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# Mechanisms of intrinsic and impurity electron-hole trapping centers in K_3 Y 〖(SO_4)〗 _3-Eu and LiY 〖(SO_4)〗 _2-Eu double cation phosphors

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ORIGINAL STUDY

# Mechanisms of Intrinsic and Impurity Electron-hole Trapping Centers in $K_3Y(SO_4)_3 - Eu$ and $LiY(SO_4)_2 - Eu$ Double Cation Phosphors

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## Abstract

**The mechanisms of creation of intrinsic and impurity trapping centers, the interaction of which forms combined or induced electronic states under the conduction band, have been studied using optical and thermal activation spectroscopy. It is shown that electron-hole trapping centers are created when free electrons are trapped by impurities and ions of the $SO_4^{2-}$ matrix, as well as a result of charge transfer from the excited $SO_4^{2-}$ anion to impurities and to neighboring ions by the reaction ($O^{2-} - Eu^{3+}$) and ($O^{2-} - SO_4^{2-}$). The hole component of the trapping center is formed when holes are localized above the valence band. During relaxation, the combined emission state decays with the transfer of energy from its own matrix to the emitters.**

***Keywords:*** Electron, Hole, Trapping center, Luminescence, Spectroscopy, Recombination

## 1. Introduction

The mechanisms of creation of intrinsic and impurity trapping centers in sulfates of alkaline, alkaline earth, rare earth metals and phosphates with double cations activated by $Eu^{3+}$ ions are being widely studied over last decades. Inorganic materials doped with $Eu^{3+}$ ions are successfully used as thermoluminescent dosimeters, phosphors of orange-red emission used in lighting, light-emitting diodes, electroluminescent and plasma displays [1–9].

In Ref. [10], the mechanisms of interaction of ionizing emission of phosphate $Ca_3(PO_4)_2$, activated by ions $Eu^{3+}$ and $Dy^{3+}$ were studied. It has been shown that as a result of excitation by photons with an energy of about 5 eV, emissions arise at 593 nm and 615 nm, associated with the $5D_0 - 7F_1$ and $5D_0 - 7F_2$ transitions in the $Eu^{3+}$ ion. The mechanism of emission proceeds as follows: during photon irradiation at 243 nm, $Ca_3(PO_4)_2 - Eu$ undergoes a charge transfer from the 2p state of the anion ($O^{2-} \rightarrow E^{3+}$) to the $Eu^{3+}$ ion. Simultaneously, $Eu^{2+}$ should be formed. The resulting electron from this state relaxes to the excited $5D_0$ state and then to the ground $4f^7$ state, emitting light from the $Eu^{3+}$ ion.

Based on studies of $SrSO_4 - Eu$ phosphors [11] annealed at temperatures ranging from 200 °C to 1000 °C, it has been shown that the emission arising from $Eu^{2+}$ and $Eu^{3+}$ ions, as well as the defect-induced emission in the host lattice resulted from a UV excitation. During this process, an electron from the ground state $4f^7$ was excited to the $4f^6 5d^1$ state of the $Eu^{2+}$ ion. After relaxation, intracenter emission was appeared at 378 nm of the $Eu^{2+}$ ion. Similarly, during the radiative recombination of electrons from the excited state $5D_0$ of the $Eu^{3+}$ ion with holes located in the ground $^7F_{0,1,2}$ state, emissions of the $Eu^{3+}$ ion appeared with maxima at 584 nm, 591 nm and 612 nm.

After annealing, the intensity of the blue emission at 378 nm increased sharply 30 times, and the intensity of the long-wave emission gradually decreased. It has been assumed that the reduction of $Eu^{2+}$ and $Eu^{3+}$ ions

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was related to the formation of an oxygen vacancy VO during annealing.

In Ref. [12] the luminescence of $KSrPO_4 - Eu^{3+}$ at room temperature has been studied. According to the authors' assumption, the 610 nm emission band arising during synchrotron irradiation with a photon energy of 7.65 eV corresponds to the excitons arising in the phosphate group. An intense wide excitation band of about 272 nm should correspond to the charge transfer electron transition in the phosphate group $O^{2-} - Eu^{3+}$ impurities. In the spectral range below 300 nm, a number of lines are observed due to $f - f$ transitions in $Eu^{3+}$.

