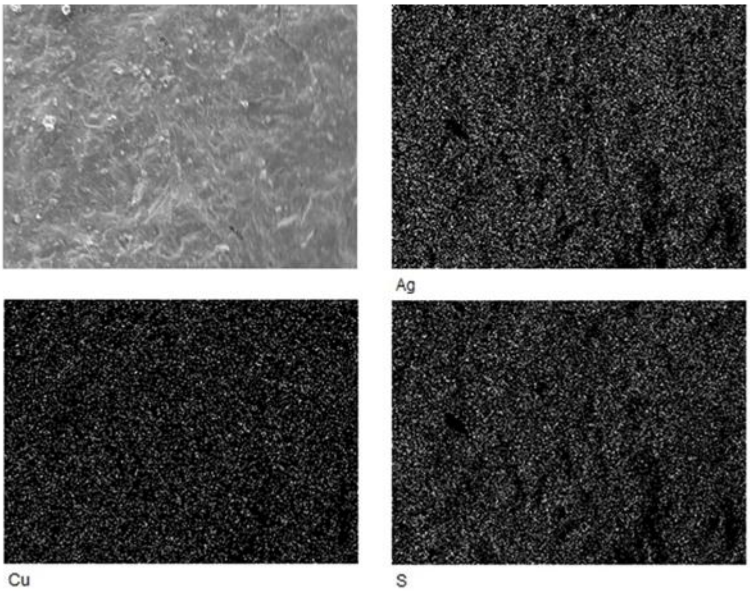


**Figure 3.** Map of chemical elements in AgCuS compound

As can be seen from Figure 3, the concentrations of Ag, Cu and S atoms are almost the same. This is an indicator of being synthesis high-quality of the AgCuS compound. It is known that it is also possible to obtain the energy spectrum of chemical elements with modern SEM microscopes. That's why the energy spectrum of the chemical elements that organizes the crystal was assigned during the researches. The obtaining spectrum was given in Figure 4.

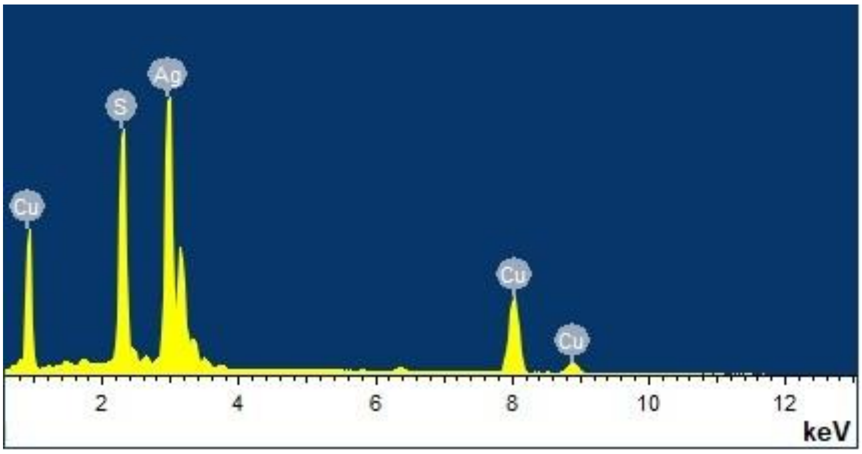


**Figure 4.** Energy spectrum of chemical elements in AgCuS compound

It can be seen from the spectra were given in Figure 4 that the crystal contains Ag, Cu and S atoms enough. The amount of these chemical elements with percentage was also determined during the researches. The obtaining results were given in Table 1.

**Table 1.** Amount of chemical elements with percentage in AgCuS compound

| Chemical element | Mass, % | Atom, % |
|---|---|---|
| S (Sulfur) | 14.20 | 30.54 |
| Cu (Copper) | 32.78 | 35.57 |
| Ag (Silver) | 53.02 | 33.89 |
| Total | 100 | 100 |

The studies conducted for the polycrystal of the AgCuS compound were also conducted for the thin film of the Ag-Cu-S system. It was determined with the weight method that the thickness of the thin layer obtaining on the glass is h = 1 μm. One of the main problems are the phase formation process during the acquisition of thin layers. Obtaining the structure of the starting material in small sizes is one of the main conditions for obtaining thin layers (Normurodov *et al*., 2021; Rysbaev *et al*., 2021; Touil & Mirouh, 2019). In order to study the process of phase formation in AgCuS thin film, SEM researches were conducted. The surface structure which obtaining normal condition and at room temperature was given in Figure 5.

