Optics

Influence of cation substitution on optical constants of $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals

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Abstract. $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals were grown using the vertical zone crystallization method, they have been shown to crystallize in cubic structure ($F\bar{4}3m$). The diffuse reflection spectra for the powders of $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals were measured at room temperature. Refractive indices and extinction coefficients for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals were obtained from spectral ellipsometry measurements. A nonlinear decrease of the energy pseudogap and a nonlinear behavior with the maximum of refractive index have been revealed with increasing the Ag content. The dispersion of refractive indices of $(Cu_{1-x}Ag_x)_7SiS_5I$ has been described in the framework of different models.

Keywords: mixed crystal, spectral ellipsometry, refractive index, diffuse reflection, energy pseudogap.

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1. Introduction

Cu_7SiS_5I and Ag_7SiS_5I crystals belong to the family of compounds with argyrodite structure [1, 2]. At room temperature, they crystallize in the cubic symmetry (space group $F\bar{4}3m$). It should be noted that the electrical conductivity of Cu_7SiS_5I and Ag_7SiS_5I crystals at room temperature was found to be rather high and typical for the advanced superionic conductors [3, 4]. Due to the high ionic conductivity, they are attractive materials for applications as the solid electrolytes in a new generation of power sources as well as for studying the order-disorder processes. Recently, argyrodites were fabricated in the form of composites [5, 6], ceramics [7] and thin films [8].

Optical studies of Cu_7SiS_5I crystals were performed in Ref. [9]. It was shown that within the temperature interval 77…300 K the optical absorption edge has an exponential shape, which temperature behavior is described by the Urbach rule [9]. The Urbach behavior of absorption edge in Cu_7SiS_5I crystal is caused by strong electron-phonon interaction, which is well explained in Dow–Redfield model. In Ref. [9], we have shown that the optical pseudogap energy and Urbach energy are well described in Einstein model, and contributions of structural and temperature disordering into the Urbach energy at 300 K are equal to 69% and 31%, respectively.

Structural, electrical and optical properties of Cu_7SiS_5I-based mixed crystals were studied in Refs. [10, 11]. In Ref. [11], it was shown that in $Cu_7(Ge_{1-x}Si_x)S_5I$ mixed crystals, like to the Cu_7SiS_5I crystal, the optical absorption edge has the Urbach shape and the corresponding Urbach bundles are revealed. With Si content increase in $Cu_7(Ge_{1-x}Si_x)S_5I$ mixed crystals, a nonlinear increase of the optical pseudogap energy and typical for mixed crystals variation of Urbach energy are observed [11].

This paper is devoted to study of the growth process and optical properties of $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals obtained by means of directed crystallization from the melt (Bridgman–Stockbarger method). Besides, the paper is aimed at studying the compositional dependences of the optical parameters of $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals.

2. Experimental

The process of crystals growth was performed for both individual chemical compounds and for solid solutions based on them. The growth was made by means of directed crystallization from the melt method (zonal crystallization). The specifics of $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals growth method, as in the case of obtaining individual compounds, is related with the fact that synthesis and growth of the crystals were performed in the same ampoule without overloading the charge. It enables to obtain mixed crystals of the defined composition without deviations from stoichiometry in the whole interval of concentrations.

Synthesis of $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals was performed according to the following procedure: the temperature of both zones increases to 1023 K within 10 hours, hereupon the 24-hour exposure is performed, then the temperature increases for the day to the maximum temperature values 1473 K for "hot" upper zone and 873 K for "cold" lower zone. The temperature in the melt zone was kept by 50 K above the melting point to prevent substances from partial thermal dissociation. After that, the 120-hour exposure was performed, at which the full homogenization of melt took place.

The vertical zone crystallization method was used to obtain the homogeneous single crystals of solid solutions under investigation. After shifting the ampoule with crystal into the annealing zone, the homogenizing annealing required for relaxation of thermal stresses in crystals was being carried out for 3 days. Thus, $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals with 30…40-mm length and 10…15-cm diameter were obtained (Fig. 1).

The spectroscopic ellipsometer M-2000V was used to measure optical constants. The latter were carried out within the spectral range 370 to 1000 nm at the angle of incidence close to 70°. The diffuse reflection measurements of $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals were performed using the LOMO MDR-3 spectrometer.

