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INFLUENCE OF METALLOPHILIC INTERACTIONS ON PHYSICOCHEMICAL PROPERTIES OF ION-CONDUCTING GLASS SYSTEM (1-x)(0.27Sb_2Se_3–0.73$GeSe_2$) -$x$$Ag_2Se$[1]

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Abstract: The influence of silver selenide concentration on plasticity, microhardness and softening temperature interrelationship, energy of metal atoms in chalcogenide glass system (1-x)(0.27Sb_2Se_3–0.73$GeSe_2$)-$x$$Ag_2Se$ is presented. Particular attention is paid to a multiple increase in plasticity with an increase in silver selenide content in chalcogenide glass. The observed effects are associated with the formation of metallophilic interactions of silver-silver. The studies are supplemented with the results of impedancemetry, due to the fact that metallophilic interactions in chalcogenide glass can actively influence not only the glass transition temperature, but also many other important properties, including the mechanism of electronic and ion conductivity.

Keywords: *chalcogenide glass, silver selenide, ionic conductivity, plasticity, metallophilic bonds Ag-Ag, glass mesh*

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INTRODUCTION

Flexible electronics are predicted to revolutionize the electronics industry of the 21st century, so it is not surprising that this area is currently developing very intensively [1–3]. Such rapid changes are facilitated not only by the convenience of using flexible devices in technology, but also by the possibility of placing various sensors based on them directly on controlled mobile objects, for example, on the human body or clothing [4, 5]. In practice, all components of flexible electronic devices, including functional components, must withstand repeated mechanical deformations in order to ensure structural and functional integrity, including at above room temperatures [6–8].

It should be noted that most of the known inorganic semiconductors are brittle at room temperature [9–11], and are poorly suited for flexible electronics purposes. Therefore, the discovery of the plasticity of the Ag_2S semiconductor at room temperature [12–14] is a breakthrough in solving this long-standing dilemma between mechanical deformability and electrical characteristics and may open up wide opportunities for more intensive development of flexible electronics [15–17].

Semiconductor materials that are currently used in flexible electronics can be divided into three main groups: inorganic nanocrystalline [18–20], inorganic amorphous [21–23] and organic [24–26]. The group of inorganic nanocrystalline semiconductor materials has relatively high stability of functional properties, but low flexibility and plasticity. Organic semiconductors are quite plastic, but their properties degrade relatively quickly. Inorganic amorphous semiconductors, represented mainly by amorphous silicon, occupy an intermediate position in this series, but their electronic properties are not well-regulated. A separate group of promising materials used in flexible electronics could be plastic glasslike materials [27–29], which however as a rule, are characterized by high brittleness, which causes their low resistance to mechanical stress and temperature fluctuations [30].

Nevertheless, chalcogenide glasses with a high silver content have been found not only to have significant ionic conductivity over silver, but also, as previously shown [31–33], to exhibit increased plasticity compared to other chalcogenide glasses. The latter is related to the ability of silver atoms to form non-directional metallophilic bonds at high concentrations in the glass mesh.

On the other hand, it is known that glasses have a number of advantages compared to crystals:

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[1] Based on the materials of the report at the 17th International Meeting “Fundamental and applied problems of solid state ionics”, Chernogolovka, June 16–23, 2024.

their properties change smoothly with changes in composition, ionic conductivity is higher than that of a crystal of the same composition. As disordered systems they are less susceptible to impurities, modern glass technologies make it possible to manufacture products of almost any shape, etc. [34, 35].

Nevertheless, it is known that plastic crystalline inorganic semiconductors based on Ag_2S, Ag_2Se, and Ag_2Te [36–38], which relate to solid electrolytes and are currently being intensively studied for these purposes, are one of the materials in demand for creating flexible sensors.

Summarizing the above, we can assume that glass capable of including at least 20 mol% silver chalcogenide in their composition without loss of glass-forming ability, may be promising materials used in flexible electronics. These observations may lead to an interest in Ag_2Se-based glass.

