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Temperature dependence of dielectric properties of the liquid crystal 6CB with the embedded Ag_7GeS_5I nanoparticles

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Abstract. Within the frequency range 10^{-1}–10^5 Hz under the temperatures 293–295 K, the dielectric properties of a planar-oriented nematic liquid crystal 6CB with the embedded of 0.1% superionic conductor Ag_7GeS_5I nanoparticles have been investigated. It has been shown that for the whole temperature range, the obtained frequency dependences of the components ε' and ε" composing the complex dielectric function can be separated into 3 sections. The dispersion of ε' and ε" for the lowest frequencies (less than 10^2 Hz) is described by the Debye equation and is caused by the rotation of the dipole moments of LC molecules under the action of electric field within the angles corresponding to the fluctuations of the order parameter in a thin near-electrode layer. It has been shown that the temperature dependence of the value of inverse relaxation time for such a process is described by straight lines in the Arrhenius coordinates within each mesophase. The activation energies for these dependences have been estimated for each mesophase. It should be noted that within the middle range of frequencies (10^2–10^4 Hz), for each temperature, one can separate a section where the magnitude of the conductivity does not depend on the frequency. The conductivity in these sections is equal to the conductivity of LC with the nanoparticles. It has been found that both the value of inverse relaxation time and the value of conductivity change according to the Arrhenius law on the temperature. The activation energies for the temperature dependence of conductivity and the temperature dependence of inverse relaxation time have been estimated, and it has been shown that they are close (for the nematic phase) and equal (for the isotropic phase). In the highest frequency section of the dielectric spectrum (10^4–10^5 Hz), the conductivity of the mixture 6CB + 0.1 wt.% Ag_7GeS_5I changes according to the power law of the frequency. It has been suggested that a sharp increase in the conductivity of 6CB with nanoparticles of the superionic conductor Ag_7GeS_5I at the concentration 0.1 wt.% was caused by the sharp increase in the electronic component of conductivity through Ag_7GeS_5I nanoparticles.

Keywords: dielectric properties, superionic conductor, frequency dependence, temperature dependence, Debye dispersion, relaxation time, activation energy.

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1. Introduction

After the vast majority of displays throughout the world began to be made on the basis of liquid crystals (LCs), the task of expanding the LC functional characteristics became topical. From the viewpoint of many researchers, the solution of this problem will provide additional opportunities for the practical use of unique properties of LC. One of the most common ways to extend the functional properties of LC is to introduce different types of nanoparticles into these materials [1–3]. This approach to modification of LC properties is closely related to the wide implementation of nano-technologies in researches and production.

From the analysis of recent publications, it follows that different types of nanoparticles (metals, semiconductors and dielectrics) are used to change LC properties. In our publications, it has been shown that the changes in LC properties significant by their value (in particular dielectric properties) can be obtained introducing the nanoparticles of superionic conductors under low concentrations [4–6], in particular the Ag_7GeS_5I nanoparticles [7].

It should be noted that Ag_7GeS_5I compounds have an argyrodite structure and are characterized by the high ionic conductivity [8–10]. Therefore, the argyrodites are promising materials for creation of the solid-state batteries, supercapacitors and other electrochemical devices. For these purposes they were obtained in the different forms of composites, ceramics and thin films [11–14].

In the work [7], we investigated the effect of Ag_7GeS_5I nanoparticles on the electrical properties of nematic liquid crystal 6CB in comparison with similar action of Cu_7GeS_5I nanoparticles [4] at the temperature 293 K. It has been shown that the conductivity of 6CB with the introduction of the maximum concentration chosen for researches (0.1 wt.% of Ag_7GeS_5I nanoparticles) almost 5-fold increases, while at the same concentration of Cu_7GeS_5I nanoparticles, the conductivity increased by only 1.3 times.