3. Results and discussion

Fig. 2 shows the diffuse reflection spectra for the powders of $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals. The short-wave edge of the diffuse reflection spectra for the powders of these crystals is shown to shift towards longer wavelengths with substitution of Cu atoms with the Ag ones. It should be noted that variation of the reflectance edge spectral position at the Cu $\rightarrow$ Ag substitution is typically observed for solid solution rows [12]. From the spectral position of the short-wave edge of the diffuse reflection spectra, the energy pseudogap value was estimated. It was revealed that the compositional dependence of the energy pseudogap has shown the nonlinear behaviour with the downward-bowing. The compositional dependence of energy pseudogap can be described using the equation [13]:

$$E_g(x) = E_g(0) + \left[E_g(1) - E_g(0)\right]x - cx(1-x) , \qquad (1)$$

where $E_g(x=0) \equiv E_g(0)$ and $E_g(x=1) \equiv E_g(1)$ are the energy pseudogap values for Cu_7SiS_5I and Ag_7SiS_5I crystals, respectively, c is the bowing parameter that is a measure of deviation from $E_g(x)$ function linearity. The best agreement, when describing the experimental dependence $E_g^*(x)$ by using Eq. (1), was obtained by means of the following parameters $E_g^*(0) = 2.277\text{ eV}$, $E_g^*(1) = 1.732\text{ eV}$ and c = 0.65 eV. It should be noted that the positive value of c is the evidence of downward-bowed compositional dependence of the energy pseudogap. According to the Refs. [14, 15] the bowing of the energy pseudogap plot can be caused by energy band deformation due to the change of lattice parameters in solid solutions, change of electronegativity, and structural changes due to the cation bond length variation.

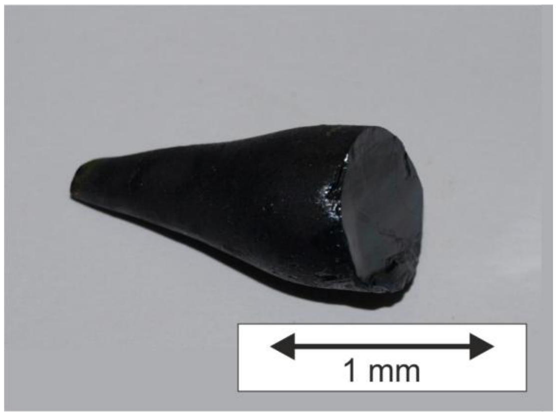

Fig. 1. Image of $(Cu_{0.5}Ag_{0.5})_7SiS_5I$ mixed crystal.

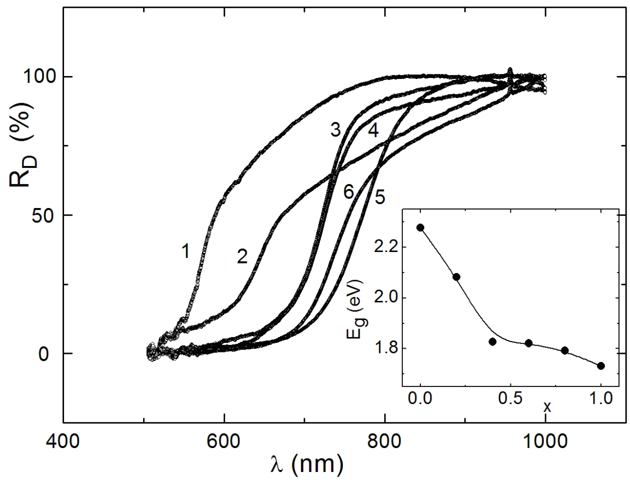

Fig. 2. Diffuse reflection spectra of $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals: Cu_7SiS_5I (*1*), $(Cu_{0.75}Ag_{0.25})_7SiS_5I$ (*2*), $(Cu_{0.5}Ag_{0.5})_7SiS_5I$ (*3*), $(Cu_{0.25}Ag_{0.75})_7SiS_5I$ (*4*), Ag_7SiS_5I (*5*). The inset shows the compositional dependence of energy pseudogap.