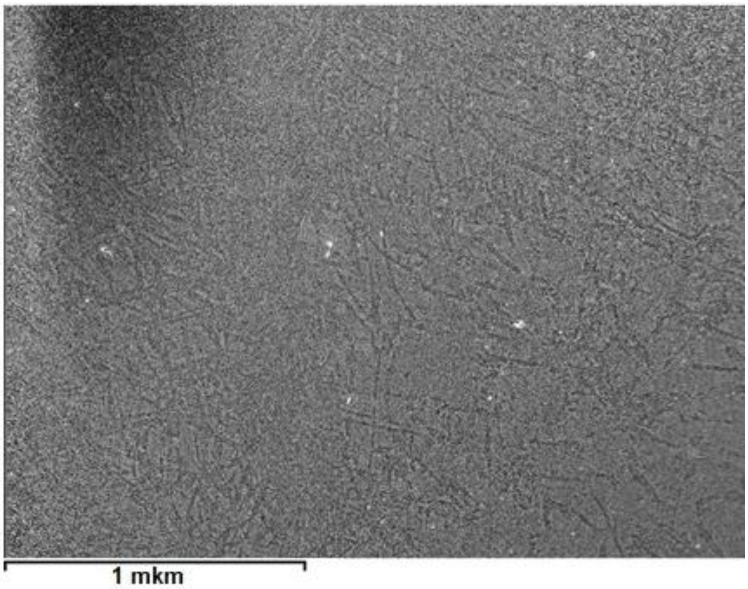


**Figure 5.** Surface structure of AgCuS thin layer

As can be seen from Figure 5, thin layers of the AgCuS compound were obtained with high accuracy. The additional sulfur atoms given to the system were solved stoichiometric amount and therefore weren't detected in a residue form. In order to see more clearly solving of the chemical elements included in the composition of the thin layer, maps of the chemical elements were obtained seperately (Figure 6).

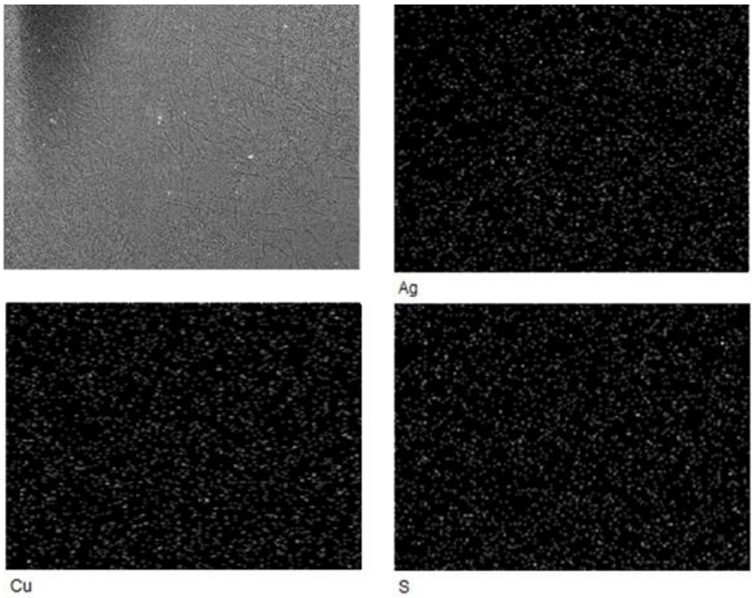


**Figure 6.** Map of chemical elements included in AgCuS thin films

As can be seen from the pictures in Figure 6, the concentrations of Ag, Cu and S atoms in the thin layer are almost the same. This is an indication that the thin film is obtained according to the AgCuS compound. But, if we compare the maps obtained in Figure 3 and Figure 6, we will see that the concentration of elements in the crystal is higher. In thin layers, the density of chemical elements is lower compared to the crystal. In order to obtain more information about the structure of the thin layer, the energy spectrum of the chemical elements was also determined. The obtaining spectrum was given in Figure 7.

