Polarization processes in thin layers of glassy hybrid system Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$

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The results of the investigations of the polarization properties in thin layers of vitreous Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ are presented. A process of dipole-relaxation polarization was discovered, the activation energy of which turned out to be equal to $E_a = (0.97 \pm 0.14)$ eV. It has been determined that charge transfer in the Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ hybrid system is a thermally activated process with an activation energy of $E_a = (0.54 \pm 0.01)$ eV. The calculation results allow us to conclude that the glass-forming capacity of the (Ge$_{28.5}$Pb$_{15.0}$S$_{56.5}$)$_{100-x}$Fe$_x$ system decreases linearly with an increase in the proportion of metal in the glass structure.

Keywords: charge carrier hopping, thin layers, hybrid chalcogenide, lone-pair electrons.

1. Introduction

Unique properties of chalcogenide glassy semiconductors (CGS), on the one hand, and simplicity in manufacture as compared to crystalline semiconductors, on the other hand, condition the prospects of their application as initial materials for the formation of fiber-optic light-emitting diodes and infrared fiber optics lasers $(5-15 \mu m)$. These compounds are used in the manufacture of holographic, xerographic equipment and targets in video camera tubes of the „vidicon" type [1–4].

Close attention is paid in the recent decade to the creation of non-volatile Phase Change Memory (PCM) devices, operating by the principle of a reversible phase transition „amorphous — crystalline state" in CGS. Local structural transformations in the nanodimensional layer of a material occur due to an electric pulse or a light pulse. A successful example of practical use of the said effect is optical DVD-RW disks [5–8].

No less interesting is the study of impurities' influence on electrotransport and charge formation processes in chalcogenide glasses. The experimental results in successful transformation of the *p*-type of conductivity of doped CGS into the *n*-type of conductivity open up new wide opportunities for the engineering of *p*−*n*-transitions and devices operating on their basis [9–13].

The CGS structure peculiarities presuppose a situation when two *p*-electrons form a so-called lone electron pair (LEP), not involved in the formation of covalent bonds. Kastner suggested the term „lone-pair semiconductors" to describe CGS [14]. It has been proved that electrons of lone pairs form the valence band top, their role in memory switchover phenomena and formation of other CGS properties has been noted [15].

One of the methods to control the concentration of lone pairs in a material and thereby to control the properties (including the glass-forming capacity) can be a modification of conventional CGS by transition metals. The authors of [16] studied the charge transfer processes in thin layers of a chalcogenide hybrid system Ge$_{28.5}$Pb$_{14.5}$Fe$_{0.5}$S$_{56.5}$. They found a power dependence of specific conductivity on frequency and a decrease of the value of power index *s* with temperature increase. It was revealed that charge transfer is a thermally activated process with activation energy $E_a = (0.64 \pm 0.02)$ eV. The share of lone electron pairs, calculated for the first time for the given system, showed that the material remains in the glassy state upon injection of iron.

The goal of the present paper is to reveal the peculiarities of polarization processes in thin layers of the glassy Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ system by low-frequency dielectric spectroscopy, to estimate the impact of iron percentage on the glass-forming capacity of the hybrid system under study.

2. Experimental procedure

The peculiarities of polarization processes were studied in thin films of the Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ system obtained by „impact" vacuum deposition. Deposition was performed

Table 1. Element composition of samples of the Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ system (atomic % of chemical element content)

Element	Atomic fraction, %
S	56.48
Ge	28.47
Pb	13.96
Fe	1.09

onto glass substrates located at a distance of $15-30$ cm under the residual gas pressure of 10^{-5} mm Hg. Layer thickness was monitored by the ellipsometric method and was $\sim 2.0\,\mu$m. Samples' amorphism was monitored by the X-ray method on monochromatic radiation with the wavelength of $\lambda = 1.5406$ Å. The obtained X-ray images, measured at large angles 2θ of X-ray scattering in the range of 10 to 80°, indicate the amorphous nature of the studied films.

The samples' element composition was studied using a Carl Zeiss EVO 40 scanning election microscope (SEM). Points on the scans, for which spectra of samples' atomic content were obtained, were chosen for determination of films' atomic composition. Table 1 gives the data on chemical element content in the studied samples. The obtained ratio of elements in glass makes it possible to conclude that the studied system corresponds to the composition Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$.

