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Thermally induced relaxation of optical absorption and photochromic effect in $Bi_{12}SiO_{20}$:Cu crystals

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Spectral kinetics characteristics of photochromic effect and optical destruction of these effect in BSO:Cu crystals have been investigated. It has been shown that both effects are due to the photogeneration and optical destruction of the $Cu_{Bi}{}^{2+}$–O^- and $Bi_{Si}{}^{3+}$–O^- color centers with Cu^{2+} ions in Bi^{3+} positions and Bi^{3+} ions in Si^{4+} positions. The relaxation parameters of these processes have been shown to depend on temperature.

Исследованы спектрально-кинетические характеристики фотохромного эффекта и его оптического стирания в кристаллах BSO:Cu. Показано, что оба эффекта обусловлены процессами фотогенерации и оптического разрушения центров окраски $Cu_{Bi}{}^{2+}$–O^- и $Bi_{Si}{}^{3+}$–O^- с ионами Cu^{2+} в позициях Bi^{3+} и ионами Bi^{3+} в позициях Si^{4+}. Выявлена зависимость релаксационных параметров данных процессов от температуры .

The tasks of optical information recording, treatment, and storage defines the interest in research and modification of optical properties of photorefractive crystals. Among those, the sillenite family crystals $Bi_{12}MO_{20}$ (BMO, with M= Si, Ge, Ti) stand out due to high sensitivity to light from the visible and near IR wavelength range. The sensitivity defines a number of photoinduced effects (photochromic, photorefractive, photoresistive, etc.). That is why the BMO crystals are used as functional media in different types on light-modulating and holographic devices. At the hologram recording, photorefractive and photochromic holographic elements arise in BMO crystals. To date, a considerable attention has been given to experimental and theoretical research of photorefractive effect and also of reversible changes in refractive index and nonlinear interaction of optical rays. In contrast, the photochromism of BMO crystals is studied insufficiently. It is known that the doping influences essentially the optical absorption and spectral characteristics of photochromic effect (PE). In particular, doping by Al, Ga, Ca, Fe and V ions quenches PE, while impurity Cr, Mn and Cu amplify and change its spectral characteristics [1]. The spectral features of PE are connected with intra-center transitions in the doping ions of transitional metals [1, 2]. However, the mechanism of charge states change in those ions under illumination, the kinetics of thermal excitation and optical extinction and also relaxation characteristics of PE, were almost not studied. The complex research results of spectral and kinetic characteristics of PE in $Bi_{12}SiO_{20}$ crystals doped with copper (BSO:Cu) are presented in this work.

The BSO:Cu crystals have been grown by the Czochralski method. Copper concentration in doped crystals was 0.01 wt %. The samples were shaped as plane-parallel plates of 4.6 and 0.2 mm thickness and with large (001) surfaces. The optical transmission spectra $t(\lambda)$ were measured using Cary-5E, Specord M40, Specord NIR61 spectrophotometers in the wavelength range $\lambda = 0.3$ to 3 μm. The temperature was varied in the range $T = 85$ to 700 K. A nitrogen cryostat with quartz windows was used for low temperatures. The absorption spectra $\alpha(\lambda)$ were calculated using the method described in [3].

