# OBTAINING AND MEASURING IMPEDANCE CHARACTERISTICS OF THE Ag<sub>8</sub>SiSe<sub>6</sub> COMPOUND

R.M. Sardarly<sup>a,b</sup>
M.B. Babanly<sup>c</sup>
N.A. Aliyeva<sup>a,d\*</sup>
L.F. Mashadiyeva<sup>b</sup>
R.A. Mamadov<sup>a</sup>
G.M. Ashirov<sup>b</sup>
A.A. Saddinova<sup>e</sup>
S.Z. Damirova<sup>e</sup>

<sup>a</sup>Institute of Radiation Problems, Azerbaijan National Academy of Sciences, B. Vagabzadeh street 9, AZ 1143, Baku, Azerbaijan

<sup>b</sup>Department of Applied and General Physics, National Aviation Academy, Baku, Azerbaijan

<sup>c</sup>Named after Academician M. Naghiyev Institute of Catalysis and Inorganic Chemistry, Ministry of Science and Education Republic of Azerbaijan, H. Cavid str., 113, AZ1143, Baku, Azerbaijan

<sup>d</sup>Azerbaijan University of Architecture and Construction, Ayna Sultanova st. 5, AZ-1073, Baku, Azerbaijan

<sup>e</sup>Institute of Physics, Ministry of Science and Education Republic of Azerbaijan, H. Javid str., 131, AZ1143, Baku, Azerbaijan

\*Corresponding Author e-mail: gunel imanova55@mail.ru

Received July 23, 2024; revised October 13, 2024; accepted January 9, 2025

Samples of the Ag<sub>8</sub>SiSe<sub>6</sub> compound were obtained by direct alloying of initial components and subsequent pressing of powders under a pressure of 0.7 GPa. Temperature dependences of conductivity ( $\sigma$ (T)) and permittivity ( $\epsilon$ (T)) of the Ag<sub>8</sub>SiSe<sub>6</sub> compound were studied in constant and alternating electric fields. In a constant field, the electrical conductivity is found to decrease over time ( $\sigma$ (t)). This phenomenon is associated with the process of charge accumulation at the interface of the ion sample and the blocking electrode. Complex impedance spectra were studied in the frequency range of 20–10<sup>6</sup> Hz.

Keywords: Compound; Ionic conductivity; Complex impedance spectra

PACS: 71.20.-b, 71.20.Nr, 71.70.Ch

## 1. INTRODUCTION

The development of modern materials for micro- and nanoelectronics, super capacitors, ionic conductors, minibatteries and other devices depends on the search for new compounds that have the appropriate crystal structure, electronic and phonon spectrum, which makes it possible to implement these devices. Promising materials for these purposes include compounds of the mineral argyrodite (Ag<sub>8</sub>GeS<sub>6</sub>) family and phases based on them with the general formula  $A_{(12-n)/m}^{m+}B^{n+}X_6^{2-}$ , where *m* and *n* are the valences of *A* and *B* cations, respectively (where *A*-cations are Cu<sup>+</sup>, Ag<sup>+</sup>, Li<sup>+</sup>, Cd<sup>2+</sup>, Hg<sup>2+</sup>; *B*-cations are Ga<sup>3+</sup>, Si<sup>4+</sup>, Ge<sup>4+</sup>, Sn<sup>4+</sup>, P<sup>5+</sup>, As<sup>5+</sup>; *X*-anions are S<sup>2-</sup>, Se<sup>2</sup>, Te<sup>2-</sup>. These phases have a number of valuable functional properties and are being intensively studied by many research groups [1-6]. Most of these materials are of special interest due to their phase transitions, which take place close to ambient temperature [7]. High-temperature modifications, as a rule, crystallize in a cubic structure and have high ionic conductivity. Especially, copper and silver containing compounds of this family are well-known super ionic semiconductors, due to the presence of highly mobile Cu+ and Ag+ ions [8-12]. In recent years, silicon-containing argyrodites, have attracted particular interest as eco-friendly thermoelectric and ion-conducting materials [13-15].