Dielectric spectra for the studied layers (temperature-frequency dependence of components of complex dielectric quantities) were obtained using „Concept-81" spectrometer (Novocontrol Technologies GmbH) in a wide range of frequencies ($f = 10^0-10^5$ Hz) and temperatures ($T = 273-403$ K).

In the course of the experiment, we measured the values of the imaginary and real parts of impedance of the cell with the measured sample:

$$Z^*(\omega) = R + \frac{1}{i\omega C} = Z' + iZ'' = \frac{U_0}{I^*(\omega)}. \quad (1)$$

The impedance values were used to calculate the spectra of complex permittivity and conductivity based on the following mathematical representations:

$$\varepsilon^* = \varepsilon' - i\varepsilon'' = \frac{-i}{\omega Z^*(\omega)} \frac{1}{C_0}, \quad (2)$$

$$\sigma^* = \sigma' - i\sigma'' = \frac{-i}{\omega Z^*(\omega)} \frac{S}{d}, \quad (3)$$

where $C_0 = \frac{\varepsilon_0 S}{d}$ — empty cell capacity. The relative experiment error did not exceed $\pm 5\%$.

In order to obtain the values of the system's relaxation parameters, the obtained dielectric spectra were approximated by a two-parametric empiric function of Havriliak−Negami using the Novocontrol Winfit software. On the basis of

this approximation, the positions of the maxima of dielectric losses were found and the Havriliak−Negami (HN) parameters for the studied relaxation processes were determined [17]:

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\Delta\varepsilon}{[1 + (i\omega\tau)^{\alpha_{HN}}]^{\beta_{HN}}}, \quad (4)$$

where ε_∞ — high-frequency limit of the real part of permittivity, $\Delta\varepsilon$ — dielectric increment (the difference between low-frequency and high-frequency limits), $\omega = 2\pi f$, α_{HN} and β_{HN} — shape parameters respectively describing the symmetric ($\beta = 1$ — Cole−Cole distribution) and asymmetric ($\beta \neq 1$ — Cole–Davidson distribution) extension of the relaxation function.

3. Low-frequency dielectric relaxation in thin layers of a hybrid chalcogenide system Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$

The frequency dependence of permittivity ε' at different temperatures for films of the Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ system is shown in Fig. 1. It is seen from the figure that ε' increases with frequency decrease, which is most probably due to a manifestation of the dipole-relaxation mechanism of polarization.

A sharp decrease of ε' with frequency in the frequency range of $f > 10^3$ Hz can be explained by a decreased contribution of dipole-relaxation polarization, since it lasts longer than other polarization types (electronic, ionic, polarization of space charge). Dipoles are unable to turn quickly enough, and their rotation lags behind the electric field change. Since frequency continues increasing, the dipole will be unable to follow the field, and orientation polarization will stop. Thus, ε' decreases, approaching a constant value at frequencies of $> 10^6$ Hz, due to the contribution of only electronic polarization and polarization of space charge [12].
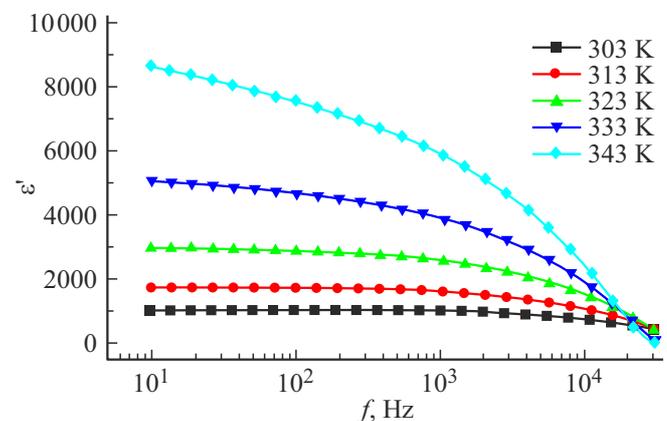

Figure 1. Frequency dependence of permittivity ε' at different temperatures for films of the Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ system.

Figure 2 shows the temperature dependence of permittivity ε'. It is seen from the figure that ε' rises the same as temperature. The rise of ε' with temperature can be assigned to the fact that dipoles in polar materials cannot orient themselves at low temperatures. With temperature rise, dipole orientation eases and the orientation polarization increases, and ε' also increases.