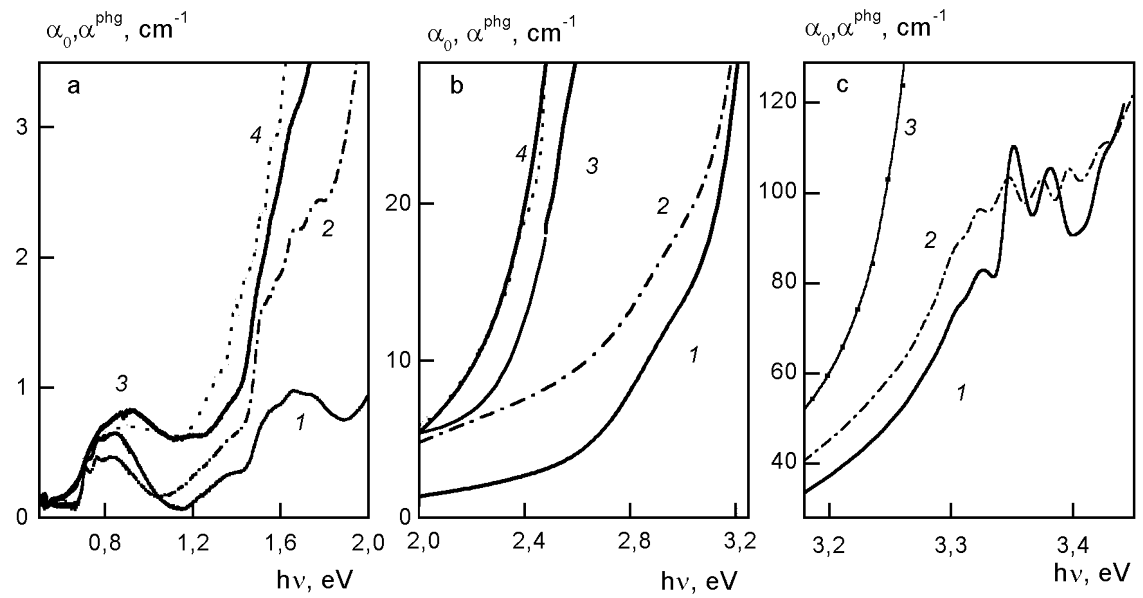

Fig. 1. Spectra of stationary optical absorption $\alpha_0(h\nu)$ (a, *1*, *3*; b, *1*, *3*; c, *1*, *3*) and optical absorption $\alpha^{phg}(h\nu)$ after illumination with energy of quanta $h\nu$ = 2.73 eV (a, *2*, *4*; b, *2*, *4*; c, *2*) at temperature 85 (a, *1*, *2*; b, *1*, *2*; c, *1*, *2*) and 300 K (a, *3*, *4*; b, *3*, *4*; c, 3). Thickness of BSO:Cu crystal d = 0.46 (a, b) and 0.02 cm (c).

The PE was excited by light pulses with λ = 480–450 nm (dark blue) of $t = 1$ s duration (at the same quantum energy $h\nu$). The PE was quenched optically by illuminating with light of λ = 760–620 nm (red). Excitation and optical destruction (OD) was studied at $T_1 = 85$ and $T_2 = 300$ K. The stationary $\alpha_0(h\nu)$, photoinduced $\alpha_i^{phg}(h\nu)$ absorption and its optical destruction $\alpha_i^{phr}(h\nu)$ spectra were investigated for i^{th} illumination dose. The PE and optical destruction spectra were determined as differences $\Delta\alpha_i^{phg}(h\nu) = \alpha_i^{phg}(h\nu) - \alpha_0(h\nu)$ and $\Delta\alpha_i^{phr}(h\nu) = \alpha_{max}^{phg}(h\nu) - \alpha_i^{phr}(h\nu)$ respectively, where $\alpha_{max}^{phg}(h\nu)$ is the maximal value of photoinduced absorption. Kinetics of PE and OD was represented by dependences $\Delta\alpha_i^{phg}(t)$ and $\Delta\alpha^{phr}(t)$, where t is the total duration of photoactive illumination doses. Influence of temperature on optical absorption (termochromic effect (TE)) was estimated from the $\Delta\alpha_i^{Ti}(h\nu) = \alpha_i^{Ti}(h\nu) - \alpha_0^{To}(h\nu)$ spectra obtained at T_o (initial) and T_i (current) temperature values. Spectra of thermal destruction of color centers were presented as $\Delta\alpha^{\mathrm{TD}}(h\nu) = \alpha_{01}^{To}(h\nu) - \alpha_{02}^{T_o}(h\nu)$, where $\alpha_{01}^{To}(h\nu)$, $\alpha_{02}^{To}(h\nu)$ are stationary spectra prior to and after heating.