Previously, in [16], we studied the temperature dependences of the conductivity and permittivity of the  $Ag_8GeSe_6$  compound in direct and alternating electric fields. It was shown that this phenomenon is associated with the process of charge accumulation at the interface of the sample with ionic conductivity and the blocking electrode and the formation of a double electric layer. The observed in the studied sample dielectric relaxation is associated with the transition of the crystal to the super ionic state. It has been established that hopping conduction with a variable hopping length over localized states near the Fermi level takes place in an  $Ag_8GeSe_6$  crystal.

The purpose of this work was to establish the features of the temperature and frequency dependence of conductivity, dielectric properties of Ag<sub>8</sub>SiSe<sub>6</sub> compound in a constant and alternating field. The impedance spectra of the Ag<sub>8</sub>SiSe<sub>6</sub> compound were studied in order to establish the mechanism of charge transfer and the nature of relaxation losses in the volume and at the interface between the ionic conductors and the blocking contact.

The Ag<sub>8</sub>SiSe<sub>6</sub> melts congruently at 1203 K [7, 17], or 1258 K according to [18], 1268 K according to [19]. This compound has two polymorph transitions at 315 and 355 K and crystallize in 3 crystalline structures [7, 11, 13, 17]. The high-temperature (HT) modification of the Ag<sub>8</sub>SiSe<sub>6</sub>, like all other compounds of the argyrodite family, crystallizes in a face-centered cubic structure (Sp.gr. *F*-43*m*) with complete disordering of the cationic sublattice. HT-Ag<sub>8</sub>SiSe<sub>6</sub> has the lattice periods: a = 1.097 nm [7, 17], or a = 1.09413(1) nm [13]. The intermediate modification (IT) has a simple-cubic structure (Sp.gr. *P*2<sub>1</sub>3 [11] or *P*4<sub>2</sub>32 with a lattice constant a = 1.087 nm [7]). The IT-Ag<sub>8</sub>SiSe<sub>6</sub> has a tetragonal structure (Sp.gr. *I*-4*m*2, a = 0.7706, b = 1.10141 nm).

Cite as: R.M. Sardarly, M.B. Babanly, N.A. Aliyeva, L.F. Mashadiyeva, R.A. Mamadov, G.M. Ashirov, A.A. Saddinova, S.Z. Damirova, East Eur. J. Phys. 1, 233 (2025), https://doi.org/10.26565/2312-4334-2025-1-24

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### 2. EXPERIMENTAL METHODS AND OBTAINING SAMPLES

High-purity elements (at least 99.999 wt.% purity) were used for synthesis of  $Ag_8SiSe_6$  compound. The synthesis was carried out in ampoules evacuated to  $10^{-2}$  Pa. To avoid a reaction between silicon and the walls of the quartz ampoule, the inner walls of the tube were graphitized by pyrolysis of toluene.

Taking into account the high vapor pressure of selenium at the melting point of Ag<sub>8</sub>SiSe<sub>6</sub>, this compound was synthesized in an inclined two-zone furnace. Stoichiometric amounts of elementary components were loaded into a quartz ampoule, which, after being evacuated and sealed, was placed in an inclined tube furnace. Two-thirds of the ampoule was in the oven (hot zone) and the rest of the upper part was outside the oven (cold zone). The furnace was heated to 1300 K. To prevent the ampoule from exploding due to overheating, the "cold" zone was cooled with water. After the interaction of a larger mass of components, the ampoule was completely placed in the furnace. After keeping in the oven for 4-5 hours, the ampoule was gradually cooled by in the switched off furnace mode.

The completion of the synthesis of the  $Ag_8SiSe_6$  and the identification of this compound were controlled by differential thermal analysis (DTA) and X-ray diffraction (XRD) technique.

Figure 1 presents the DTA data for the Ag<sub>8</sub>SiSe<sub>6</sub>. This compound melts at 1278 K (this temperature closer to the data Piskach L.V. et al [19]). There are also two endothermic effects at 315 and 354 K on the heating DTA curve, corresponding to the polymorphic transitions.