Measurement of the factor of dielectric losses in the layers of the studied system at different temperatures (Fig. 3) has revealed a dispersion of this quantity in a wide frequency range. This peculiarity may be indicative of a relaxation process that conditions the relaxation losses in the samples [18]. This hypothesis should be confirmed by plotting a Cole−Cole diagram, i.e. the dependence $\varepsilon'' = f(\varepsilon')$ (Fig. 4). As seen from Fig. 4, we observe a deviation from the hemispherical dependence with hemisphere radius $(\varepsilon_s - \varepsilon_\infty/2)$, which indicates the presence of a relaxation process associated with distribution of relaxors by relaxation times.

It is known that an offset or distortion of Debye semicircles in different models (Cole–Cole, Davidson–Cole,
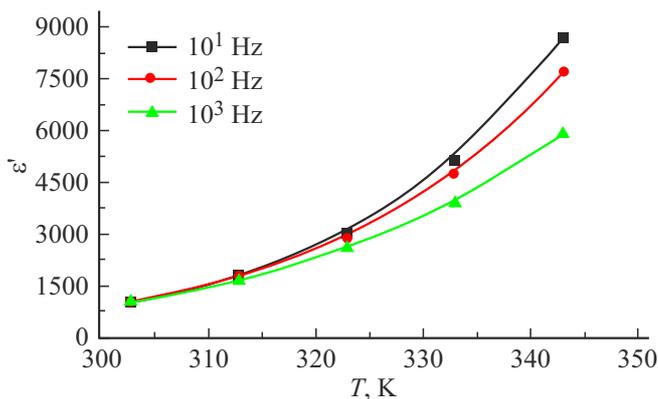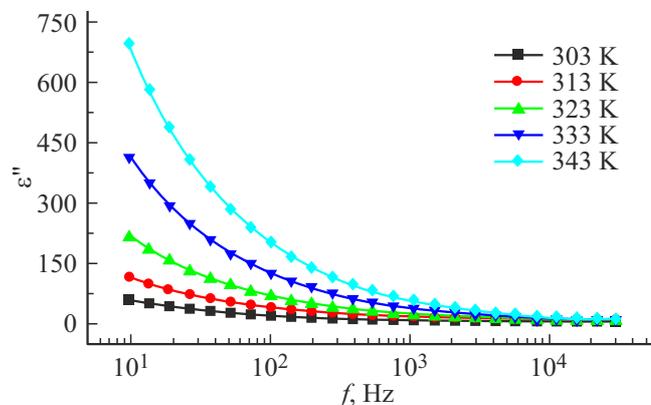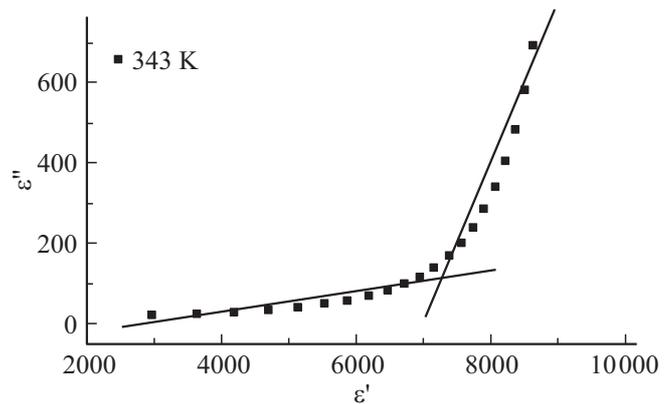

Figure 4. Cole−Cole diagram for samples of the $Ge_{28.5}Pb_{14.0}Fe_{1.0}S_{56.5}$ system at $T = 343\,K$.

Fuoss–Kirkwood, Frelich, Havriliak–Negami) is assigned by certain parameters which characterize either the degree of relaxation time distribution τ, or the measure of relaxors' interaction. However, the said models do not rely on a specific microscopic polarization mechanism, therefore the physical nature of distribution τ or the mechanism of relaxors' interaction remains still indefinite. In this case we can describe the dependence $\varepsilon''(\varepsilon')$, obtained close to a linear dependence for two areas of the given frequency range (Fig. 4), within the framework of the universal Jonscher law for dielectrics, which is based on the energy approach [19]. Polarization in his model is associated with ion or electron hops in long or short chains. A discrete displacement of charges is accompanied with shielding of occurring polarization due to lattice relaxation. Such dependences are associated with a hopping mechanism of conductivity, implemented due to electron (hole) hops across localized energy states near the Fermi level [20,21].