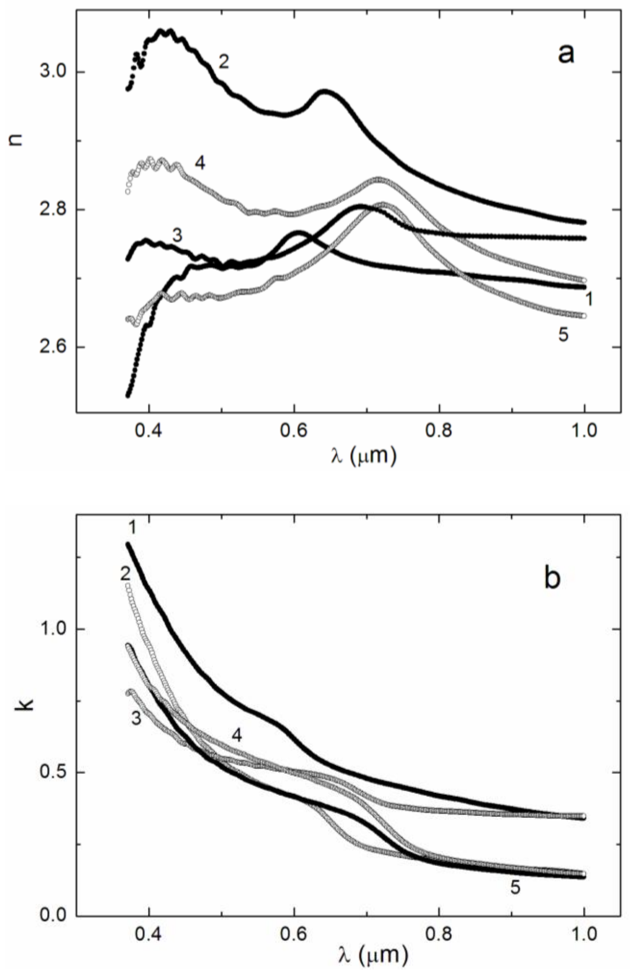

Fig. 3. Spectral dependences of the refractive index n (a) and extinction coefficient k (b) for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals: Cu_7SiS_5I (*1*), $(Cu_{0.75}Ag_{0.25})_7SiS_5I$ (*2*), $(Cu_{0.5}Ag_{0.5})_7SiS_5I$ (*3*), $(Cu_{0.25}Ag_{0.75})_7SiS_5I$ (*4*), Ag_7SiS_5I (*5*).

Refractive indices n and extinction coefficients k for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals were obtained from spectral ellipsometry measurements that were carried out within the spectral range of 0.37…1.0 μm (Fig. 3). In the transparency range, the slight dispersion of refractive index is observed, besides, the dispersion of refractive index increases when approaching to the optical absorption edge. The two anomaly dispersion regions of refractive index were revealed in the ranges of the extinction coefficient increase (Fig. 3). The long wavelength (*e.g.*, λ = 644 nm or E = 1.926 eV for $(Cu_{0.75}Ag_{0.25})_7SiS_5I$)) anomaly corresponds to the band-to-band optical transition and the spectral position of this anomaly relates to the energy pseudogap value. Another short wavelength (*e.g.*, λ = 428 nm or E = = 2.897 eV for $(Cu_{0.75}Ag_{0.25})_7SiS_5I$)) broad and smeared for some mixed crystals anomaly possibly corresponds to the interband Van Hove–Phillips singularity. This type of singularity was also revealed in another argyrodites, such as Cu_6PS_5I crystals [16].

Fig. 4 presents the compositional dependences of the refractive index at λ = 1 μm and energy pseudogap E_g obtained from spectral ellipsometry measurements for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals. It should be noted that E_g values in this case were determined as the spectral position of the knee that was observed on the spectral dependences of extinction coefficient k (Fig. 3b). It has been shown that, with Ag content increase, nonlinear behavior with the maximum of refractive index as well as the nonlinear decrease of energy pseudogap are observed (Fig. 4).

It should be noted that the energy pseudogap E_g values obtained from the analysis of diffuse reflection spectra (inset in Fig. 2) and spectral dependences of extinction coefficient (Fig. 3b) for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals do not differ more than 5%. Moreover, E_g value (2.250 eV) determined from the absorption coefficient spectrum [9] for Cu_7SiS_5I crystal is in a good agreement with E_g values determined from the above mentioned diffuse reflection spectrum (2.277 eV) and spectral dependence of extinction coefficient (2.125 eV).

The model for the refractive index dispersion is of great importance, because it defines the number of the unknown parameters and their functional relation. Some of the most popular models are named after the scientists that have proposed them: Cauchy, Drude, Sellmeier, Lorentz, *etc*. [17]. It should be noted that the Cauchy dispersion law is purely empirical [17]:

$$n(\lambda) = n_0 + \frac{b}{\lambda^2} + \frac{c}{\lambda^4} + \ldots \ , \qquad (2)$$

where b, c, … are some adjustable parameters. The number of the terms can reach 10…15. The Cauchy model calculations using two parameters are shown in Table 1.