In Ref. [13] the phosphor $Ba_2Y_5B_5O_{17} - Eu^{3+}$ has been obtained using high-temperature solid-phase reaction. All materials demonstrated bright red luminescence when excited by UV in blue emission. The emission spectra were dominated by the emission corresponding to the transitions $5D_0 - 7F_2$ and $5D_0 - 7F_4$ at 615 and 705 nm. Phosphor demonstrated high thermal stability. The compounds doped with 5%, 10% and 25% $Eu^{3+}$ showed quantum efficiency close to 100% with excited emission with a wavelength of 394 nm.

The gamma-dosimetric properties of the radio-photoluminescent phosphor $CaSO_4 - Eu^{3+}$ with a new reading device were considered in Ref. [8]. The transformation of $Eu^{3+}$ into $Eu^{2+}$ caused by the gamma irradiation has been studied in this phosphor using stimulation of a UV LED with a wavelength of 320 nm. A reproducible gamma radiation dose response within ±20% is achieved in the synthesized phosphors. The radio-photoluminescence (RPL) phosphor has advantages in terms of obtaining fast and multiple cumulative measurements of absorbed gamma emission doses in dosimeters.

From a brief review of literature data, it follows that phosphors activated in sulfates and phosphates by $Eu^{3+}$ and $Eu^{2+}$ ions emit intracenter blue emission of the $Eu^{2+}$ ion. As a result of annealing or irradiation with ionizing radiation, the interconversion of $Eu^{3+}$ ions into $Eu^{2+}$ ions was observed.

The current study is focused on the mechanisms of formation of impurity and intrinsic electron and hole trapping centers in sulfates with complex cations $K_3Y(SO_4)_3 - Eu$ and $LiY(SO_4)_2 - Eu$. We found the formation of combined electron-emitting states from intrinsic and impurity trap centers and their decay, resulting in energy transfer from the matrix with the $Eu^{3+}$ emitter.

This work also examines the nature of intracenter emission from $Eu^{2+}$ and $Eu^{3+}$ impurities, as well as a new induced emission center that arises upon excitation by photons with energies exceeding the band gap in $K_3Y(SO_4)_3 - Eu$ and $LiY(SO_4)_2 - Eu$ phosphors.

## 2. Objects and methods of research

The studied compounds were synthesized using the slow evaporation method. To prepare the compound, lithium sulfate and yttrium sulfate were used as starting materials. $Li_2SO_4$ and $Y_2SO_4$ (Sigma Aldrich) were mixed in a quantity of 0.8 g and 0.4 g respectively. Double distilled water was added to the mixed $LiY(SO_4)_2$ powder. 0.28 g of $Eu_2O_3$ was added to the resulting solution. After making sure that the solution was transparent, each lanthanide was added and mixed one by one, checking the transparency of the solution. In a separate container, $Eu_2O_3$ was dissolved in a deionized solution of 20 ml at a temperature of 35–40 °C. Then both solutions were mixed at 35–40 °C for 1 h.

$LiY(SO_4)_2 - Eu$ was dried at 550 °C in air. The resulting powders were pressed into tablets with a diameter of 8–9 mm and a thickness of 1–2 mm. It was found that the optimal concentration of the Eu impurity in the and $LiY(SO_4)_2$ matrix is 0.3 mol%. The resulting Eu impurity in a concentration of 0.3 mol% turned out to be the optimal for spectroscopic measurements. A similar method was used for compound $K_3Y(SO_4)_3 - Eu$.