In [32], the glass transition temperature of chalcogenide glasses with ionic conductivity was analyzed depending on the content of silver chalcogenides. Here, the peculiarities of the change in the glass transition temperature of a silver chalcogenide-based material are explained by the coexistence of covalent silver–chalcogen (Ag–Ch) bonds and metallophilic silver-silver (Ag–Ag) bonds. When assessing the degree of constraint of a chalcogenide glass grid, it is traditionally assumed that the number of bonds formed by each atom coincides with its degree of oxidation. However, the analysis showed that a large number of studied chalcogenide glass-forming systems containing silver demonstrate a common relationship, which allows us to conclude that the silver bonding coefficient in chalcogenide glass significantly exceeds its formal degree of oxidation, since silver forms metallophilic bonds in addition to covalent ones.

Expecting that metallophilic interactions affect not only the glass transition temperature, but also many other important properties of these glasses, including ionic conductivity, the authors measured the conductivity as a function of the concentration of silver selenide in the glass of the $(1\text{-}x)(0{,}27Sb_2Se_3–0{,}73GeSe_2)\text{-}xAg_2Se$ system.

The choice of this system for research was determined by the following considerations.

The authors of [39], examining glass of the $Ag_2Se–Sb_2Se_3–GeSe$ system, convincingly confirmed the assumption of high silver coordination in chalcogenide glass, leading to a corresponding change in the properties of the glass, in particular the softening temperature T_g. According to these data, the replacement of Sb_2Se_3 with Ag_2Se leads to a significant increase in T_g.

Considering that the introduction of Ag_2Se into the glass of the $Sb_2Se_3–GeSe_2$ system leads to a significant increase in plasticity [32], the authors of this article paid increased attention to the study of this system.

In addition, the concept of the existence of metallophilic bonds in chalcogenide glass is new and therefore needs comprehensive consideration and additional justification.

According to our data [33] obtained for glass of the $Ag_2Se\text{-}As_2Se_3(Sb_2Se_3)\text{-}GeSe_2$ system, T_g does not decrease even when trivalent metal selenides are replaced by Ag_2Se.

Considering in more detail the data we obtained in [33], it can be assumed that $GeSe_2$, as a compound containing a metal with a maximum coordination number in the glass-forming system under study, will mainly determine the value of T_g. It was also concluded that Ag_2Se in the composition of the studied glasses has the same effect on the value of T_g as the trivalent metal selenides As and Sb. This is consistent with the conclusion of the authors [40] that the average atomic coordination in glasslike nanodomains of the Ag_2Se composition is close to 2.4.

In [31], the dependence of the plasticity of the $(1\text{-}x)(0.27Sb_2Se_3–0.73GeSe_2)\text{-}xAg_2Se$ system glass synthesized by us on the Ag_2Se concentration calculated using the Milman ratio [41] is presented. The observed increase in plasticity can contribute to a significant improvement in functional properties, in particular, improved resistance to temperature fluctuations and good prospects for use in flexible electronics.

Thus, based on the presented brief overview of glass, the authors selected the $(1\text{-}x)(0.27Sb_2Se_3–0.73GeSe_2)\text{-}xAg_2Se$ system for research, which exhibits high plasticity and is promising for use in flexible electronics.

The purpose of this work is to develop the concept of metallophilic interactions of silver atoms in chalcogenide glass, to study their effect on the energy of interatomic interactions by the XPS method and on ion transport, according to impedance spectroscopy. These results are discussed in conjunction with experimental results on the softening temperature and plasticity of glasses.

MATERIALS AND METHODS

Synthesis of glass (GS). Chalcogenide glass was synthesized from simple substances and contained the following amounts of the main component: Sb (99.995%), Se (99.997%), Ag (99.990%) and Ge (99.999%) according to the procedure presented in [42]. The following sample compositions were synthesized with the appropriate numbering: (0) $x = 0.00$; (1) $x =$

0.05; (2) $x = 0.10$; (3) $x = 0.15$; (4) $x = 0.20$; (5) $x = 0.25$; (6) $x = 0.30$; (7) $x = 0.35$; and (8) $x = 0.40$; (9) $x = 0.45$. For each case, the components of the appropriate composition were placed in quartz ampoules, which were then pumped out to a pressure of 10^{-4} mm Hg and sealed. This ensured the consistency of the glass composition during the manufacturing process. For all compositions, synthesis was carried out in a muffle furnace at a temperature of 900 °C for 3 hours with constant stirring. In order to increase the cooling rate, ampoules were placed in ice water immediately after synthesis. All the research methods used, with the exception of impedance spectroscopy, do not impose special requirements to the shape of the samples. The samples for impedance spectroscopy were annealed and then processed to obtain plane-parallel plates with a thickness of 3 mm.