Figure 1. Heating DTA curve for the Ag<sub>8</sub>SiSe<sub>6</sub>

The diffraction pattern of Ag<sub>8</sub>SiSe<sub>6</sub> is completely indexed in a simple-cubic lattice with Sp.gr.  $P4_232$  and a = 1.0891(3) nm. Obtained crystal lattice parameters almost coincided with the data of the above works [7, 11]. DTA measurements were recorded using a multichannel device based on the electronic "TC-08 thermocouple data logger" with chromel-alumel thermocuples in sealed quartz tubes. XRD was performed at room temperature using a Bruker D8 ADVANCE diffractometer (with Cu-K $\alpha_1$  radiation) within  $2\theta = 10^\circ$  to 70°. The unit cell parameters were calculated by indexing powder patterns using Topas V3.0 software.

In order to measure the temperature dependences of the permittivity and electrical conductivity of Ag<sub>8</sub>SiSe<sub>6</sub> compound samples, capacitors were made, the dielectric in which was plates of the studied materials, which were compressed tablets from the powdered Ag<sub>8</sub>SiSe<sub>6</sub> compound. The capacitor plates were obtained by applying silver conductive paste to the surface of the plates. The study of complex permittivity and electrical conductivity was carried out with a digital immittance meter E7-20 in the frequency range of  $20\div10^6$  Hz in the temperature range of  $200\div450$  K. The amplitude of the measuring field was in the range of 1-5 V·cm<sup>-1</sup>.

# 3. Results and discussion 3.1 Temperature dependence of electrical conductivity and permittivity a. Electrical conductivity

The temperature dependence of the electrical conductivity of the Ag<sub>8</sub>SiSe<sub>6</sub> compound in a constant field is given in Fig. 2. As can be seen from the figure, at a temperature of ~300 K, there is a leap in the dependence  $\sigma(T)$ . The inset to the figure shows the curve ln( $\sigma$ ·T) on 1/T above the temperature leap as a function of  $\sigma$  on 1/T. According to [20-22], the temperature dependence of ionic conductivity has an activation character

$$\sigma \cdot \mathbf{T} = \sigma_0 \exp(-\Delta \mathbf{E}/\mathbf{kT})$$

where,  $\Delta E$  is the activation energy of electrical conductivity. As can be seen from the inset to Fig. 1, the experimental points are well straightened, while  $\Delta E$  – for Ag<sub>8</sub>SiSe<sub>6</sub> turned out to be equal to 0.1 eV. It is known that this type of electrical conductivity indicates the predominant nature of ionic conductivity above the temperature leap in the  $\sigma(T)$  curve.

The observed sharp increase in the conductivity in  $Ag_8SiSe_6$  compound at temperatures above 300 K is probably due to the increase in the number of ions in states where they have high mobility. From crystallographic considerations, we can assume that the structure of  $Ag_8SiSe_6$  is most favorable to the mobility of  $Ag^{+1}$  cations, that is, the temperature increase in conductivity is due to the diffusion of  $Ag^{+1}$  ions through vacancies in the silver sublattice of the  $Ag_8SiSe_6$  compound.



Figure 2. Temperature dependences of the electrical conductivity  $\sigma(T)$  of the AgsSiSe<sub>6</sub> compound in a constant field. The inset to the figure shows the dependence of ln( $\sigma$ ·T) on 1/T above the temperature leap as a function of  $\sigma$  on 1/T, that is, in the region of ionic conductivity

It is known that with the transition of the system to a state with predominantly ionic conductivity, the disorder of the cationic sublattice in the compound will increase, that is, the ions will shift from their positions at the lattice sites to the interstitial site, leaving a vacancy at the site. Collective interaction of all interstitial ions and vacancies can lead to the emergence of superionic conductivity. It is also possible that the migration of interstitial  $Ag^+$  ion occurs by direct leap from one interstitial site to another, followed by displacement of translationally equivalent ion (Ag) in the interstitial site. In this case, the most probable type of emerging defects will be Frenkel defects, namely, mobile interstitial  $Ag^+$  ions associated with cation vacancies. The conductivity of the  $Ag_8SiSe_6$  compound in the superionic phase at T>300K is ~ 3·10<sup>-3</sup> Ohm<sup>-1</sup>cm<sup>-1</sup>, the activation energy of conductivity is 0.1 eV. The structure is favourable for the free motion of  $Ag^+$ .

In this case, there will also be a charge exchange between the defects in the crystal and the resulting local polarization will lead to an increase in permittivity [23-28].