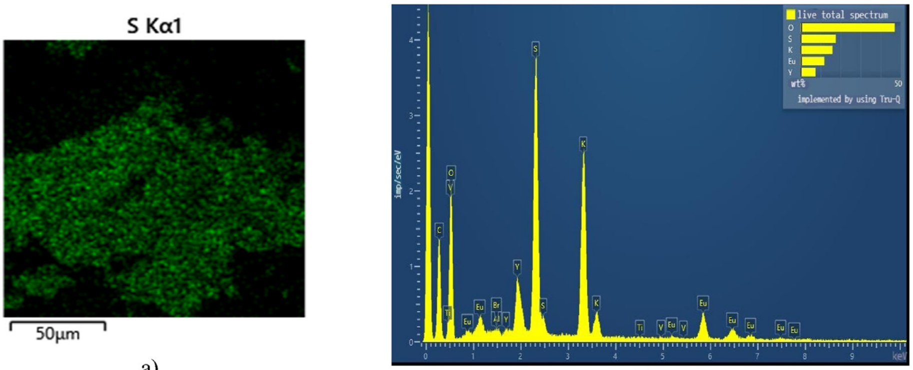
The emission and excitation spectra (excitation energy of 1.5–6.2 eV) were acquired using the CM2203 spectrofluorometer (Minsk, Belarus). Measurements of radiation at low temperatures (77 K) were performed using a liquid nitrogen cryostat. All the measurements were performed in a $10^{-4}$ Pa vacuum.

The same cryostat was used to measure thermally stimulated luminescence (TSL), a good method to detect trapping centers in dielectrics. To obtain TSL curves, the compounds under investigation were cooled to the temperature of liquid nitrogen (77 K). Under these conditions, the crystal is excited for some time with UV or X-ray radiation. After the excitation is stopped, the crystal is heated at a constant rate of 0.2 °C/s, and the intensity of the emitted radiation is measured.

## 3. Results

In this study, we investigate the nature of the intracenter emission of the $Eu^{3+}$ impurity and new Raman emissions resulting from the interaction of impurities with the electronic excitations of the matrix.

Fig. 1 shows the results of the analysis of the morphology and chemical composition of the powder $K_3Y(SO_4)_3 - Eu$ compound. We used a scanning electron microscope (SEM) equipped with an energy-dispersive X-rays spectrometer (EDX). The grains have different sizes of approximately 5–200 μm (Fig. 1a). The EDX spectrum (Fig. 1b) confirms the chemical composition of a $K_3Y(SO_4)_3$ compound and the Eu content of 0.3 mol%.



a)

b)

*Fig. 1. The results of the SEM-EDX analysis of the* $K_3Y(SO_4)_3 - Eu$ *powder. S* $K_{\alpha 1}$ *elemental map (a), EDX spectrum from the area of 150 × 150* $\mu m$*.*

The emission spectra of the $K_3Y(SO_4)_3 - Eu$ phosphor irradiated with photons with an energy of 6.2 eV at 77 K (curve 1) and at 300 K (curve 2) are shown in Fig. 2. It is clear that intracenter emission bands of the impurity $Eu^{3+}$ appear at 1.7 eV, 1.8 eV, 1.9 eV and 2.06 eV at 300 K. When the phosphor is irradiated at 77 K, new emission bands appear at 2.4 eV, 2.6 eV, 2.9 eV, 3.0 eV and 3.1 eV. The intensity of intracenter emission simultaneously decreases by 30%. A similar intracenter emission of the $Eu^{3+}$ impurity at 2.0 eV, 2.02 eV, 2.09 eV and 1.76 eV found previously [10,11,13] in the $CaSO_4 - Eu, SrSO_4 - Eu$, $Ba_2Y_5B_5O_{17} - Eu^{3+}$ phosphors. A negligible spectral shift upon excitation in the spectral region of about 5 eV at 300 K, as well as blue UV radiation has been observed in comparison with our phosphors.