X-ray structure analysis (XRD). X-ray phase analysis of all synthesized glasses was performed using the method presented in [42] using an ARL X'TRA diffractometer in the θ—2θ scanning mode (CuKα radiation, $\lambda = 1.541$ Å) in the angle range $2\theta = 20°—60°$, with a scanning step of 0.04° and an exposure time of 2 s per point. This technique, despite the controversial opinion regarding its usefulness for studying the structure of glass, has long been in the field of view of researchers [43, 44].

Differential scanning calorimetry (DSC). A high-sensitivity Netzsch DSC 204 F1 Phoenix differential scanning calorimeter with a μ-sensor was used to measure the T_g value of the glass [45]. The concept of the measuring chamber is based on homogeneous heating of the disk μ-sensor in order to obtain a stable and reproducible baseline, and an efficient cooling system. The glass of the studied compositions was pre-crushed in an agate mortar and placed in an aluminum crucible. The analysis was carried out in the temperature range 30—350 °C, and the heating rate was 10 degrees/min.

Together with high temperature accuracy, the μ-sensor provides a high level of sensitivity, which was previously unattainable in calorimetry.

X-ray photoelectron spectroscopy (XPS). The method of photoelectron spectroscopy based on the phenomenon of the photoeffect is a modern method for studying filled electronic states in a solid and is able to provide additional information about the properties of the studied glass [46].

To measure the glass of the studied compositions using the XPS method, the analytical module of photoelectron spectroscopy of the Nanolab platform and the Thermo Fisher Scientific Escalab 250Xi integrated photoelectronic and scanning Auger electron spectrometer were used.

Impedance spectroscopy (IS). The glass of the studied compositions was measured by impedance spectroscopy using an Elins Z-1000P impedance meter (Elins LLC, Russia) in a two-contact cell with reversible Ag electrodes in the frequency range of 1—106 Hz [47]. The glass samples in the form of a cube with a side size of 3 mm were carefully polished. A silver paste was applied to the opposite faces of all the glass items as a reversible electrode. All measured impedance hodographs were processed using a special ZView program and the Origin graphics software package from OriginLab Corporation. The resistance of the sample was determined based on the data obtained and electrical conductivity was calculated.

RESULTS AND THEIR DISCUSSION

The introduction of monovalent metal compounds into chalcogenide glass leads to a decrease in the average number of bonds per atom. This reduces the degree of constraint of the glass grid. The result is a rapid decrease in T_g. When the concentration of Ag_2Se increases to 20 mol there is a possibility that silver atoms will form not only covalent bonds with selenium, but also metallophilic interactions with each other. This leads to a significant slowdown in the decrease in T_g. The result is that glass containing 40 mol.% Ag_2Se has T_g=200 °C. This is more than the softening temperature that such classical chalcogenide glass as As_2S_3 and As_2Se_3 has.

Synthesis of glass. All synthesized glass samples had a black color and a typical conchoidal fracture.

X-ray phase analysis. According to the results of X-ray phase analysis of all synthesized glass, diffractogram systems contain exceptionally wide amorphous peaks (halos) characteristic of glasslike materials, which confirms the absence of crystalline inclusions. The exception is the 9th sample, which contains, according to the XRD data, crystalline inclusions.

Differential scanning calorimetry (DSC). Fig. 1 shows the measured DSC curve of a glass sample having a composition of x=0.30. The glass softening effect is characterized by a good resolution, which makes it possible (as shown in Fig. 1) to analyze the concentration dependences of not only the glass softening temperature (T_g) itself.