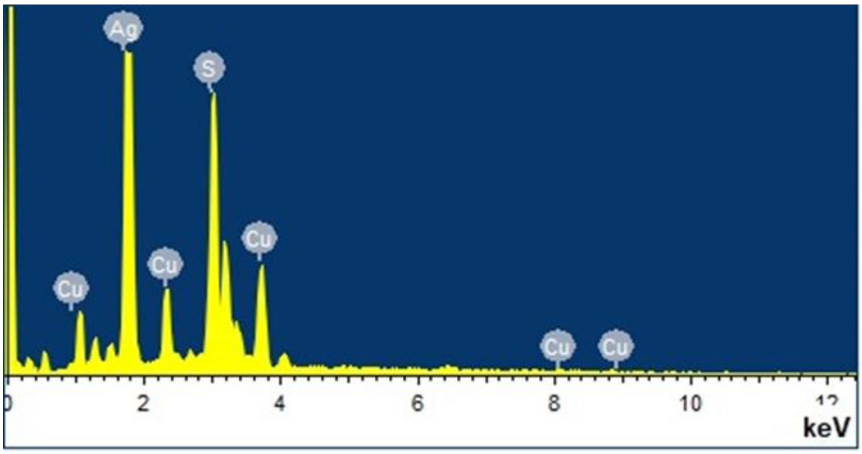


**Figure 7.** Energy spectrum of chemical elements included in AgCuS thin films

It is known that the stoichiometric amount of atoms included in the material in the system should be sufficient during the production of epitaxial thin films. Otherwise, the phase which corresponding to the crystal cannot be fully formed. It can be seen from the spectra given in Figure 7 that in the composition of thin layers are enough Ag, Cu and S atoms. The amount of these chemical elements with percentage was also determined during the SEM analysis. The obtaining results are given in Table 2.

**Table 2.** Amount of chemical elements with percentage included in AgCuS thin films

| Chemical element | Mass, % | Atom, % |
|---|---|---|
| S (Sulfur) | 13.22 | 31.56 |
| Cu (Copper) | 30.52 | 32.52 |
| Ag (Silver) | 56.26 | 35.82 |
| Total | 100 | 100 |

Chemical composition is almost the same when comparing the prices given in Table 2 with the prices given in Table 1. But a slight decrease was observed in copper atoms. This effect can be related to the concentration of atoms located on the surface during analysis with SEM analysis. Thus, electrons are scattered from atoms on the surface during SEM studies and therefore cannot penetrate into the samples. The concentration of atoms on the surface of the sample can be slightly different from the concentration of atoms inside. But atoms on the surface play an important role in the formation of structure, optical properties and electronic processes in thin layers (Ashraf *et al*., 2020; Rysbaev *et al*., 2014; Hashimov & Isayeva, 2024). During the chemical composition analysis of AgCuS polycrystal and thin layer was determined that there are no oxygen atoms on the surface. It is known that oxidation occurs on the surface of non-oxide materials. An oxide layer forms on the surface as it time passes. At this time, even the color of the materials changes. In order to prevent this, the bonds formed by the metal atoms which included in the composition of the material must be saturated case. Therefore, special attention is paid to the shape of ions and the saturated case of the bonds formed by them in chalcogenide semiconductors and during the production of their thin films (Tang *et al*., 2024; Normuradov *et al*., 2007; Rysbaev *et al*., 1997; Müller *et al*., 2024). Sulfur atoms which added to the system during the production of AgCuS thin films caused silver and copper of atoms to form bonds in a monovalent state. Therefore, oxygen atoms weren't observed on the surface of the obtaining thin layers.

## 4. Conclusions

The morphology and chemical composition of AgCuS polycrystal and thin film were comparatively studied. The researches were fulfilled with a Scanning Electron Microscope. It was determined that it is possible to obtain thin layers of AgCuS compound by the condensation method from the molecular beam. The chemical composition of the obtaining thin layers and the concentration of the elements included in the composition corresponded to the AgCuS compound. Sulfur atoms were additionally introduced into the system for participating of Cu and Ag atoms in a monovalent case in thin layers and creating of covalent bonds. The optimal condition for obtaining Ag-Cu-S epitaxial thin films was determined.

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