To clarify the nature of new emissions arising in the spectral region of 3.1 eV and 2.9 eV in the studied phosphors irradiated at 77 K, excitation spectra were measured. Excitation spectra of the $K_3Y(SO_4)_3 - Eu$ phosphor pre-irradiated with photons with an energy of 6.2 eV with radiation-induced trapping centers at 3.1 eV (curve 1) and 2.9 eV (curve 3) at 77 K are shown in Fig. 3. The appearance of new emission bands was also discovered by other authors [14—17] upon irradiation with UV radiation and X-rays, where free electron-hole pairs are created. The recombination emission bands are excited at photon energies of 3.9—4.0 eV and 4.4—4.5 eV in the transparency region of the phosphor matrix. The excitation spectra of recombination emission at 3.1 eV (curve 2) and 2.9 eV (curve 4) in the $LiY(SO_4)_2 - Eu$ phosphor at 77 K are shown in Fig. 3 as well. The recombination emission in $LiY(SO_4)_2 - Eu$ is also excited at photon energies of 3.9—4.0 eV and 4.4—4.5 eV in the spectral region of phosphor transparency.

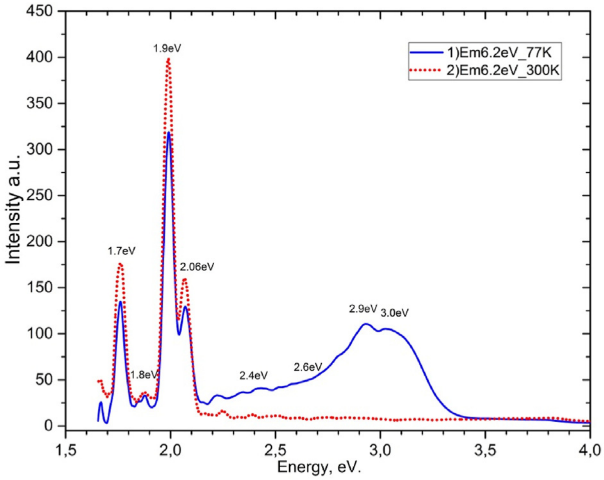


*Fig. 2. Emission spectrum of* $K_3Y(SO_4)_3 - Eu$ *irradiated by photons with an energy of 6.2 eV at 77 K (curve 1) and at 300 K (curve 2).*

It should be noted that the recombination emission is excited in $LiY(SO_4)_2 - Eu$ phosphors at photon energies of 5.5—6.2 eV, where it creates electron and hole trapping centers as a result of energy transfer from the excited anionic complex to $Eu^{3+}$ impurities or a neighboring ion or when electrons are trapped by impurities or anionic complexes.

It is assumed that this recombination emission is created in phosphors at photon energies of 5.5—6.2 eV, where electron and hole trapping centers are created.

The formation of the $Eu^{2+}$ centers in irradiated $CaSO_4 - Eu$ phosphors during the capture of free electrons by the $Eu^{3+}$ centers can be described by the following reaction: $Eu^{3+} + e^- \rightarrow Eu^{2+}$ [18]. In this case, the hole components of electron-hole pairs are localized as $SO_4^-$- radicals above the valence band (see band diagram, Fig. 8). The formation of electron trapping centers $SO_4^{3-}$ has been revealed in irradiated sulfates using an electron paramagnetic resonance (EPR) technique [19]. This is a result of electron localization in the anionic complex $SO_4^{2-}$ and can be described by the following reaction: $SO_4^{2-} + e^- \rightarrow SO_4^{3-}$. Considering

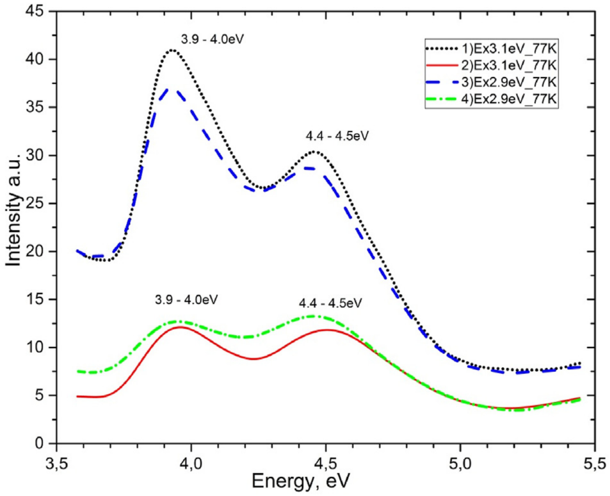


*Fig. 3. Excitation spectra for $K_3Y(SO_4)_3 - Eu$ for emission bands at 3.1 eV (curve 1) and 2.9 eV (curve 3) at 77 K and for $LiY(SO_4)_2 - Eu$ for emission bands at 3.1 eV (curve 2) and 2.9 eV (curve 4) at 77 K.*