Fig. 2 shows the dependence of the glass transition temperature on the concentration of Ag_2Se according to DSC data. The T_g value of Ag_2Se-free glass is 280 °C and is in satisfactory agreement with the literature data [48].

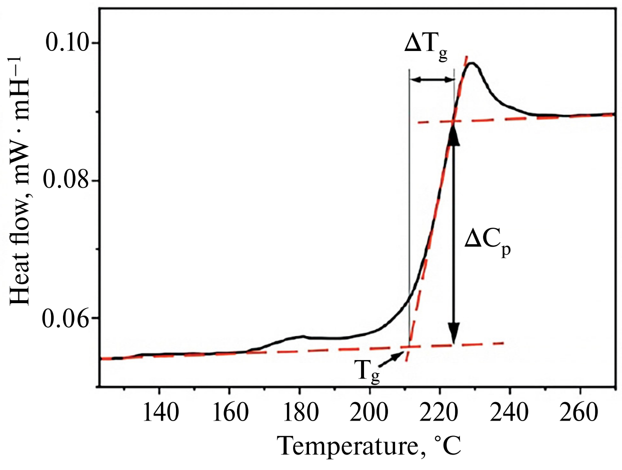

Fig. 1. DSC curve of a glass sample with x=0.30, showing how to determine the values of T_g, ΔC_p and ΔT_g based on the graph.

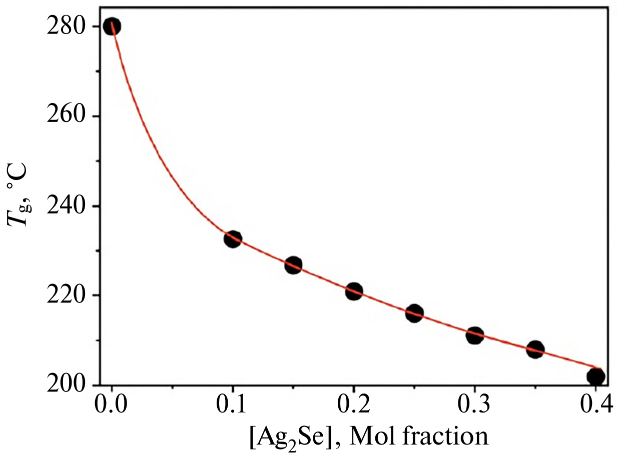

Fig. 2. Dependence of the glass transition temperature, determined by the DSC method for the glass of the studied system, on the concentration of Ag_2Se.

Photoelectron spectroscopy. The XPS of all elements (with the exception of Ag spectra) consists of several lines. Therefore, after deconvolution of the spectrum, the concentration dependence of the weighted average binding energy of the entire spectrum for each element was calculated using the formula:

$$E_s = \frac{\sum_i E_i \cdot A_i}{\sum_i A_i},$$

where E_i is the position of the maximum of the i–th band of the spectrum; A_i is its integral relative intensity.

The obtained dependences of the binding energy (E_S) of each element included in the glass on the concentration of Ag_2Se are shown in Fig. 3.

According to the presented results, the following assumptions can be made.

The Sb binding energy (Fig. 3*a*) is practically independent of the glass composition. The binding energy of the remaining elements (Fig. 3*b*–3*d*) increases with the increasing silver content. The ratio of concentrations of antimony and germanium selenides does not change. Therefore, it can be assumed that the changes are associated with an increase in the content of Ag_2Se.

XRF did not detect crystalline inclusions in glasses with $x \leqslant 0.4$. At x=0.45, crystalline inclusions of the compound Ag_8GeSe_6 appear. Therefore, it can be assumed that the growth of E_s for Ag, Ge, and Se is the result of the formation of triple structural units of the specified compound in the glass grid. However, the $GeSe_2$ content in glass with x=0 is high (73 mol.%). Therefore, for the first Ag_2Se additives, there are no difficulties in forming the structural units of the Ag_8GeSe_6 compound and E_s^{Ag} should not depend on the composition. On the other hand, E_s^{Ge} should grow linearly with an increase in the Ag_2Se content. The observed changes in E_s for Ag, Ge, and Se have a zero derivative at low Ag_2Se concentrations. This means that the effect is described by a power-law dependence on the content of Ag_2Se with a degree index of at least 2. Metallophilic Ag-Ag interactions satisfy this requirement.