# **b.** Permittivity

The electrical conductivity of compounds in the superionic phase is exponential in nature [29], while the temperature dependence of the permittivity is also exponential.



**Figure 3.** Temperature dependence of permittivity of Ag<sub>8</sub>SiSe<sub>6</sub> compound. Measurements were performed at different frequencies of the measuring field:  $1 - 10^3$ ,  $2 - 10^4$ ,  $3 - 10^5$ ,  $4 - 5 \cdot 10^5$ ,  $5 - 10^6$  Hz

Fig. 3 shows temperature dependences of permittivity of  $Ag_8SiSe_6$  compound. The measurements were performed in the temperature range of 200-400 K and frequency range of  $10^2 - 10^6$  Hz. The figure shows that both with increasing temperature and with increasing frequency of the measuring field, the peak value of permittivity drops and it shifts towards high temperatures. In all measured frequency and temperature ranges, the same feature is observed in dependence  $\varepsilon(T)$ . Such behavior of temperature dependence of permittivity is typical for Debye-type relaxation processes in which several relaxators participate. The shift of the peak  $\varepsilon(T)$  to the high-temperature region with increasing measurement frequency may indicate that polarization processes occurring in the volume begin to play a significant role in the relaxation processes occurring at the interface. The polarization of  $Ag_8SiSe_6$  may be due to the diffusion of  $Ag^{+1}$  ions over vacancies, which leads to the transition of the compound to the superionic state In addition, the exponential growth of the  $\varepsilon(T)$  dependence measured in the low temperature region (i.e., at temperatures preceding the transition to the superionic state) can be explained by the influence of localized states on polarization in an alternating electric field. Polarization can be related to the hopping of charge carriers to the positions that are energetically favorable due to the creation of an electric field in the crystal, and due to lattice relaxation, there is a shift of charges, which is accompanied by the screening of the resulting polarization [23-25].

# 3.2. Time dependence of electrical conductivity

The feature of charge transfer of ionic conductors is a large contribution to the conductivity of ions in comparison with electron transfer. The ionic conductivity of high resistivity crystals and semiconductors, under normal conditions, is 6-7 orders of magnitude lower than the contribution of electron transfer to the overall conductivity of the sample. The Wagner polarization method was used to elucidate the relative contribution of the electronic and ionic component to the overall conductivity of  $Ag_8SiSe_6$  samples at different temperatures. In compounds with a mixed electron-ion nature of charge transfer, a time dependence of the conductivity is observed when the conductivity is measured at direct current. The process is associated with the formation of a double electric layer in the near-surface layer of the sample. When blocking contacts are used as electrodes, mobile ions are delayed at the sample-electrode interface; as a result, a concentration gradient is created in the sample volume under the influence of the electric field, which in turn leads to the emergence of a diffusion flux of ion directed in the opposite direction to the drift flux of ion. Thus, since the drift and diffusion fluxes of ion are compensated, a current flows through the sample due to charge transfer by electrons only.

Fig. 4 shows the time dependence of the conductivity of the electrochemical cell of  $Ag_8SiSe_6$  compound. Silver contacts blocking ionic contribution to the conductivity are used as electrodes. A constant potential difference of ~ 5V/cm is applied to the plates. The current through the sample is measured at certain time intervals.

As can be seen from the figure, in a constant field, the time dependence of electrical conductivity is nonlinear. As a result, during measurements at the initial moment of time, the total current of ions and electrons flows, while in the steady state the charge transfer is carried out only by electrons. Thus, the result of mutual compensation of the space charge region near the interface (Ag<sub>8</sub>SiSe<sub>6</sub>) - silver contact, which has a blocking character, is a decrease of the electric current over time in a constant field (Fig. 4)



**Figure 4.** Time dependence of the reduced electrical conductivity  $J(t)/J_0$  for Ag<sub>8</sub>SiSe<sub>6</sub> compound. The measurements were performed using silver electrodes at room temperature. 1-300K, 2-350K

# 3.3. Impedance

The nature of electrical conductivity of samples with mixed electron-ion charge transfer is affected by relaxation processes at the interface, which lead to the blocking of charge carriers through the contact and polarization of the nearcontact region. To obtain a complete picture of the conductivity of the studied sample, we have measured the complex impedance with subsequent construction of hodographs in the Cole-Cole representation.