these experimental results, we can assume that the electron trapping centers in our phosphor should arise according to the similar reactions as follows: $SO_4^{2-} + e^- \rightarrow SO_4^{3-}$, $Eu^{3+} + e^- \rightarrow Eu^{2+}$. In this case, the electron-hole trapping centers of $SO_4^{3-} - SO_4^-$ and $Eu^{2+} - SO_4^-$ should appear.

Thus, recombination or tunnel emission of 3.1 eV and 2.9 eV arising after irradiation of the phosphor corresponds to local states located in the matrix transparency regions, that is, electron-hole trapping centers. To prove that the regions of the photon energies of 3.9—4.0 eV and 4.4—4.5 eV, which excite recombination emission, correspond to the excitation spectrum of the trapping centers, the reverse procedure was applied. That is, when we use excitation energies of 3.9—4.0 eV and 4.4—4.5 eV, a recombination emission of 3.1 eV and 2.9 eV should be excited.

Fig. 4 shows the recombination emission of 3.1 eV and 2.9 eV photons being excited with energies of 3.9 eV (curve 1) and 4.4 eV (curve 2) in $K_3Y(SO_4)_3 - Eu$ and $LiY(SO_4)_2 - Eu$ phosphors with radiation-induced trapping centers. Phosphors were pre-irradiated with UV photons to create trapping centers at 77 K. With such excitation, in addition to recombination emission, impurity emission appears. This will be discussed below.

Excitation spectrum of intracenter emission at 2.0 eV (curve 2) and 2.06 eV (curve 1) at 77 K of the phosphor $K_3Y(SO_4)_3 - Eu$ with induced electron-hole intrinsic and impurity trapping centers is shown in Fig. 5. The intracenter emissions at 2.0 eV and 2.06 eV are excited in the intracenter transitions of the $Eu^{3+}$ ion and are excited due to the charge transfer from the anion complex to the $Eu^{3+}$ ion ($O^{-2} - Eu^{3+}$). During the charge transfer, $Eu^{2+}$ ion can be formed. In subsequent relaxation processes, the electron localized on the $Eu^{2+}$ ion may relax from the excited $D$ state and then recombine with a hole in the $7F_j$ state, resulting in a photon emission from the $Eu^{3+}$ ion.

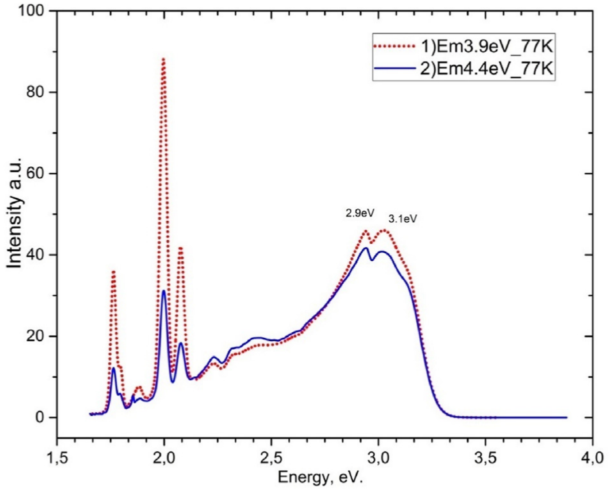


*Fig. 4. Emission spectrum of $K_3Y(SO_4)_3 - Eu$ after excitation at 3.9 eV (curve 1) and 4.4 eV (curve 2) at 77 K.*

The temperature dependence of the intensity of the new emission at 3.1 eV and 2.9 eV in the $K_3Y(SO_4)_3 - Eu$ phosphor pre-induced with UV photon emission of electron and hole trapping centers is shown in Fig. 6. The temperature dependencies of the intracenter emission intensity at 2.06 eV (curve 1) and 2.0 eV (curve 2) with radiation-induced electron-hole trapping centers in the temperature range of 77—450 K are shown as well. The intensity of recombination or tunneling emission at 2.9 eV and 3.1 eV in the