Experimental frequency dependences of the loss factor $\varepsilon'' = (f)$ or tangent of the dielectric loss angle $\operatorname{tg}\delta = (f)$ can be used to estimate the distribution of relaxation times. The values of relaxation parameters obtained by approximation of the experimental curves within the framework of Havriliak−Negami approach (4) (Fig. 3) also confirm the existence of relaxor distribution by relaxation times according to the Cole−Davidson and Cole−Cole models for an asymmetrical and symmetrical distribution of relaxors by relaxation times respectively (see Table 2). Temperature dependence of frequency (relaxation time) $f_m(\tau_m)$ allows for determining the experimental activation energy E_a, i.e. the energy barrier for dipole orientation. The activation energy, calculated according to the temperature dependence of the most probable relaxation time (Fig. 5), was equal to $E_r = (0.97 \pm 0.13)\,eV$.

The found dispersion of permittivity and the existence of a maximum of dielectric losses in the low-frequency area can be explained within the framework of the model proposed in [22]. According to this model, the chalcogenide glass structure is a set of dipoles formed by charged defects

**Figure 2.** Temperature dependence of permittivity ε' for three frequencies of the applied measurement field.

Figure 3. Frequency dependence of the loss factor ε'' at different temperatures for films of the $Ge_{28.5}Pb_{14.0}Fe_{1.0}S_{56.5}$ system. Solid lines — approximation of experimental dependences by a Havriliak−Negami function.

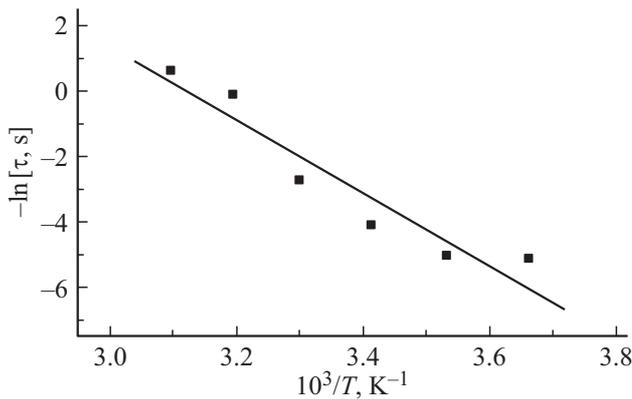**Figure 5.** Temperature dependence of the most probable relaxation time for layers of the hybrid Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56}$ system.

of the D^+ and D^- type. Each dipole has its own relaxation time, which depends on the activation energy required to overcome the potential barrier by charge carriers. The existence of the potential barrier is based on the Coulomb interaction between neighboring defect states forming a dipole. Correlation between the results of this paper and the conclusions made in [23] confirms the hypothesis of existence of a spectrum of defect states, electron exchange between which creates quasidipoles accountable for the permittivity dispersion and dielectric losses in the low-frequency area.

4. Charge transfer in thin layers of the hybrid chalcogenide Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ system

Based on the results of the previous section, we can conclude that the use of the Jonscher's idea of a universal relaxation law presupposes that frequency dependence of alternating-current electrical conductivity follows the law

$$\sigma'(\omega) \sim \omega^s, \qquad (5)$$

where ω is circular frequency on condition that $0 < s < 1$.

Indeed, we found during the experiment that frequency dependence of specific conductivity σ' of the Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ layers at different temperatures is

Table 2. Value of relaxation parameters for samples of the hybrid Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ system

T, K	Tau, s	Alpha	Beta
273	156	0.59	0.69
283	150	0.6	0.72
293	119	0.62	0.72
303	1.6	0.51	1
313	1.1	0.52	1
323	0.5	0.52	1

characterized by its increase with increase of electric field frequency (Fig. 6). It is known than the said dependence is typical for most amorphous semiconductors, for which the frequency dependence of conductivity follows the law (5).