In the optical absorption spectra of BSO:Cu crystals, there are four wide bands in the photon energy ranges $h\nu$ = 0.69 to 1.05 eV (A), 1.46 to 1.9 eV (B), 2.5 to 3.1 eV (C, at T = 85 K), and 3.3 to 3.4 eV (D at t = 85 K) (Fig. 1). The presence of Cu ions causes appearance of the A and B absorption bands in near IR region (Fig. 1a), because undoped BSO crystals in these range show considerably weaker and monotonously falling values (at hν reduction) [4]. On the long-wave edge of the A band, an electron vibrational structure must be apparent, because the energy distance between its first two components, $\Delta h\nu$ = 0.0624 eV, corresponds to the frequency of optical longitudinal phonons (ν_{phon} = 506 cm^{-1}) in BSO crystals [6].

The B band contains four components of almost equal in intensity at 85 K, which form a "small comb" structure that is typical of intra-center (d-d) optical transitions in Cu^{2+} ions (electron configuration $3d^9$) centering octahedrons of C_2 local symmetry [5]. In BSO crystals, such situation will be realized when Cu^{2+} ions replace Bi^{3+} ones (Cu_{Bi}^{2+}) located in the center of oxygen pseudo-octahedrons with the symmetry near to C_2. The feature of those pseudo-octahedrons consists in that the centering Cu^{2+} cation (as well as Bi^{3+}) is seven-coordinated, i.e. one of six Cu–O bonds is replaced by two longer bonds. The charge compensation is realized due to the hole centers O^-, however, the excess positive charge of the O^- ion (hole) is not localized but jumps over from one top to other (Fig. 2a).

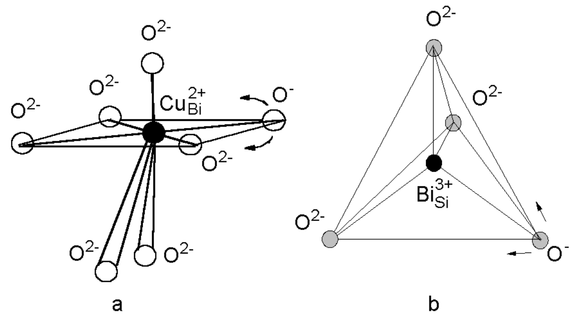

Fig. 2. The oxygen pseudo-octahedron centered by Cu^{2+}_{Bi} ions (a) and oxygen heptahedron in a center with the ions Bi^{3+}_{Si} (b).

The wide C band is in the "shoulder" absorption region of undoped BSO crystals. The intrinsic BSO defects, anti-structural Bi_{Si}^{3+} and Bi_{Si}^{5+} ions replacing Si^{4+} ions in oxygen tetrahedrons, are responsible for absorption in this band (Fig. 1b) [4]. The charge compensations of Bi^{3+}_{Si} ions occurs also, as well as for the Cu_{Bi}^{2+} centers, due to the hole centers O^- with a delocalized hole. In the case of Bi^{5+}_{Si}, the compensations is possible due to cation vacances in the second coordinating sphere or due to that a certain number of the pair centers Bi^{3+}_{Si} and Bi^{5+}_{Si} have (on the average) the charge of Bi^{4+}_{Si} (Fig. 2b).

The intense absorption in the D band adjoining to the fundamental absorption edge of BSO crystals (Fig. 1c) is caused most likely by charge-transfer optical transitions of the "ligand-metal" (L-M) type: $O^{2-} \leftrightarrow Cu_{Bi}^{2+}$. Such transitions is one more typical feature of Cu^{2+} ions with coordination number (c. n.) 6 and octahedron local symmetry C_2 [5]. The absorption in all bands increases as the temperature rises (from 85 to 300 K), thus, the shift of the absorption edge masks the D band structure (Fig. 1). A similar temperature behavior was observed, for example, for Cu^{2+} ions with c. n. 6 in $CrCuCl_3$ crystals [5].