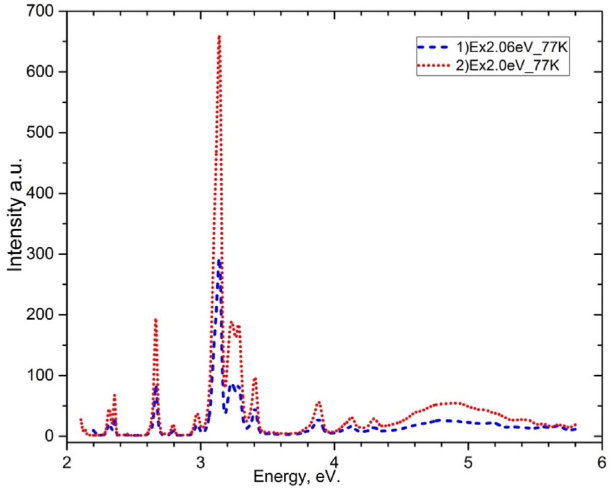


*Fig. 5. Excitation spectrum of $K_3Y(SO_4)_3 - Eu$ for emission bands: at 2.06 eV (curve 1) and 2.0 eV (curve 2) at 77 K.*

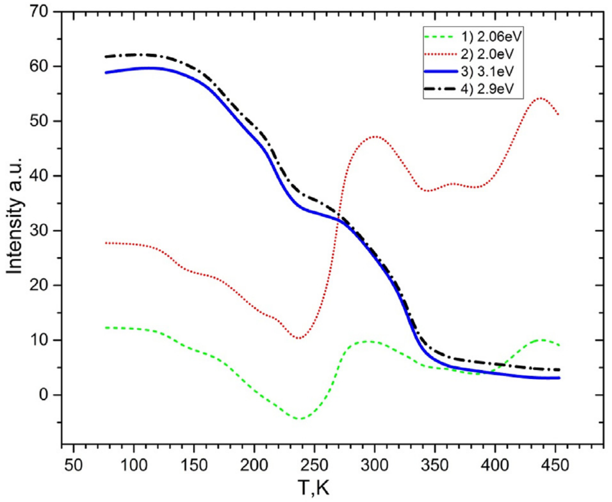


*Fig. 6. The temperature dependence of the intensity of intracenter emission at 2.06 eV (curve 1) and 2.0 eV (curve 2) and recombination emission at 3.1 eV (curve 3) and 2.9 eV (curve 4) in the $K_3Y(SO_4)_3 - Eu$ phosphor.*

temperature range of 150—230 K and of 250—350 K gradually decreases to a minimum value. The intensity of the emission at 2.06 eV and 2.0 eV slowly decreases in the temperature range of 77—230 K and increases rapidly in the temperature range of 250—430 K. This emission is increased due to the $Eu^{2+} \rightarrow Eu^{3+} + e^-$ ionization.

The thermally stimulated luminescence (TSL) of the $LiY(SO_4)_2 - Eu$ phosphor irradiated with X-rays for 5 min at 77 K is shown in Fig. 7. TSL appears in two temperature ranges: of 150—220 K, 250—350 K and 370—430 K, where a new recombination emission state decays at 3.1 eV and 2.9 eV.

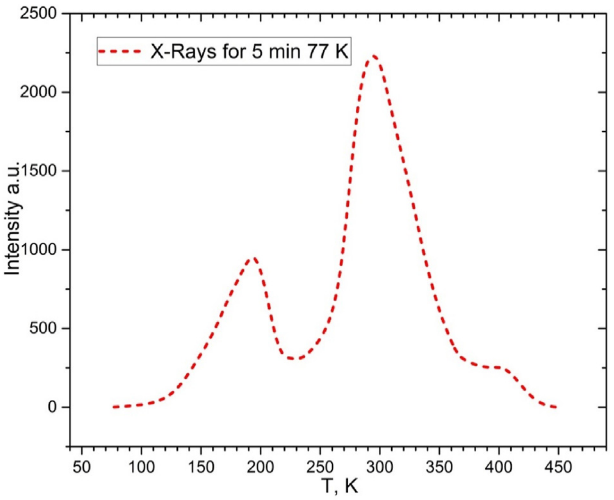


*Fig. 7. TSL spectrum of the $LiY(SO_4)_2 - Eu$ phosphor irradiated with X-rays for 5 min at 77 K.*