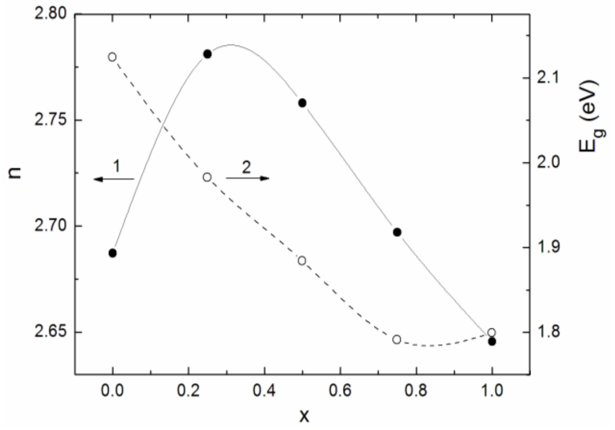

Fig. 4. Compositional dependences of the refractive index n (*1*) and energy pseudogap E_g (*2*) for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals.

Table 1. Parameters of the Cauchy model for description of refractive index dispersion for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals.

Crystal	Parameters		
	n_0	b	c
Cu_7SiS_5I	2.683	–0.005	0.013
$(Cu_{0.75}Ag_{0.25})_7SiS_5I$	2.789	–0.071	0.064
$(Cu_{0.5}Ag_{0.5})_7SiS_5I$	3.136	–0.506	0.171
$(Cu_{0.25}Ag_{0.75})_7SiS_5I$	2.696	–0.089	0.090
Ag_7SiS_5I	2.703	–0.195	0.136

Table 2. Parameters of the Sellmeier model for description of refractive index dispersion for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals.

Crystal	Parameters			
	B_1	C_1	B_2	C_2
Cu_7SiS_5I	0.045	0.337	6.134	0.011
$(Cu_{0.75}Ag_{0.25})_7SiS_5I$	0.478	0.309	5.909	0.022
$(Cu_{0.5}Ag_{0.5})_7SiS_5I$	0.050	0.457	6.630	0.016
$(Cu_{0.25}Ag_{0.75})_7SiS_5I$	0.580	0.366	5.373	0.002
Ag_7SiS_5I	0.515	0.386	5.113	0.005

We also applied the Sellmeier dispersion relation that is semi-empirical for describing the refractive index dispersion of $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals [17]:

$$n^2(\lambda) = 1 + \frac{B_1\lambda^2}{\lambda^2 - C_1} + \frac{B_2\lambda^2}{\lambda^2 - C_2} + \frac{B_3\lambda^2}{\lambda^2 - C_3} + \ldots, \quad (3)$$

where B_1, C_1, B_2, C_2,… are some adjustable parameters. More terms can be added for different oscillator positions. The Sellmeier model calculations using two polynomials are shown in Table 2.

Among the models that describe the refractive index dispersion based on the relationship between the refractive index and the energy gap, one should first of all mention the well-known Wemple–DiDomenico (WD) model [18]. Wemple and DiDomenico have proposed the model where the refractive index dispersion is studied in the transparency region below the gap, using the single-oscillator approximation [18]. In this case, the energy dependence of refractive index can be described by the relationship [18]

$$n^2(E) - 1 = \frac{E_d^{\mathrm{WD}} E_0^{\mathrm{WD}}}{\left(E_0^{\mathrm{WD}}\right)^2 - E^2}, \quad (4)$$

where E_0^{WD} is the single-oscillator energy, and E_d^{WD} – dispersion energy. The dispersion energy E_d^{WD} characterizes the average strength of interband optical transitions and relates with the changes in the structural ordering of the material (ionicity, anion valency and coordination number of the material). From the dependences $\left(n^2 - 1\right)^{-1}$ on E_2, using Eq. (4) the E_0^{WD} and E_d^{WD} values were determined. The above mentioned parameters of the WD model for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals are listed in Table 3. It is shown that with the increase of Ag content the single-oscillator energy E_0^{WD} and dispersion energy E_d^{WD} nonlinear decrease for $Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals with maximum at x = 0.5 (Fig. 5). The nonlinear and non-monotonous compositional behaviour of the parameters of the WD model (Fig. 5) as well as the refractive index and energy pseudogap (Fig. 4) is explained by the compositional disordering of crystal lattice typical for argyrodite-type mixed crystals [11, 12]. According to the relation $E_0^{\mathrm{WD}} \approx 2E_g^{opt}$ [19], the optical band gap value E_g^{opt} was estimated and presented in Table 3. It should be noted that E_g^{opt} values and the energy pseudogap E_g values obtained from the analysis of diffuse reflection spectra for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals do not differ more than 13%.