The frequency dependence of the real Z'(v) and imaginary Z''(v) parts of the complex conductivity of Ag<sub>8</sub>SiSe<sub>6</sub> is presented in Fig. 5. The measurements were carried out at temperatures of 200, 250, 300, 350 and 400 K. As can be seen from the figures, there is a smooth decrease in the values of the real and imaginary parts of the complex impedance with increasing operating frequency. Frequency dispersion is observed in all experimental conditions, but the nature of the dispersion is different for each measurement. A sharp decrease in Z'(v) and Z''(v) is observed in the low-frequency frequency range of 20-10<sup>3</sup> Hz, however with further increase in frequency, there is a weakening of the frequency dependence of the spectra. The observed dispersion of impedance spectra indicates the presence of electrical relaxation, which can be conveniently analyzed using impedance hodograph curves.

Impedance spectroscopy methods are widely used in measuring the dielectric parameters of solids using alternating current. This method allows to obtain the most complete information both on the relaxation processes occurring in the

thickness of the object and on the characteristics of complex conductivity at its boundaries, as well as to determine the nature of relaxation losses.



Figure 5. Frequency dependences of the real Z'(v) and imaginary Z''(v) parts of the complex impedance of the Ag<sub>8</sub>SiSe<sub>6</sub> compound. The measurements were carried out at *a* - 200, *b* - 250, *c* - 300, *d* - 350 and *e* - 400 K

Fig. 6 shows the results of dependence of Z''(v)/Z'(v) at temperatures of 200, 250, 300, 350 and 400 K for Ag<sub>8</sub>SiSe<sub>6</sub> compound. As can be seen from the curves in Fig. 6, the diagrams in the complex plane (Z" - Z") are semicircles (curves in Figs. *a* and *b*), while in Figs. *d* and *e*, measured at temperatures of 350 and 400 K, "rays" appear in addition to the semicircles. These rays in the low-frequency region of the hodograph obtained at 350 and 400 K are characteristic of the Warburg impedance. The idea of the Warburg impedance is based on the fact that the diffusion of carriers in the frequency range of the applied sinusoidal signal does not reach the boundary of the diffuse layer. The reason for the appearance of the diffuse Warburg impedance circuit is shown in Fig. 6, it has  $Z_1 = R_1$  - active resistance and  $Z_2 = 1/j\omega C$  - the capacitance C (j is the imaginary unit,  $\omega$  is the frequency). Thus, the Warburg impedance models the process in which a sinusoidal voltage that is applied to an electrochemical cell, at one polarity causes diffusion of mobile ions from the electrode. In this case, the process does not go beyond the diffuse layer region. [16, 23]



Figure 6. Hodograph of impedance Z''(Z') of Ag<sub>8</sub>SiSe<sub>6</sub>. The measurements were performed at temperatures: a - 200, b - 250, c - 300, d - 350 and e - 400 K. An equivalent circuit is shown in the inset to the figure

# 3.4. Frequency dispersion ε' and ε'' for Ag<sub>8</sub>SiSe<sub>6</sub> compound

The real and imaginary parts of the complex permittivity were calculated from the relations  $\varepsilon' = Cd/\varepsilon_0 S$  and  $\varepsilon'' = tg\delta\varepsilon$ . Fig. 7 shows the frequency dependences of the real and imaginary parts of the complex permittivity for Ag<sub>8</sub>SiSe<sub>6</sub> compound measured at temperatures of 200, 250, 300, 350 and 400K. As can be seen from the figures, the  $\varepsilon''(f)$  dependence shows one relaxation peak at a frequency of  $\sim 10^2$  Hz (Fig. 7). In the curves of  $\varepsilon$ "(f) dependence obtained at temperatures 350 and 400K, the feature is absent at  $10^2$  Hz, while it appears at  $10^4$  Hz. The above characteristics reveal dispersion, which manifests itself in in the decrease of the complex impedance values with increasing frequency of the applied electric field. From the curves in Fig. 7, it is seen that  $\varepsilon'(f)$  decreases by several orders of magnitude with increasing frequency from  $10^1$  to  $10^6$  Hz. At low frequencies,  $\varepsilon'(f)$  dependence decreases sharply; at high frequencies, the real part of the complex permittivity depends weakly on frequency, and at frequencies of  $\sim 1$  MHz, it takes the value  $\sim 10$ . The value of high-frequency permittivity for Ag<sub>8</sub>SiSe<sub>6</sub> obtained from impedance measurements is also the value of optical (high-frequency -  $\varepsilon \infty$ ) permittivity of the investigated compound.