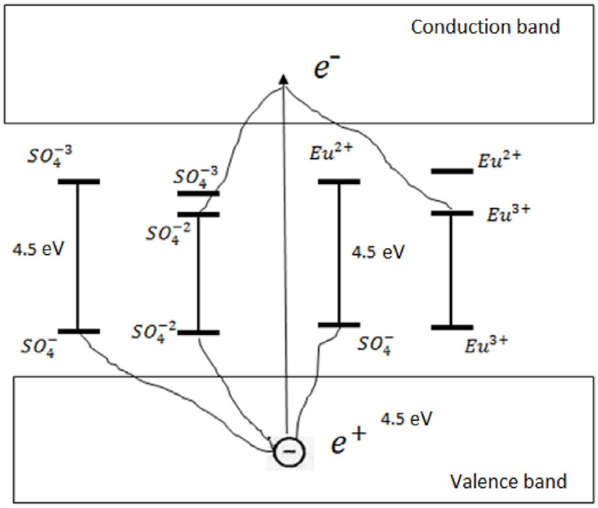


*Fig. 8. Band diagram of $K_3Y(SO_4)_3 - Eu$ phosphors with induced intrinsic and impurity trapping centers.*

## 4. Discussion

Our study was focused on the mechanisms of creation of the intrinsic and impurity electron and hole trapping centers in $K_3Y(SO_4)_3 - Eu$ and $LiY(SO_4)_2 - Eu$ phosphors with double cations with impurities of rare-earth $Eu$ ions. It has been shown previously [20—25] that in alkali and alkaline earth metal sulfates $LiKSO_4, Li_2SO_4, K_2SO_4, CaSO_4$ and $BaSO_4$ activation by impurities such as $Cu^+, Mn^{2+}$ and $Dy^{3+}$ induces electron-hole emission states at 2.95 eV and 3.1 eV. The electronic states of $Li_2SO_4, Na_2SO_4, CaSO_4$ phosphors activated by $Cu^+, Mn^{2+}$ and $Dy^{3+}$ include impurity electron and hole trapping centers [26—31]. In all mentioned sulfates the impurity atoms replace the alkali or alkaline earth atoms.

In $K_3Y(SO_4)_3 - Eu$ phosphors, the $Eu^{3+}$ ion replaces both $K^+$ and $Y^{3+}$ ions in simultaneously. This substitution creates new emission states at 2.9 eV and 3.1 eV. In $LiY(SO_4)_2 - Eu$ phosphors, new induced electron-emitting states are formed at 2.85 eV and 3.1 eV. Because the $Li^+$ cations are small, cation vacancies can form during the $Li^+ \rightarrow Eu^{3+}$ substitution in the first coordination sphere of the $Eu^{3+}$ ions for charge compensation. In this case, the $Eu^{2+}$ vacancywill be perturbed by the nearest neighboring vacancy. The shift of the newly created electron-emitting state from the previously known position of 2.95 eV to the position of 2.85 eV may be related to this perturbation.