The temperature dependence of the power index in equation (5) shows a decrease of this parameter with temperature rise ($s = 0.47-0.59$) (Fig. 7). The revealed power dependence of conductivity on frequency (5) and the decrease of parameter s with temperature rise suggest a hopping mechanism of conductivity, in which charge carriers (ions, electrons) make thermally activated hops through disordered structures of a semiconductor system.

The hopping conductivity theory includes random processes both in space (random walk) and in time (distribution of wait times). The physical interpretation of random walk consists in that a hopping electron, being in the localized state at a certain time moment, has the finite probability of hopping to one of several possible free adjacent places. As we have demonstrated in [16], the value of systems' polarization parameters can be estimated based on ideas of the hopping mechanism of transfer in the studied hybrid system. The calculation results are given in Table 3.

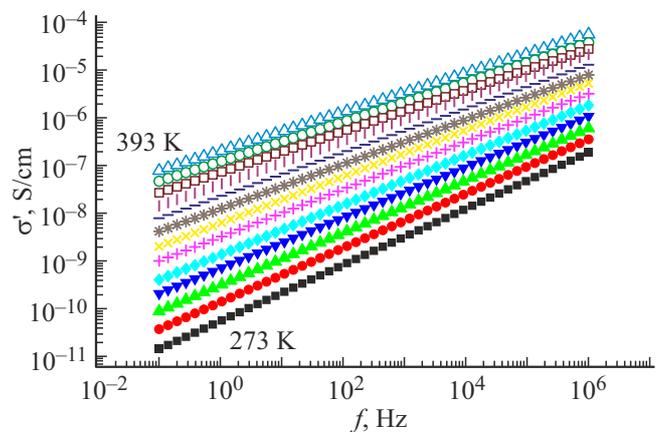Charge transfer in the studied system is a thermally activated process, temperature dependence of specific con-

Figure 6. Frequency dependence of specific conductivity σ' of Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ layers at different temperatures.
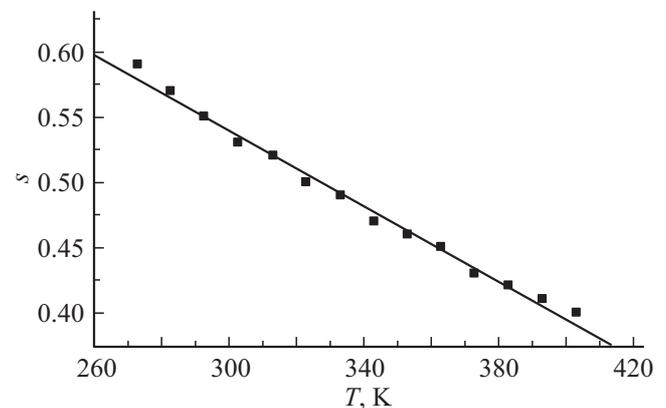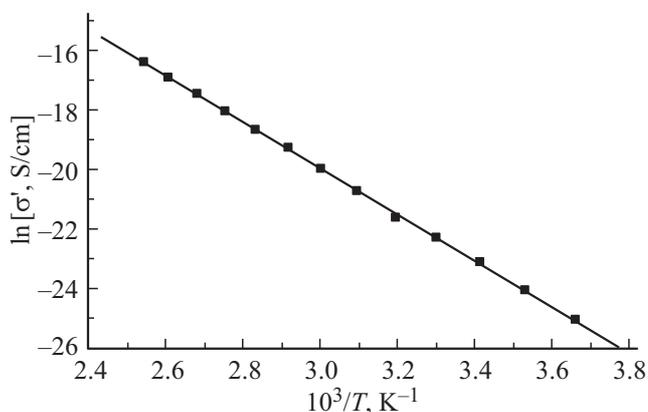

Figure 7. Temperature dependence of power index s.