We also revealed a non-typical concentration dependence of conductivity as compared with the results obtained previously. Namely, it was shown that at the maximum of the chosen concentrations *c* (0.1 wt.%) the conductivity of 6CB LC increased sharply, while at the same values of *c* but for another type of superionic nanoparticles, the conductivity was less dependent of the *c*-value [5, 6] than at lower concentrations of impurity or reached saturation [4]. To determine the possible causes of such an effect, it is necessary to carry out some additional studies not only at room temperature but also at the other ones.

Therefore, the purpose of this work was to investigate the dielectric properties of 6CB LC with the impurities of 0.1 wt.% of Ag_7GeS_5I superionic conductor nanoparticles over a wide temperature range, which would include not only the nematic but also the isotropic phase of LC.

2. Materials and methods

A polar nematic liquid crystal belonging to the cyanobiphenyl family, 4-cyano-4-n-hexylbiphenyl (6CB) (BDH Limited Poole England), was used. It has the nematic phase between 14.5 and 29 °C.

The synthesis of Ag_7GeS_5I compounds was performed from Ag, S, and P taken in accordance with the stoichiometry and placed in an evacuated ampoule made of silica. The ampoule was heated at the rate of 50 K/h to the temperature 673 ± 5 K and kept at this temperature for 24 h. Then the temperature was increased up to the value 973 ± 5 K, and the ampoule was kept at this temperature for 3 days. Further, the melting zone was heated up to 1380 ± 5 K, which is by 50 K above the melting temperature with 24-h ageing. This ageing resulted in nucleation. The annealing of the formed seeds was performed for 48 h.

Ag_7GeS_5I powders were milled with a planetary ball-mill (Fritsch Pulverisette 6). Approximately 2 g of powder was milled in a stainless steel vial of 80 ml volume using four stainless steel balls of 20-mm diameter in each run. The rotation speed was 400 rpm, and the milling atmosphere was 2 GPa Ar. The maximum milling time of the powder was 30 min.

6CB liquid crystals without/with Ag_7GeS_5I nanoparticles were studied in a sandwich-type cell with transparent ITO electrodes. The electrodes were coated with an appropriately processed polymer layer to provide the planar orientation of the liquid crystal molecules. The concentration of nanoparticles of the sphere-like shape with the average size close to 220 nm in the liquid crystal was 0.1 wt.%. The cell thickness was 20 μm. The LC cell was filled using the capillary method at the temperature by 5–10 K above the nematic-to-isotropic phase transition temperature.

Dielectric properties of the prepared sandwich cells were investigated within the frequency range $0.1–10^5$ Hz under the temperatures 293–315 K by using the oscilloscopic method [15]. The amplitude of the measuring signal possessing the sinusoidal shape was 0.2 V. Assuming that the equivalent circuit of the measuring cell is the parallel impedance and capacitor, the values of resistance *R* and capacitance *C* of the samples at different frequencies were determined, and based on these values and geometric dimensions of the cell, respectively, the imaginary (ε'') and real (ε') components of the complex dielectric function were determined as well. According to the resistance *R* value in the section of frequency dependence, where the resistance is independent of frequency, the conductivity of the liquid crystal on alternating current was determined. Temperature stabilization with an accuracy of 0.1 K was provided by using the developed thermostat with low noise, which was important for reducing the measurement error of *R* and *C* values. To reduce the influence of transition (when the temperature changes) processes, the measurements were performed with a constant decrease in temperature, starting from its maximum (315 K) value.

3. Results and discussion

Fig. 1 shows the frequency dependences of the components of the complex dielectric function ε' (*1*, *3*) and ε" (*2*, *4*) of the mixture 6CB + 0.1 wt.% Ag_7GeS_5I at the temperatures 295.5 K (*1*, *2*) and 313 K (*3*, *4*).

As it follows from the analysis of Fig. 1, the whole dielectric spectrum (frequency dependences of ε' and ε") can be separated into 3 sections. The first section of the dielectric spectrum (for the frequencies less than 10^2 Hz) shows significant changes in the values of ε' and ε" with the frequency. Our analysis of this region in the spectrum on the basis of the dependences ε"(ε') (Cole–Cole diagrams) showed that the dielectric spectra can be described by the relation

$$\varepsilon^* = \varepsilon_\infty + \frac{\varepsilon_0 - \varepsilon_\infty}{1 + i\omega\tau}, \qquad (1)$$

where ε* is the complex dielectric permittivity, ε_0 and ε_∞ are the dielectric permittivities for the frequencies $f = 0$ and $f = \infty$, and τ is the relaxation time.