Thus, E_s^{Ag} increases with the increasing silver content due to metallophilic Ag-Ag interactions. According to the induction mechanism, this growth extends to the elements located in the first and second coordination spheres of silver. These elements include selenium and germanium. Antimony is not one of them, since structural units of compounds based on Ag, Ge, and Se, which do not include Sb, are formed in glass. This compound is released into an independent crystalline phase during crystallization of alloys as a result of exceeding the critical content of Ag_2Se.

Impedance spectroscopy. The measured dependences of the imaginary part of the impedance Z'' on the real Z' have a typical form for solid electrolytes (Fig. 4).

Based on this, the obtained experimental values of resistivity were attributed to ionic conductivity.

According to the results of impedance spectroscopy, the dependence of resistivity on the Ag_2Se content in glass (x) was found. According to the equations proposed to describe the transport of singly charged cations in chalcogenide glasses [49, 50]: $\lg R \sim -\sqrt[3]{M}$ (where R is the resistivity, M is the atomic fraction of the singly charged cation). The experimental data plotted in these coordinates shown in Fig. 5 demonstrate the observance of this relationship.

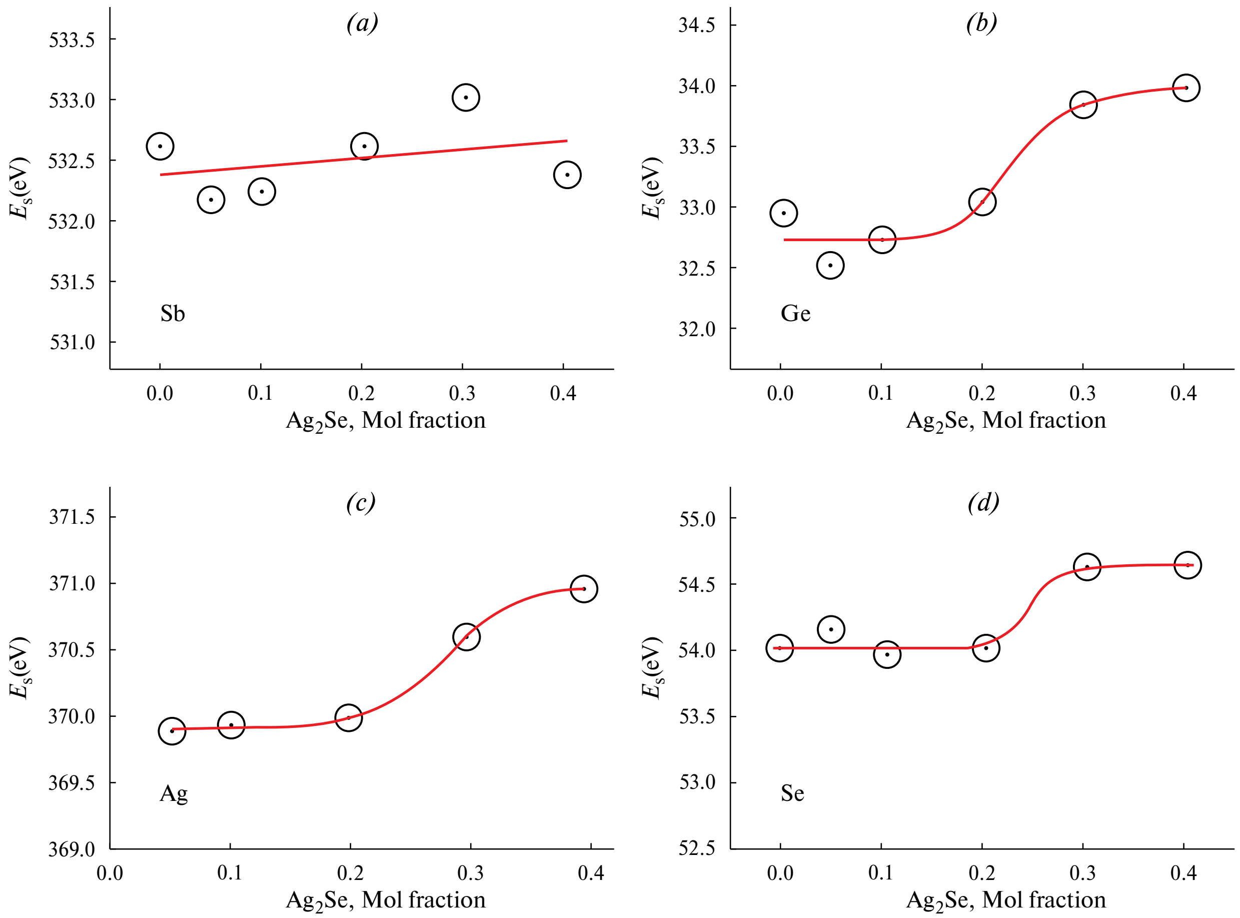

Fig. 3. Dependence of the weighted average binding energy of each element in the glass of the (1-x)(0.27Sb_2Se_3–0.73$GeSe_2$)-$x$$Ag_2Se$ system on the concentration of Ag_2Se.

Moreover, extrapolation of the found dependence to the silver concentration corresponding to the Ag_2Se composition gives a resistivity value of 54 ohms cm. Considering that we are not talking about a crystalline compound of the specified composition, but about hypothetical glass, this resistivity value can be considered reasonable.