Table 3. Value of polarization parameters in thin layers of the glassy $Ge_{28.5}Pb_{13.0}Fe_{2.0}S_{56.5}$ system

T, K	ε'	$tg\,\delta$	s	σ', S/cm	W_m, eV	N, m^{-3}
273	20.84	$7.63 \cdot 10^{-2}$	0.59	$1.33 \cdot 10^{-11}$	0.34	$7.24 \cdot 10^{26}$
283	35.95	$1.18 \cdot 10^{-1}$	0.57	$3.53 \cdot 10^{-11}$	0.34	$5.83 \cdot 10^{27}$
293	61.31	$1.73 \cdot 10^{-1}$	0.55	$8.84 \cdot 10^{-11}$	0.33	$4.34 \cdot 10^{28}$
303	103.96	$5.49 \cdot 10^{-2}$	0.53	$2.09 \cdot 10^{-10}$	0.32	$3.05 \cdot 10^{29}$
313	176.02	$6.73 \cdot 10^{-2}$	0.52	$4.13 \cdot 10^{-10}$	0.31	$1.84 \cdot 10^{30}$

ductivity for different frequencies follows the Arrhenius law (Fig. 8). A calculation of activation energy at frequency $f = 10^{-1}$ Hz gives the value $E_a = (0.54 \pm 0.01)$ eV.

According to the CBH model (correlated barrier hopping model) [24], a charge is transferred by way of electron hopping through a potential barrier W between two localized states (centers of equilibrium). Thereat, the height of a barrier between two centers is determined by Coulomb interaction between the neighboring defect states, which can be charged defects of the D^+ and D^- type, forming a dipole. Electron exchange in case of the glassy $Ge_{28.5}Pb_{15}S_{56.5}$ system can take place, for instance, between germanium atoms being in the two- and four-valent state respectively [25].

It is known that the injection of iron impurity into glass causes a change of the conductivity type from the hole type in pure glass to the electron type in the doped system [25]. The Mössbauer spectroscopy method has shown that the glassy $Ge_{28.5}Pb_{15-x}Fe_xS_{56.5}$ and $Ge_{27}Pb_{17-x}Fe_xSe_{56}$ systems have two charge states of impurity iron atoms — $^{57}Fe^{3+}$ and $^{57}Fe^{2+}$, electron exchange between which is not observed. Thereat, impurity iron atoms in the mobility slit form single-electron donor levels: Fe^{2+} ions correspond to neutral centers, and Fe^{3+} ions — to ionized centers. Electrons of iron donor levels fill the local states in the mobility slit, located above the Fermi level so that the Fermi level shifts from the mobility slit middle (in pure glass) to the iron level (in doped glass).

Figure 8. Temperature dependence of specific conductivity σ' at frequency $f = 10^{-1}$ Hz.

Taking into account the structural peculiarities of the studied chalcogenide systems, we can assume that in case of lower iron concentrations its atoms will embed into the glass mesh due to the interaction of lone electron pairs of the chalcogen and d-orbitals of the transition metal. Taking into account the fact that both germanium atoms and iron atoms are in two charge states, a charge transfer can be anticipated, which is associated not only with electron exchange between germanium atoms, but also between germanium and iron atoms [16]. In this case the behavior of charge transfer processes will depend on the nature of iron atoms' embedding into the glass mesh (iron concentration), i.e. an important role will be played by concentration of lone electron pairs.

5. Determination of the share of lone electron pair in the structure of the hybrid chalcogenide $Ge_{28.5}Pb_{14.0}Fe_{1.0}S_{56.5}$ system

The behavior of polarization processes in thin layers of $Ge_{28.5}Pb_{14.0}Fe_{1.0}S_{56.5}$ shows that the limit concentrations of iron impurity which ensure the existence of a single-phase system, whose kinetic properties are defined by interaction of a lone electron pair (LEP) of chalcogen with Fe atoms, must be found during synthesis of such systems. Another important issue is the dependence of the glass-forming capacity of the chalcogenide system on metal percentage.

Thus, as we have demonstrated in [16], the share of LEPs in a chalcogenide system can be determined by the ratio

$$L = V - m, \tag{6}$$

where V is the number of valent units in the system, m is the average coordination number determined according to values of coordination numbers for the elements in the system N_{Ge}, N_{Pb}, N_S, N_{Fe}:

$$m = \frac{aN_{Ge} + bN_{Pb} + cN_S + dN_{Fe}}{a + b + c + d}. \tag{7}$$

According to the Dembowski criterion [26], the glass-forming capacity is determined by the LEP share, namely, in good glass former the share of lone pairs $\Phi = (VEC - K)/VEC$ (where VEC is the total number of valence electrons, K is the coordination number) shall be