As it is known from Ref. [16], Eq. (1) corresponds to the Debye dispersion. In Ref. [17], we showed that in the case of planar orientation of molecules this relaxation process is caused by rotation of the molecular dipoles (within the range of angles corresponding to fluctuations of the order parameter) in a thin near-electrode layer with the thickness of order of tens of nanometers.

From the analysis of Eq. (1), it follows that the frequency range of the dispersion of ε' and ε" is largely dependent of the τ-value. Since for further analysis of the obtained data related to the dielectric properties of 6CB with the Ag_7GeS_5I nanoparticles, we will need a temperature dependence not of the τ-value, but the value of inverse τ (*i.e.*, τ^{-1}), then we will analyze just the temperature dependence of τ^{-1} further. The temperature dependence of τ^{-1} is shown in Fig. 2.

From the analysis of Fig. 2, we can conclude that within each mesophase (nematic phase N or isotropic phase I) in the Arrhenius coordinates lg τ^{-1} (T^{-1}), a linear dependence is observed. *I.e.*, the temperature dependence of τ^{-1}-value can be described using the relation

$$\tau^{-1} = \tau_0^{-1} e^{-\frac{E_\tau}{kT}}, \qquad (2)$$

where τ_0^{-1} is the value of inverse relaxation time for an infinite temperature, E_τ is the activation energy for the temperature dependence inherent to the value of inverse relaxation time, and *k* is the Boltzmann constant.

The performed estimations of E_τ-value, based on the analysis of the data in Fig. 2, showed that for the nematic phase $E_{\tau N} = 0.72 \pm 0.04$ eV and for the isotropic phase $E_{\tau I} = 0.41 \pm 0.04$ eV.

To analyze the dielectric spectrum for the frequencies higher than 10 Hz, we first consider the frequency dependence of conductivity of two different values of temperature. These data are shown in Fig. 3.

In Fig. 3, two sections can be distinguished. In the first one, the conductivity does not depend on the frequency (this section is marked by a horizontal line in each graph). As it is characteristic of most liquids [16], their resistance is independent of the frequency. Therefore, the conductivity determined in this section can be considered as the conductivity of a liquid crystal with nanoparticles (without taking into account the conductivity through the nanoparticles).

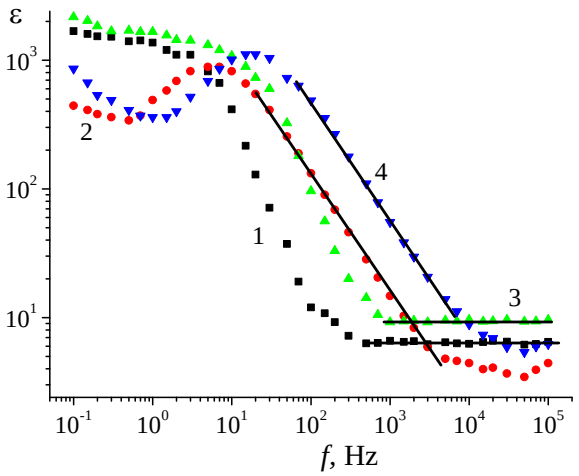

Fig. 1. Frequency dependences of the components of the complex dielectric function ε' (*1*, *3*) and ε" (*2*, *4*) of 6CB LC with the impurity of 0.1 wt.% nanoparticles of the superionic conductor Ag_7GeS_5I at the temperatures 295.5 K (*1*, *2*) and 313 K (*3*, *4*). The thickness of the samples is 20 μm.