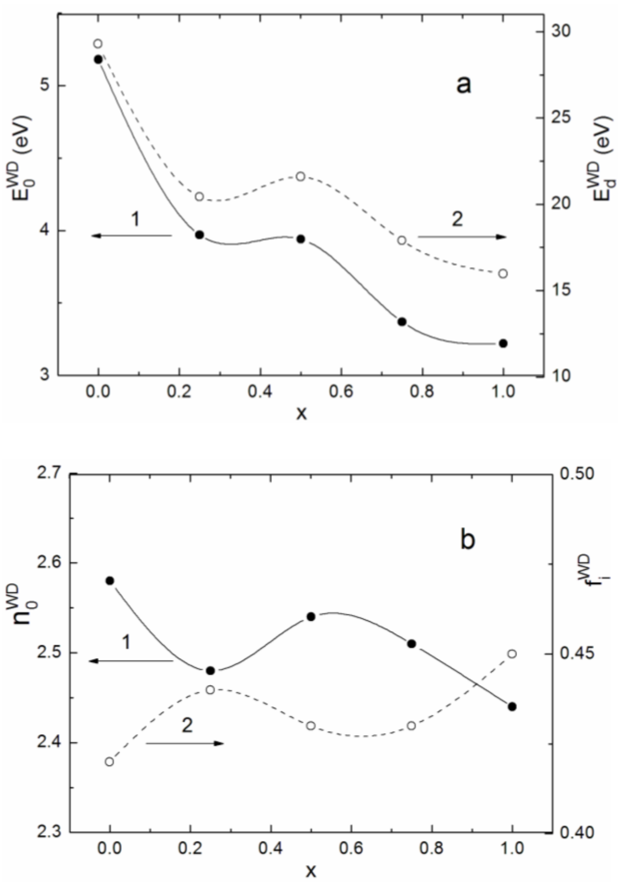

Fig. 5. Compositional dependences of the parameters of the WDD model for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals: (a) single-oscillator energy E_0^{WD} (*1*) and dispersion energy E_d^{WD} (*2*), (b) static refractive index n_0 (*1*) and ionicity (*2*).

Table 3. Parameters of Wemple and DiDomenico model, optical band gap, static refractive index and ionicity for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals.

Crystal	E_0^{WD} (eV)	E_d^{WD} (eV)	E_g^{opt} (eV)	n_0^{WD}	f_i^{WD}
Cu_7SiS_5I	5.18	29.30	2.59	2.58	0.42
$(Cu_{0.75}Ag_{0.25})_7SiS_5I$	3.97	20.43	1.99	2.48	0.44
$(Cu_{0.5}Ag_{0.5})_7SiS_5I$	3.94	21.60	1.97	2.54	0.43
$(Cu_{0.25}Ag_{0.75})_7SiS_5I$	3.37	17.90	1.69	2.51	0.43
Ag_7SiS_5I	3.22	15.97	1.61	2.44	0.45

The static refractive index n_0^{WD} for the mixed crystals under investigations were calculated using the equation

$$n_0^{WD} = \left[1 + \frac{E_d^{WD}}{E_0^{WD}}\right]^{1/2}. \qquad (5)$$

The variations of the static refractive index n_0^{WD} on the composition for $(Cu_{1-x}Ag_x)$7SiS_5I mixed crystals are summarized in Table 3. It has been revealed that with the increase of Ag content the static refractive index n_0^{WD} has the tendency to nonlinearly decrease in $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals.

Using the parameters of the WD model, one can calculate such an important parameter for superionic conductors as the ionicity f_i^{WD} [20]:

$$f_i^{WD} = \left[\frac{E_0^{WD}}{E_d^{WD}}\right]^{1/2}. \qquad (6)$$

Values of ionicity for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals are gathered in Table 3. It is shown that at cation $Cu^+ \rightarrow Ag^+$ substitution the ionicity of mixed crystals under investigation has the tendency to nonlinearly increase. This fact is in a good agreement with the recent studies of electrical conductivity in $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals [21]. It is shown in Ref. [21] that the ratio of ionic to electronic components of electrical conductivity increases with the increase of Ag content in $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals.

4. Conclusions

$(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals were obtained by means of the developed growing technique. For this purpose, the vertical zone crystallization method was employed. The optical (diffuse reflection and spectral ellipsometry) studies were performed both on powders and single crystals. The diffuse reflection spectra, spectral dependences of the refractive index and extinction coefficient for $(Cu_{1-x}Ag_x)_7SiS_5I$ mixed crystals were measured in the wide spectral range. The nonlinear decrease of energy pseudogap as well as the nonlinear behavior with a maximum for refractive index have been revealed.

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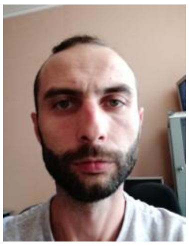

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