It can be assumed that, along with Cu_{Bi}^{2+} ions, Cu^{3+} ions are present in the BSO crystals that are replacing Bi ions in oxygen pseudo-octahedrons (Cu_{Bi}^{3+}). The charge compensation is not required here. Theoretical estimations show that optical transitions in Cu_{Bi}^{3+} ($3d^8$) ions, located in the octahedron give three wide (about 0.31 to 0.37 eV halfwidth) absorption bands: A′, B′, and C′ [6]. From experimental spectra of transitional metal ions with $3d^8$ electron configuration, spectral absorption of these bands has been determined: $h\nu$ = 0.94 to 1.07 (A′), 1.6 to 1.8 (B′), and 2.98 to 3.18 eV (D′) [6].

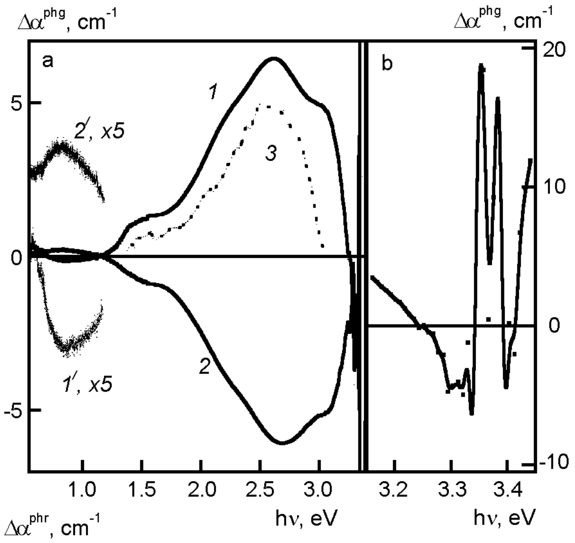

Fig. 3. Spectra of photoinduced absorption $\Delta\alpha^{phg}(h\nu)$ after illumination at quantum energy $h\nu$ = 2.73 eV (a, *1*, *3*, *1′*; b) and optical destruction of photochromic effect $\Delta\alpha^{phg}(h\nu)$ after illumination at quantum energy $h\nu$ = 1.63–1.98 eV (a, *2*, *2′*) at 85 K. The curves *1′* and *2′* are fragments of curves *1* and *2* displaced along vertical axis by +1 and −1, respectively.

As the A band in the absorption spectra of BSO:Cu crystals corresponds to the A′ band, it can be supposed that to be due to transitions in Cu_{Bi}^{3+} ions with c. n. 6. B′ and D′ bands get (or are partly overlapped) into the B and D bands of Cu_{Bi}^{2+} ions with c. n. 6. In tetrahedral crystalline field (replacing of ions of Si^{4+} by Cu^{3+} ones), the Cu^{3+} ion absorption bands are considerably shifted towards IR region of the BSO crystal lattice absorption, and should not be observed. Entering of Cu^+ ions instead of Si^{4+} and Bi^{3+} is unlikely due to of large distinctions in charges and ionic radius.

The PE results in increased absorption in the B, C bands (Fig. 1a, b, Fig. 3a), reduced A band absorption (Fig. 1a, Fig. 3a) and redistribution in the D band (Fig. 1d, Fig. 3b). The band structure of PE $\Delta\alpha^{phg}(h\nu)$ spectra is represented in Fig. 3. It is to note also that the PE can be essentially completely optically destruction under illumination in the 0.4 to 1.5 eV range, i.e., the stationary absorption spectra are reproducible (Fig. 3). The temperature increase from 85 to 300 K results in decreased PE in A and B bands related to Cu ions (Fig. 1a, b,