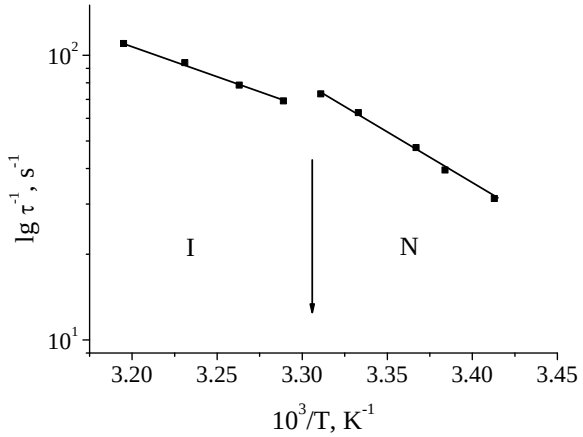

Fig. 2. Temperature dependence of the lg τ^{-1} value for the planar-oriented mixture 6CB + 0.1 wt.% Ag_7GeS_5I. The vertical arrow indicates the temperature of phase transition "nematic-isotropic".

Determined in these sections was the conductivity on alternating current σ_{AC} of liquid crystal 6CB with impurities of superionic conductor nanoparticles at different temperatures. To calculate the value of the conductivity by the known value of ε", we used the ratio

$$\sigma_{AC} = \varepsilon_0 \varepsilon'' \omega, \qquad (3)$$

where ε_0 is the electric constant and $\omega = 2\pi f$ is the cyclic frequency.

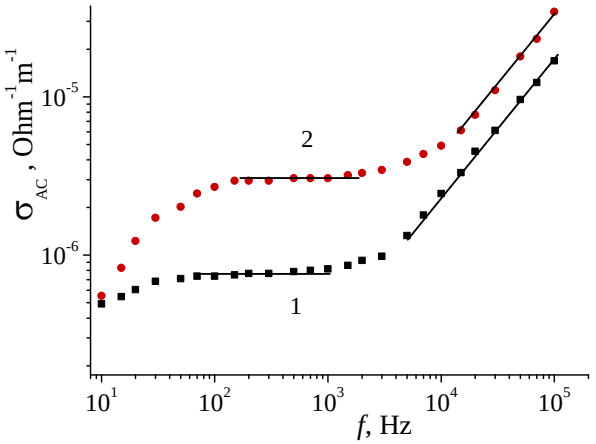

Fig. 3. Frequency dependences of the conductivity of a planar-oriented mixture 6CB + 0.1 wt.% Ag_7GeS_5I for the temperatures 295.5 K (nematic phase of LC) (*1*) and 313 K (isotropic phase of LC) (*2*).

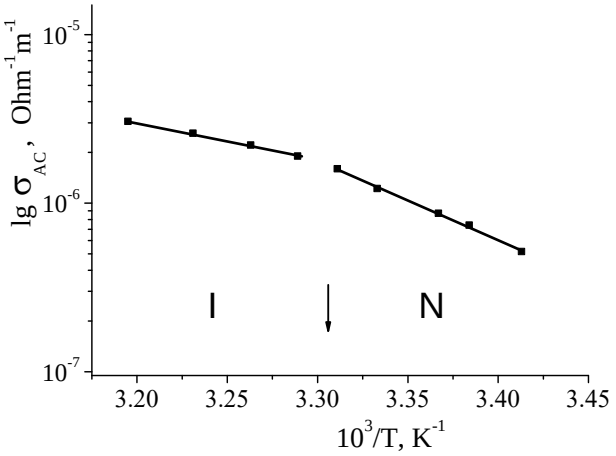

Fig. 4. Temperature dependence of the lg σ_{AC} for a planar-oriented mixture of 6CB + 0.1 wt.% Ag_7GeS_5I. The vertical arrow indicates the temperature of phase transition "nematic-isotropic".