The obtained impedance hodographs are semicircles, the center of which is located below the axis of the abscissa. The reason for this, as is known, is the fluctuation spread of the values of the characteristic time τ describing the corresponding hodograph (the inverse of the voltage frequency corresponding to the maximum value of the imaginary part of the hodograph). The wider the τ spread, the lower the center of the circle of the hodograph is located. The fluctuating nature is characteristic of the glasslike state.

Fluctuations τ can be compared to the fluctuation of potential barriers overcome by cations during their migration. The magnitude of these frozen fluctuations is determined by the glass formation temperature (T_g). For the studied glasses with x varying from 0.2 to 0.4, the deviation of T_g from its average value in this concentration range is ± 2%. This allows us to consider the fluctuation in the height of potential barriers as a constant value. The height of potential barriers itself, depending on the concentration of Ag_2Se, varies significantly, leading to a considerable change in the logarithm of resistivity. From this it can be concluded that the relative role of τ fluctuations will increase with an increase in the concentration of Ag_2Se and a decrease in R.

The parameter that numerically characterizes the displacement of the center of the circle (and, accordingly, the fluctuation τ), regardless of the resistivity value, is the tangent of the angle (tgα) formed by the abscissa and the radius of the circle drawn to the point of its intersection with the abscissa.

Fig. 6 shows the dependence of the logarithm tgα on the logarithm of resistivity.

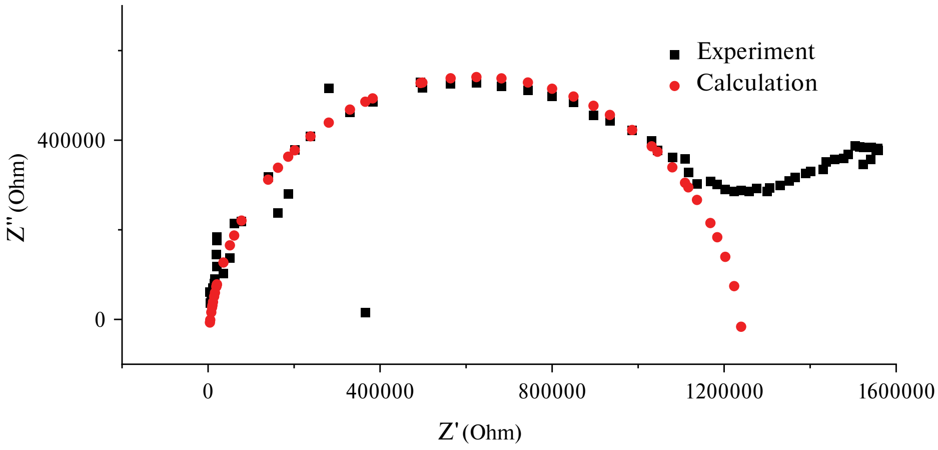

Fig. 4. Dependence of the imaginary part of the impedance on the real one for glass with x=0.4. Black dots are experimental data; red dots are the result of approximation.