In the first stage of the study, we showed that the same recombination emission appears in $K_3Y(SO_4)_3 - Eu$ at 3.1 eV and 2.9 eV and in $LiY(SO_4)_2 - Eu$ at 2.85 eV. The difference in the energy position of the emission bands in these two phosphors is associated with the different environment of the emitters: $Eu^{2+}$ without a vacancy in $K_3Y(SO_4)_3 - Eu$ and $Eu^{2+}$ with a neighboring vacancy in $LiY(SO_4)_2 -$ $Eu$. We have shown experimentally that new radiative electronic states at 2.9 eV and 3.1 eV in $K_3Y(SO_4)_3 - Eu$ and at 2.85 eV in $LiY(SO_4)_2 - Eu$ are excited at photon energies of 3.9–4.0 eV and 4.4–4.5 eV in the transparency region in these phosphors. The reverse excitation of phosphors with induced electron-hole trapping centers by the photons with the energies of 3.9–4.0 eV and 4.4–4.5 eV results in the appearance of recombination or tunneling emission at 2.85–3.1 eV and 2.9–3.1 eV. This proves that the formation of these emissions is associated with the accumulation of the intrinsic $SO_4^{3-} - SO_4^-$ and impurity $Eu^{2+} - SO_4^-$ electron trapping centers in the transparency region of the phosphors.

In the second stage of the study, we have found that in both phosphors with interacting $SO_4^{3-}$ and $Eu^{2+}$ electronic states, a mixed (or combined) state is formed near the bottom of the conduction band. The complementary hole state is localized near the top of the valence band near the ground state $Eu^{3+}$ (see band diagram, Fig. 8).

Along with the recombination emissions at 2.85–3.1 eV and 2.9–3.1 eV, the intracenter emissions at 2.06 eV and 2.0 eV appears as well at the irradiation of phosphors with induced defects with the 4.0 eV and 4.5 eV photons. The appearance of the $Eu^{3+}$ intracenter emissions under such excitation explains the temperature dependence of the emission intensity of $K_3Y(SO_4)_3 - Eu$ (Fig. 6).

The band diagram (Fig. 8) explains the relaxation processes of energy transfer from the phosphor matrix to the emitters, i.e., impurities. At 77 K the emitting centers are created while the electrons created during UV emission of phosphors are captured by the anions $SO_4^{2-}$ and impurities $Eu^{3+}$ following the reaction $SO_4^{2-}$ - $e^- \rightarrow SO_4^{3-}$, $Eu^{3+} + e^- \rightarrow Eu^{2+}$.

The electronic centers of $SO_4^{3-}$ and $Eu^{2+}$ below the bottom of the conduction band correspond to the combined or induced electronic state. The hole component $SO_4^-$ of the trapping center is located above the top of the valence band. The measurement of the excitation spectra of the recombination or combined emitter state at 3.1 eV and 2.95 eV showed that the excitation energies between the trapping centers are of approximately 4.0 eV and 4.5 eV. During heating, the $Eu^{2+}$ electron centers are ionized in the temperature ranges of 150–230 K and 250–350 K, and the $Eu^{3+}$ ion is reduced, the $e^-$ electron recombines with a hole near $Eu^{3+}$. The energy is transferred to the $Eu^{3+}$ impurity, and the emission from $Eu^{3+}$ at 2.06 eV and 2.0 eV is observed. In the reduction stage at 3.1 eV and 2.9 eV, its destruction occurs up to 350 K. Starting from these temperature, the $Eu^{3+}$ ion is reduced to $Eu^{2+}$ and we observe a rapid increase in its emission intensity. In the temperature range of 250–350 K, during recombination of the intrinsic electronic excitation of the $e^- + SO_4^-$ matrix, the released energy is transferred to the impurities.

## 5. Conclusion

New induced or combined emissions have been found at 2.9 eV and 3.1 eV in $K_3Y(SO_4)_3 - Eu$, and at 2.85 eV and 3.1 eV in $LiY(SO_4)_2 - Eu$ being excited at photon energies of 3.9–4.0 eV and 4.45–4.5 eV in the matrix transparency region. These induced states appear due to the combination of intrinsic and impurity electron trapping centers, which are formed as a result of the electron transfer described by the following reaction: $SO_4^{2-}$ - $e^- \rightarrow SO_4^{3-}$ and $Eu^{3+} + e^- \rightarrow Eu^{2+}$. The temperature dependence of the decay of new combined recombination emissions at 3.1 eV and 2.9 eV and the recovery of the emission states of $Eu^{3+}$ impurities in the temperature range of 150–440 K were measured, and the energy transfer of electronic excitations to the impurities is shown.

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## Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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