The temperature dependence of the conductivity is shown in Fig. 4. From its analysis, it follows that, as in the case of the temperature dependence of τ^{-1}, within the temperature range of the mesophase existence, in the Arrhenius coordinates the linear segments are observed. *I.e.*, both the temperature dependence of τ^{-1} and the temperature dependence of AC conductivity can be described by the relation

$$\sigma_{AC} = \sigma_0 e^{-\frac{E_\sigma}{kT}}, \quad (4)$$

where σ_0 is the electrical conductivity at infinite temperature, and E_σ – activation energy of the electrical conductivity.

If to compare the temperature dependences of the τ^{-1} (Fig. 2) and σ_{AC} (Fig. 4) values, one can clearly note the complete correlation between the temperature dependences of the τ^{-1} and σ_{AC} values.

Despite the fact that the relaxation process described by Eq. (1) is caused by near-electrode processes (namely, the oscillation of molecular dipoles between the angles corresponding to the fluctuations of the order parameter), the current flowing through the sample depends on the electrical parameters of the bulk part of the sample (namely, from the value of conductivity). The lower the conductivity of the bulk part of the sample, the slower the process will occur. Therefore, just the correlation between the temperature dependences of τ^{-1} and σ_{AC} values is one of the major confirmations that dispersion of the dielectric permittivity for the frequencies less than 10^2 Hz can be described by the near-electrode relaxation process (Eq. (1)).

Another confirmation that the low-frequency dispersion of ε' and ε'' may be described by Eq. (1) can be obtained by comparing the activation energies of the temperature dependences of τ^{-1} and σ_{AC}. Being based on the data shown in Fig. 4, it was found that the activation energy of the temperature dependence of conductivity is: $E_{\sigma N} = 0.92 \pm 0.04$ eV for the nematic phase and $E_{\sigma I} = 039 \pm 0.04$ eV for the isotropic phase. It means that, in the nematic phase, the $E_{\sigma N}$ and $E_{\sigma I}$ values somewhat differ, however, in the isotropic phase, the $E_{\sigma N}$ and $E_{\sigma I}$ values are equal to each other within the limits of measurement errors.

From the analysis of Fig. 3, it follows that for the frequencies higher than 10^3 Hz in double logarithmic coordinates (for frequency and conductivity) the linear parts are observed. In more detail than in Fig. 3, this frequency dependence of conductivity for three different temperatures (295.5, 300, and 313 K) is shown in Fig. 5. From the obtained data, we can conclude that the range of linear dependence of the σ_{AC}-value on frequency in double logarithmic coordinates is observed for all these temperatures. Our analysis showed that this dependence is observed within the whole temperature range (293–415 K).

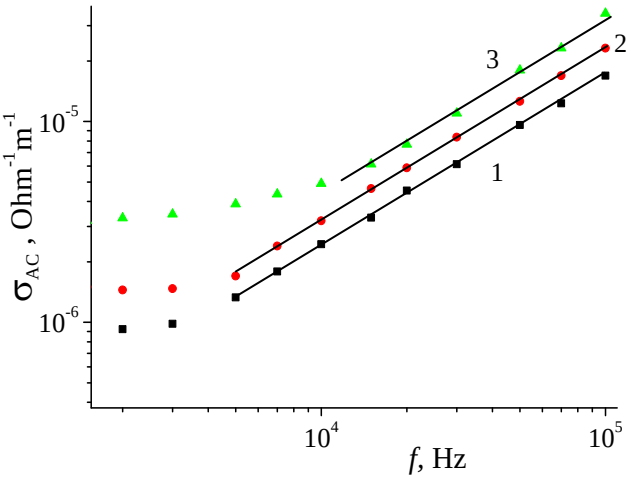

Fig. 5. Frequency dependences of conductivity for the planar-oriented mixture 6CB + 0.1 wt.% Ag_7GeS_5I for the temperatures 295.5, 300 and 313 K.

From the mathematical viewpoint, the linear part in double logarithmic coordinates means that the conductivity of the samples depends on the frequency according to the power law and can be described by the following relation

$$\sigma_{AC} = \sigma_{f0} f^m, \quad (5)$$

where σ_{f0} is the conductivity at the frequency $f = 0$ (conduction on direct current), and m is the exponent for the frequency dependence of the conductivity.