**Figure 7.** Frequency dispersion of the real ε' and imaginary ε" parts of the complex permittivity of Ag<sub>8</sub>SiSe<sub>6</sub> compound. The measurements were performed at temperatures of 200(a); 250(b); 300(c); 350(d) and 400(e) K

# CONCLUSIONS

A decrease in electrical conductivity of  $Ag_8SiSe_6$  compound over time has been found during measurements in a constant electric field using silver electrodes. It is shown that the conductivity has an ionic nature in a constant electric field above 300 K. By the study of complex impedance, it is established that in the considered frequency-temperature range, the electrical properties of  $Ag_8SiSe_6$  compound are determined by the conductivity of  $Ag^{+1}$  ions in localized states, while the other is related to the accumulation of charge on blocking electrodes.

#### ORCID

**®R.M. Sardarly**, https://orcid.org/0000-0003-3968-000X;
**®M.B. Babanly**, https://orcid.org/0000-0001-5962-3710
**®N.A. Aliyeva**, https://orcid.org/0000-0002-8448-0552;
**®L.F. Mashadiyeva**, https://orcid.org/0000-0003-2357-6195
**®R.A. Mamadov**, https://orcid.org/0000-0003-0368-1326;
**©G.M. Ashirov**, https://orcid.org/0000-0003-2786-5824
**®A.A. Saddinova**, https://orcid.org/0009-0005-9967-3575;
**®S.Z. Damirova**, https://orcid.org/0009-0008-9536-2894

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### ОТРИМАННЯ ТА ВИМІРЮВАННЯ ІМПЕДАНСНИХ ХАРАКТЕРИСТИК СПОЛУК AgsSiSe<sub>6</sub> Р.М. Сардарли<sup>а,b</sup>, М.Б. Бабанли<sup>с</sup>, Н.А. Алієва<sup>а,d</sup>, Л.Ф. Машадієва<sup>b</sup>, Р.А. Мамадов<sup>a</sup>, Г.М. Аширов<sup>b</sup>, А.А. Саддінова<sup>e</sup>, С.З. Дамірова<sup>e</sup>

<sup>а</sup>Інститут радіаційних проблем НАН Азербайджану, вул. Б. Вагабзаде, 9, АЗ 1143, Баку, Азербайджан

<sup>ь</sup>Кафедра прикладної та загальної фізики, Національна академія авіації, Баку, Азербайджан

<sup>с</sup>Інститут каталізу та неорганічної хімії імені академіка М. Нагієва Міністерства науки і освіти Азербайджанської Республіки, вул. Г. Кавіда, 113, AZ1143, Баку, Азербайджан

<sup>d</sup>Азербайджанський університет архітектури та будівництва, вул. Айна Султанова 5, AZ-1073, Баку, Азербайджан <sup>e</sup>Інститут фізики Міністерства науки і освіти Азербайджанської Республіки, вул. Г. Джавіда, 131, AZ1143, Баку, Азербайджан Зразки сполуки Ag<sub>8</sub>SiSe<sub>6</sub> отримано прямим легуванням вихідних компонентів і подальшим пресуванням порошків під тиском 0,7 ГПа. Досліджено температурні залежності провідності (σ(T)) і діелектричної проникності (ε(T)) сполуки Ag<sub>8</sub>SiSe<sub>6</sub> у постійному та змінному електричних полях. У постійному полі електропровідність зменшується з часом (σ(t)). Це явище пов'язане з процесом накопичення заряду на межі розділу іонний зразок і блокуючий електрод. Спектри комплексного імпедансу досліджували в діапазоні частот 20–10<sup>6</sup> Гц.

Ключові слова: сполука; іонна провідність; спектри комплексного імпедансу