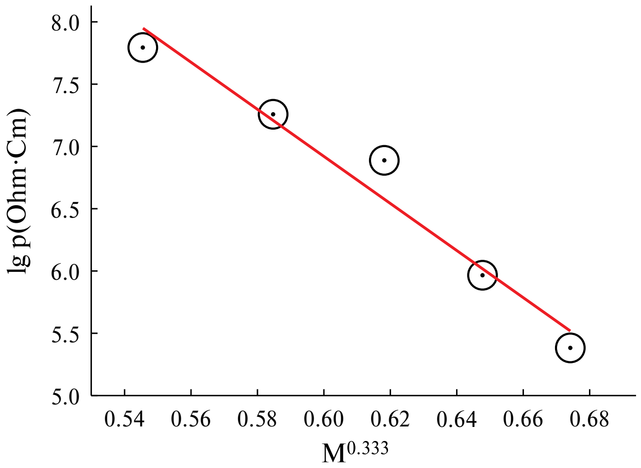

Fig. 5. Dependence of the logarithm of the resistivity of the glass of the (1-x)(0,27Sb_2Se_3–0,73$GeSe_2$)-$x$$Ag_2Se$ system on the cubic root of the atomic fraction of silver.

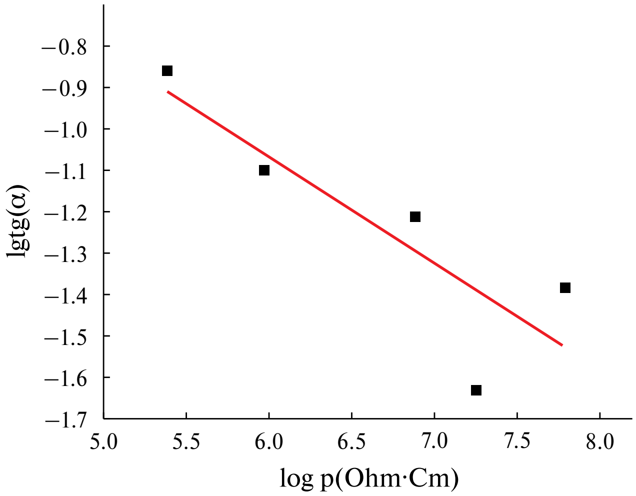

Fig. 6. Dependence of the logarithm tgα on the logarithm of the resistivity of the glass of the (1-x)(0,27Sb_2Se_3–0,73$GeSe_2$)-$x$$Ag_2Se$ system.

As expected, the increase in the resistivity of glass is accompanied by a decrease in the role of the fluctuation spread of the characteristic times of the hodographs.

CONCLUSION

The experimental results obtained and, in particular, the threshold character of the increase in the binding energy of Ge, Se, and Ag with an increase in the content of the latter confirm the formation of metallophilic Ag-Ag bonds in the glass of the (1-x)(0,27Sb_2Se_3–0,73$GeSe_2$)-$x$$Ag_2Se$ system at an Ag_2Se concentration above 20 mol.%. However, it was not possible to detect any features in the behavior of the concentration dependence of ion conductivity. Apparently, this is due to the following circumstances. Ag_2Se, chalcogenide glasses in general, including the glasses of the studied system, are formed by covalent metal-chalcogen chemical bonds with an ionic content of less than 10%. Due to this, there is no strong Coulomb repulsion between silver atoms, which makes it possible for them to converge and form a metallophilic interaction. For ion transfer, the Ag atom must acquire an electric charge, which will lead to the decay of the metallophilic interaction. Thus, the metallophilic interaction has no effect on the movement of Ag ions. However, its contribution to the process of transition of silver atoms from a covalently bonded state with a minimum effective charge to a state with an integer charge involved in ion transport cannot be excluded.

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CONFLICT OF INTERESTS

The authors declare that they have no conflict of interest.

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