As it follows from Figs 4 and 5 as well as from the analysis of the frequency dependences of the σ_{AC}-value for other temperatures, the slope of the curves corresponding to the frequency dependences of conductivity at different temperatures was the same. From Eq. (5), it means that the exponent for the frequency dependence of conductivity is independent of the temperature. Based on our estimates, m is equal to 0.86 ± 0.03. That is, there is the almost linear frequency dependence (for this dependence $m = 1$).

The dependence of conductivity on frequency described by Eq. (5) is typical for the hopping mechanism of conduction that is the most researched for inhomogeneous solids [18]. It can be assumed that for our samples, this conduction is provided by electron transfer through the nanoparticles. In this case, the hopping charge transfer will occur both inside the nanoparticles themselves due to electron transfer and when transferring the charge carriers between the nanoparticles through the liquid crystal (due to ion transfer).

In the case of Ag_7GeS_5I nanoparticles, this charge transfer mechanism may be more effective than that for the other nanoparticles studied by us. It may be the main reason for the sharp increase in the conductivity of 6CB with the 0.1 wt.% concentration of Ag_7GeS_5I nanoparticles, which was obtained in Ref. [7].

4. Conclusions

1. Within the frequency range 10^{-1}–10^5 Hz under the temperatures 293–295 K, the dielectric properties of a planar-oriented nematic liquid crystal 6CB with the embedded 0.1% superionic conductor Ag_7GeS_5I nanoparticles have been investigated. It has been shown that for the whole temperature range, the obtained frequency dependences of the components comprising the complex dielectric permittivity ε' and ε" can be separated into 3 sections.

2. It has been shown that for the frequencies lower than 10^2 Hz (low frequency section of the dielectric spectrum), the dispersion of ε' and ε" is described by the Debye equation and is caused by rotation of the dipole moments of LC molecules under the action of electric field between the angles corresponding to the fluctuations of the order parameter in the thin near-electrode layer. The values of the relaxation times τ have been estimated for this relaxation process, and it has been shown that they changes from 8 up to 50 ms. The activation energies for the temperature dependence of the inverse relaxation time τ^{-1} have been also estimated for the nematic phase ($E_{\tau N} = 0.72 \pm 0.04$ eV) and for the isotropic one ($E_{\tau I} = 0.41 \pm 0.04$ eV).

3. It has been shown that, regardless of the temperature value, in the frequency dependence of conductivity one can clearly separate the frequency range (the middle part of the dielectric spectrum) where the conductivity value is independent of the frequency. The conductivity determined in this section is equal to the conductivity of liquid crystal with nanoparticles. The temperature dependence of the conductivity within the limits of each mesophase (nematic and isotropic phases) as well as the temperature dependence of the τ^{-1}-value can be described by straight lines in the Arrhenius coordinates. The activation energies for the temperature dependence of conductivity in the nematic ($E_{\sigma N} = 0.92 \pm 0.04$ eV) and isotropic ($E_{\sigma I} = 039 \pm 0.04$ eV) phases have been estimated. The close activation energy values of temperature dependences of τ^{-1} and σ_{AC} for the nematic phase of LC and the practically equal ones for the isotropic phase confirm the proposed mechanism of the low-frequency relaxation process.

4. In the highest frequency range of the dielectric spectrum (mainly in the frequency range 10^4–10^5 Hz), the conductivity of the mixture 6CB + 0.1 wt.% Ag_7GeS_5I changes in accord with the power law of frequency. It has been found that, independently of temperature, the exponent for this dependence is the same and equal to 0.86 ± 0.03. It has been suggested that a sharp increase in the conductivity of 6CB with nanoparticles of the superionic conductor Ag_7GeS_5I at the concentration 0.1 wt.%, which was revealed in Ref. [7], is related with a sharp increase in the electronic component of conductivity through nanoparticles. Since this process occurs in the system disordered for charge transfer, it is realized by the hopping transfer of charge carriers and is described by the power law of conductivity on frequency.

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