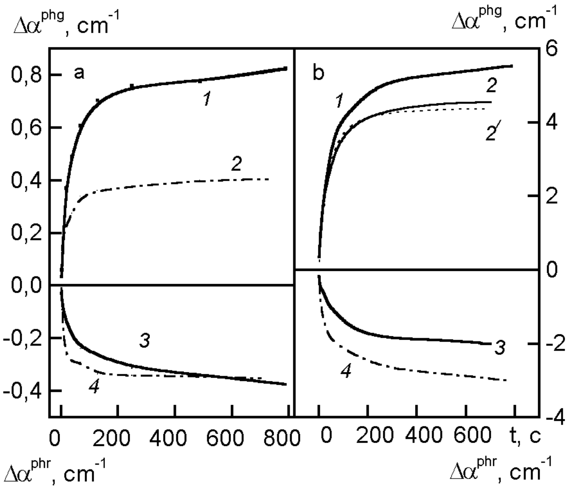

Fig. 4. Kinetic dependences of photogeneration $\Delta\alpha^{phg}(t)$ (a, *1*, *2*; b, *1*, *2*) and partial optical destruction $\Delta\alpha^{phr}(t)$ (a, *3*, *4*; b, *3*, *4*) of color centers Cu_{Bi}^{2+}–O^- (a) and Bi_{Si}^{3+}–O^- (b) in the absorption band with $h\nu = 2.73$ and 1.63 eV at 85 (a, *1*, *3*; b, *1*, *3*) and 300 K (a, *2*, *4*; b, *2*, *4*). The calculated $\Delta\alpha_{call}^{phg}(t)$ dependence for photogeneration in the band $h\nu = 2.73$ eV (b, *2'*).

Fig. 3a). The PE in the C band (Fig. 1b, Fig. 3a) drops similarly to that in undoped BSO crystals [4].

The photoinduced decrease of absorption in A and B bands is accompanied by increase of absorption in B and D bands of $\Delta\alpha^{phg}(h\nu)$ spectra (Figs. 1, 3). This allows us to assume that the PE is caused by the change of ratio between the color centers connected with change of their charge state according to the schemes: $Cu_{Bi}^{3+} + e \rightarrow Cu_{Bi}^{2+}$, $Bi_{Si}^{5+} + 2e \rightarrow Bi_{Si}^{3+}$. The charge neutrality is provided in both cases by simultaneous formation of the hole centers O^-; thus, it is just the pair centers Cu_{Bi}^{2+}–O^- and Bi_{Si}^{3+}–O^- with a hole delocalized at the vertices of oxygen pseudo-octahedrons and tetrahedrons, respectively, that are responsible for PE. The suggested mechanism of PE and OD (optical destruction) involving two types of complex centers is confirmed by the consideration of experimental $\Delta\alpha^{phg}(t)$, $\alpha^{phr}(t)$ dependences characterizing the kinetics of the formation and destruction processes as well as by temperature influence on the relaxation parameters (Fig. 4).

Each of the dependences presented in the semilogariphmic scale as $\ln(1 - \Delta\alpha^{phg}/\Delta\alpha_{max}^{phg}) = f_1(t)$ and $\ln(\Delta\alpha^{phr}/\Delta\alpha_0^{phr}) = f_2(t)$, where $\Delta\alpha_{max}^{phg}$ corresponds to the $\Delta\alpha^{phg}(t)$ value at $t \rightarrow \infty$, and $\Delta\alpha_0^{phr}$, the $\Delta\alpha^{phr}(t)$ value at $t = 0$, includes three linear sections for both types of centers. This makes it possible to describe those as follows:

$$\Delta\alpha^{phg} = \Delta\alpha_{\max}^{phg}[1 - \sum_{i=1}^{3} a_{gi}\exp(-t/\tau_{gi})], \qquad (1)$$

$$\Delta\alpha^{phr} = \Delta\alpha_0^{phr}\sum_{i=1}^{3} a_{ri}\exp(-t/\tau_{ri}). \qquad (2)$$

In other words, each of both processes include three additive exponential components, where a_{gi}, a_{ri} are the share holding

Table 1. The relaxation of parameters characterizing the photogeneration and optical destruction of color centers in BSO:Cu crystals.