Table 4. Glass-forming parameters for the hybrid $Ge_{28.5}Pb_{14.0}Fe_{1.0}S_{56.5}$ system

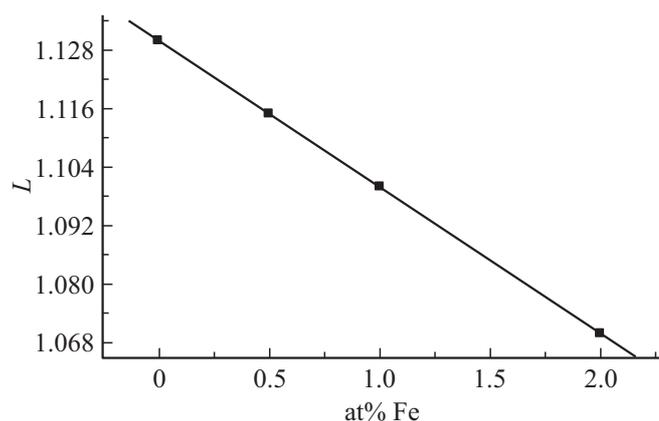| Content of Fe (at%)	V	M	L
0.0	5.130	4.000	1.130
0.5	5.125	4.010	1.115
1.0	5.120	4.020	1.100
2.0	5.110	4.040	1.070

Figure 9. Dependence of glass-forming capacity of the Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ system on iron percentage.

not less than 0-50-0.66 from the total number of valence electrons. It was noted that the given value was determined ambiguously since, firstly, the coordination number in a crystal and glass of the same composition may differ and, secondly, the first coordination shell in glass can be difficult to define unambiguously. Zhenhua [27] drew attention to the fact that the glass-forming capacity also depends on composition complexity, in particular, he demonstrated that while the LEP share for binary systems must be > 2.6, their number for ternary compounds can be > 1. The results of glass-forming capacity calculations for the samples of the Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ system using expressions (6) and (7) are given in Table 4 (Fig. 9). It can be stated that the glass-forming capacity of a chalcogenide system decreases with increase of the metal share in the glass structure. This regularity can be used in practice for estimating the possible impact of an impurity on the physical properties of other hybrid systems, and metal concentration can be estimated if sufficient experimental data is available.

It should be also borne in mind that the conditions for obtaining of an amorphous phase for thin films are less strict than for a bulk material. Thus, amorphous GeTe films can be easily obtained by magnetron sputtering, while amorphous GeTe in the bulk phase cannot be obtained [28].

6. Conclusion

The paper studied the polarization properties of thin layers in the glassy hybrid Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ system in a wide range of frequencies and temperatures. The main results of the performed research are as follows:

— Dispersion of permittivity ε' and factor of losses ε'' in thin layers of Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ are characterized by their increase with frequency decrease and temperature increase, which is most probably due to mechanisms of dipole-relaxation polarization. Activation energy for the given dielectric relaxation process was equal to $E_r = (0.97 \pm 0.14)$ eV.

— The form of the Cole−Cole diagram makes it possible to conclude that a non-Debye relaxation process is observed in the studied samples of Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ in the considered frequency and temperature range; this process is due to the existence of relaxor distribution by relaxation times according to the Cole−Cole and Cole−Davidson models.

— The found power dependence of specific conductivity on frequency and the decrease of power index s with temperature rise suggest the existence of a hopping conductivity mechanism. Charge transfer in the studied hybrid Ge$_{28.5}$Pb$_{14.0}$Fe$_{1.0}$S$_{56.5}$ system is a thermally activated process, temperature dependence of specific conductivity follows the Arrhenius law with activation energy equal to $E_a = (0.54 \pm 0.01)$ eV.

— The results of glass-forming capacity calculations for the samples of the (Ge$_{28.5}$Pb$_{15.0}$S$_{56.5}$)$_{100-x}$Fe$_x$ system make it possible to conclude that the system's glass-forming capacity decreases linearly with increase of the metal share in the glass structure. The obtained dependence of parameter L on metal percentage can be used when estimating the possible influence of an impurity on physical properties of other hybrid systems.

Conflict of interest

The authors of the paper declare that they have no conflict of interest.

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