The spectral position of color center absorption band, $h\nu$, eV	Effect	T_1 = 85 K			T_2 = 300 K		
		Relaxation time / Share of relaxer			Relaxation time / Share of relaxer		
		$\frac{\tau_1,\ s}{a_1}$	$\frac{\tau_2,\ s}{a_2}$	$\frac{\tau_3,\ s}{a_3}$	$\frac{\tau_1,\ s}{a_1}$	$\frac{\tau_2,\ s}{a_2}$	$\frac{\tau_3,\ s}{a_3}$
Cu_{Bi}^{2+}, $h\nu \approx 1.63$ eV	PE	$\frac{27}{0.58}$	$\frac{40}{0.28}$	$\frac{961}{0.14}$	$\frac{5.4}{0.32}$	$\frac{31}{0.33}$	$\frac{311}{0.35}$
	OD	$\frac{2.5}{0.66}$	$\frac{400}{0.13}$	$\frac{1600}{0.21}$	$\frac{11}{0.61}$	$\frac{389}{0.36}$	$\frac{1257}{0.03}$
Bi_{Si}^{3+}, $h\nu \approx 2.73$ eV	PE	$\frac{8}{0.17}$	$\frac{49}{0.65}$	$\frac{226}{0.18}$	$\frac{5}{0.23}$	$\frac{12.6}{0.35}$	$\frac{250}{0.42}$
	OD	$\frac{13.4}{0.14}$	$\frac{100}{0.48}$	$\frac{1800}{0.38}$	$\frac{12.7}{0.29}$	$\frac{167}{0.62}$	$\frac{1600}{0.09}$

Note: relaxation time τ_1, τ_2, τ_3 and a_1, a_2, a_3 averaged over the time τ_{1g}, τ_{2g}, τ_{3g}, a_{1g}, a_{2g}, a_{3g} for PE and τ_{1r}, τ_{2r}, τ_{3r}, a_{1r}, a_{2r}, a_{3r} for OD.

constants; τ_{gi}, τ_{ri}, relaxation times of the components defining the photogeneration (a_{gi}, τ_{gi}) and destruction (a_{ri}, τ_{ri}) processes of the color centers. The quantities a_{gi}, a_{ri} and τ_{gi}, τ_{ri} have been found using the successive discrimination of the components, starting from the most slow ones (Table 1). The results obtained allow to get the calculated dependences $\Delta\alpha_{call}^{phg}(t)$ and $\Delta\alpha_{call}^{prn}(t)$ defined by expressions (1) and (2), respectively, and reproducing the initial experimental $\Delta\alpha^{phg}(t)$ and $\Delta\alpha^{prn}(t)$ dependences to within ≤5 % (Fig. 4a).

Further, the following general features of relaxation processes are to be noted: (1) For both center types, $\mathrm{Cu_{Bi}}^{2+}$–O^- and $\mathrm{Bi_{Si}}^{3+}$–O^-, typical are large values of relaxation time τ_{gi} and τ_{ri} (Table 1) exceeding the time (~10^{-6} s) necessary for excitation of electron subsystem of the centers; (2) Both center types are characterized by sets consisting of three different relaxation times τ_{gi} (τ_{g1}, τ_{g2}, τ_{g3}) and τ_{ri} (τ_{r1}, τ_{r2}, τ_{r3}), that evidences three relaxation processes with different share contribution to be involved in photogeneration and optical destruction of color centers, the shares being defined by values of a_{gi} (a_{g1}, a_{g2}, a_{g3}) and a_{ri} (a_{r1}, a_{r2}, a_{r3}), respectively (Table 1).

Distinction of the relaxation processes related with $\mathrm{Cu_{Bi}}^{2+}$–O^- and $\mathrm{Bi_{Si}}^{3+}$–O^- centers is seen both at low (T_1 = 85 K) and room temperature (T_2 = 300 K) and consist in what follows.

1. At T_1 = 85 K, the photogeneration of $\mathrm{Cu_{Bi}}^{2+}$–O^- centers is in general a more slow process as compared to the $\mathrm{Bi_{Si}}^{3+}$–O^- center generation, with shorter relaxation times of fast (τ_{g1}) and slow (τ_{g3}) components contributing mainly (72 %) to the PE. The $\mathrm{Bi_{Si}}^{3+}$–O^- centers differ yet in that the main contribution (65 %) to their formation is provided by the component with an "intermediate" relaxation time (τ_{g2}) (Table 1).

2. Temperature rise up to T_2 = 300 K reveals a trend to decrease the relaxation time and equalizing of share parts of various contributions to PE (Table 1).

3. In contrast to PE, the decay of $\mathrm{Cu_{Bi}}^{2+}$–O^- — centers is a more fast processes as compared to $\mathrm{Bi_{Si}}^{3+}$–O^- — ones, the fast (τ_{r1}) and slow component (τ_{r3}) providing the main contribution to this process (Table 1).

4. The temperature rise up to T_2 = 300 K results in acceleration of the optical destruction processes for both color center types, the contribution of slow processes decreases considerably (<1 %) (Table 1).

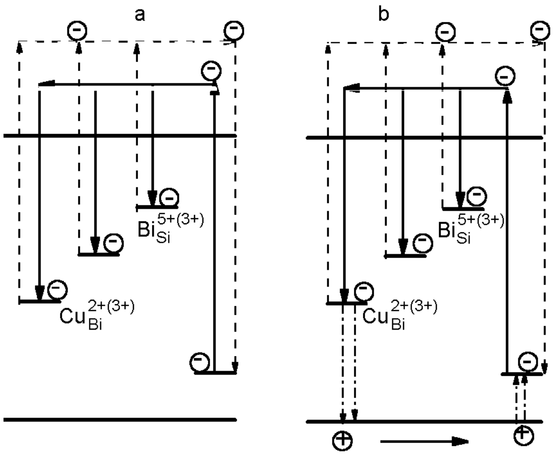

Fig. 5. Scheme of electron transitions responsible for the charge change of $\mathrm{Cu_{Bi}}^{2+(3+)}$ and $\mathrm{Bi_{Si}}^{3+(5+)}$ ions at excitation (continuous lines) and optical destruction (dashed lines) of photochromic effect without (a) and with (b) involvement of thermoinduced exchange by the charge carriers between the conductivity band and the valence band (double dotted lines) at 85 (a) and 300 K (b).

To explain the results obtained, it is to take into account that the BSO and BSO:Cu crystals are highly doped compensated wideband semiconductors, i.e. these crystals contain ionized donors and acceptors due to intrinsic defects. In the photoconductivity spectral regions corresponding to dark blue and red light, a sharp photoconductivity increase by 1 and 5 to 6 decimal orders, respectively, is observed. This is due to electron transitions from non-ionized donor levels and ionized acceptor ones to the conductivity band [7, 8]. Taking into account that the PE is excited by dark blue light and is destruction by red one for undoped BSO crystals and those doped with different impurities [1], we believe the photogeneration and optical destruction processes of color centers with the electron transitions shown in Fig. 5.

Moreover, we assume that the fast components of generation (τ_{g1}, τ_{g2}) and destruction (τ_{r1}, τ_{r2}) of color centers providing the main contribution (about 70 to 80 %) in the intensity change of B ($\mathrm{Cu_{Bi}}^{2+}$–O^- centers) and C ($\mathrm{Bi_{Si}}^{3+}$–O– centers) induced absorption bands define directly the recharging $\mathrm{Cu_{Bi}}^{3+}\leftrightarrow\mathrm{Cu_{Bi}}^{2+}$ and $\mathrm{Bi_{Si}}^{5+}\leftrightarrow\mathrm{Bi_{Si}}^{3+}$ ions at low temperatures. The recharging is provided due to optical emptying of ionized acceptors (photogeneration, illumination with dark

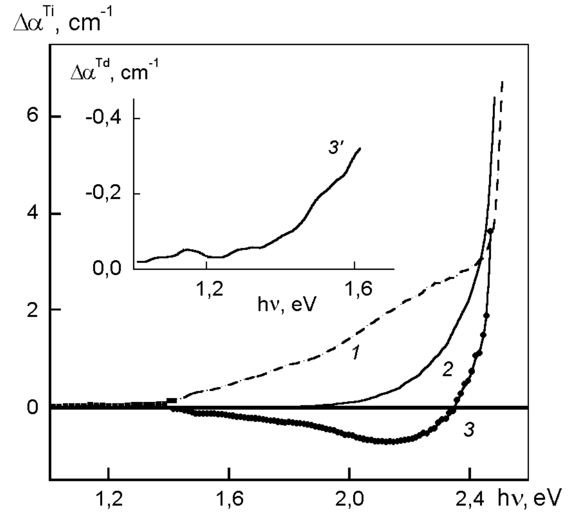

Fig. 6. Spectra of thermoinduced optical absorption $\Delta\alpha^{Ti}(h\nu)$ after heating from 300 to 620 K (*1*) and cooling from 620 to 300 K (*2*); thermal discoloration spectra $\Delta\alpha^{TD}(h\nu)$ of BSO:Cu crystals after heating-cooling cycle (*3*, *3'*).

blue light) or non-ionized donors (optical excitation with red light). The emptying of acceptors or donors is accompanied by the diffusion transport of charge carriers (electrons) in the conductivity band and re-trapping in the local levels of the forbidden gap formed by the Cu_{Bi}^{3+} and Bi_{Si}^{5+} ions (Fig. 5a). The low mobility of electrons in the BSO ($\mu \sim 10^{-5}$ cm^2/V·c) explains the large values τ_{g1}, τ_{g2} and τ_{r1}, τ_{r2}.

The decrease of τ_{g1}, τ_{g2} and τ_{r1}, τ_{r2} at the temperate increase to $T_2 = 300$ K against the sharp decrease of photoconductivity [7, 8], decrease of PE and OD effects can be explained by involvement of competitive thermal ionization processes of the acceptors and donors (Fig. 5b). Due to these processes, at heating to $T = 400–450$ K, we observed TE (additional thermally induced coloration), but a further temperature increase caused thermal decomposition of color centers (Fig. 6).

Of a particular interest is the presence of slow components (τ_{g3} and τ_{r3}) in photogeneration and optical decay processes of $Cu_{Bi}^{2+}–O^-$ and $Bi_{Si}^{3+}–O^-$ centers. Perhaps the slow processes are thermally activated association (dissociation) of the Cu_{Bi}^{2+}, $Bi_{Si}^{3+}–O^-$ defects into $Cu_{Bi}^{2+}–O^-$ and $Bi_{Si}^{3+}–O^-$ centers. The association (dissociation) is accompanied by equilibrium state establishment in the electron subsystem due to strong electron-phonon interaction in sillenite crystals of [9]. In addition, the $Cu_{Bi}^{2+}–O^-$ and $Bi_{Si}^{3+}–O^-$ centers can be oriented (disoriented) under illumination.

The physical mechanisms of such processes can be various for complex defects in various crystals. In particular, the axial $Cu_{Bi}^{2+}–O^-$ centers are oriented in the $KTaO_3$ crystals under action of polarized light with time constant of about 1620 s at 245 K [10]. The relaxation time of the mentioned long-time relaxation processes decreases with increasing temperature, that is in agreement with the experiment (Table 1). Stability of the $Cu_{Bi}^{2+}–O^-$ and $Bi_{Si}^{3+}–O^-$ centers and some increase in the PE band intensity is provided in both cases.

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Термоіндукована релаксація оптичного поглинання та фотохромний ефект у кристалах $Bi_{12}SiO_{20}$:Cu

Т.В.Панченко, К.Ю.Стрілець

Досліджено спектрально-кінетичні характеристики фотохромного ефекту та його оптичне стирання у кристалах BSO:Cu. Показано, що обидва ефекта обумовлені процесами фотогенерації та оптичного руйнування центрів забарвлення Cu_{Bi}^{2+}–O^- та Bi_{Si}^{3+}–O^- з іонами Cu^{2+} у позиціях Bi^{3+} і іонами Bi^{3+} у позиціях Si^{4+}. Виявлено залежність релаксаційних параметрів даних процесів від температури.