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O. N. Yunakova, V. K. Miloslavskii, E. N. Kovalenko, and E. V. Ksenofontova

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The absorption spectra of thin films of ternary compounds in the Rbl–Pbl₂ system

O. N. Yunakova and V. K. Miloslavskii
V.N. Karazin Kharkiv National University, pl. Svobody, 4, Kharkov 61077, Ukraine

E. N. Kovalenko
Kharkiv National University of Radio Electronics, Lenin Ave., 14, Kharkov 61166, Ukraine

E. V. Ksenofontova
V.N. Karazin Kharkiv National University, pl. Svobody, 4, Kharkov 61077, Ukraine

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Formation of compounds RbPbI₃ and Rb₂PbI₆ in the Rbl–Pbl₂ system is revealed, and their absorption spectra are investigated in an energy interval of 2–6 eV and at temperatures from 90 to 500 K. It is established that the low-frequency exciton excitations are localized in PbI₆⁻⁴ structural elements of the crystal lattice, they are classified as excitons of intermediate coupling and are of a three-dimensional character in RbPbI₃ and a quasi-two-dimensional one in Rb₂PbI₆.

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According to previous thermographic investigations, compounds RbPbI₃ and Rb₂PbI₆ are formed in the Rbl–Pbl₂ system, while ternary compounds CsPbI₃ and Cs₄PbI₆ form in the isomorphic system CsI-PbI₂. Compounds RbPbI₃ and CsPbI₃ are isostructural, and at room temperature they crystallize in the orthorhombic perovskite-like lattice with four molecules per unit cell, and similar parameters: \( a = 10.27 \text{ Å} \); \( a = 10.46 \text{ Å} \); \( b = 17.38 \text{ Å} \); \( b = 17.78 \text{ Å} \); \( c = 4.77 \text{ Å} \); \( c = 4.8 \text{ Å} \) (space group \( P_{nma} \)) in RbPbI₃ and CsPbI₃ respectively. Structural elements of the RbPbI₃ and CsPbI₃ lattices are double chains consisting of \((\text{PbI}_6)^{4-}\) octahedra and oriented along the short axis \( c \). Unlike CsPbI₃, for which the phase transition to the monochinic phase has been determined to take place in the temperature range between 563 K and 602 K (to the cubic phase with \( a = 6.29 \text{ Å} \) according to a more recent study \(^6\)), in RbPbI₃ no phase transition has been detected. \(^4\)

Early studies of the reflection spectra of single crystals RbPbI₃ and CsPbI₃ (Ref. 5) established their similarity in the measured spectral range (2.9–3.2 eV). However, measurements in Ref. 5 were conducted in a very narrow spectral range, which did not include the absorption range of the second complex compound of the Rbl-Pbl₂ and CsI-PbI₂ systems, and do not give complete information about the electronic spectrum of the compounds and the monophasic character of the crystals. While the absorption spectrum of CsPbI₃ has been studied by many authors, \(^5,9\) only one study is known regarding the spectrum of RbPbI₃, \(^5\) which, apparently, as is the case for CsPbI₃, \(^6,9\) is due to the difficulty in obtaining single-phase films of RbPbI₃ without admixture of a second compound, which is formed in the Rbl-Pbl₂ system.

The second ternary compound, Rb₂PbI₆, which forms in the \((\text{Rbl})_1(\text{Pbl}_2)_{1-x}\) system for large values of \( x \) according to Ref. 1, has not been studied.

The method developed for obtaining single-phase films of ternary compounds in the \((\text{Rbl})_1(\text{Pbl}_2)_{1-x}\) system allowed to obtain thin films of compounds and examine their absorption spectra in a wide spectral range of 2–6 eV and temperatures from 90 K to 520 K.

**Experiment**

Thin films of RbPbI₃ were prepared by vacuum evaporation of a mixture of pure Rbl and Pbl₂ powders in stoichiometric amounts onto heated quartz substrates. The mixture of powders is first melted under a screen placed between the evaporator and the substrate, and then the melt is evaporated onto the substrate. The substrate temperature was varied from 373 to 403 K. When the substrate temperature is \( T_{sub} = 393 \text{ K} \) we get monophasic RbPbI₃ films with the characteristic for the substance spectrum, similar to that of CsPbI₃, \(^5,9\) with an intense long-wavelength \( A^1\)-band at 2.975 eV (90 K) at the absorption edge. In the spectrum of thin films prepared at a lower substrate temperature \( T_{sub} \approx 353 \text{ K} \), except for the \( A^1\)-band inherent to RbPbI₃, a higher-frequency \( A^\text{II}\)-band appears, corresponding, apparently, to the second triple junction (Fig. 1, curve 1). A similar situation was observed for the compounds of the Csl-Pbl₂ system, \(^6,9\) in which at low substrate temperatures \( (T_{sub} \leq 353 \text{ K}) \) two-phase films were obtained (Fig. 1, curve 3), which after annealing for several hours at a temperature above 400 K transitioned into single-phase films with a spectrum characteristic for Cs₄PbI₁₀ (Fig. 1, curve 4). The spectra of two-phase films produced in Csl-Pbl₂ and Rbl-Pbl₂ systems are similar structurally and in the position of the main absorption bands (Fig. 1). After annealing the two-phase films \((\text{Rbl})_{0.5}(\text{Pbl}_2)_{0.5}\) for two hours at a high temperature \( T_{ann} \sim 673 \text{ K} \) their absorption spectrum changed (Fig. 1, curve 2): in terms of the structure of the spectrum and position of the main absorption bands it became similar to the spectrum of Cs₄PbI₁₀ (Fig. 1). Apparently, in the \((\text{Rbl})_1(\text{Pbl}_2)_{1-x}\) system for large values of \( x \) a compound of the same molar composition as \((\text{Csl})_1(\text{Pbl}_2)_{1-x}\) is formed. It should be noted that upon evaporation from the melt two-phase films are usually obtained that
transition into Rb$_4$PbI$_6$ at high annealing temperature, but often after this light scattering appears in the films due to the very high annealing temperature. Optically smooth indispersive Rb$_4$PbI$_6$ films are obtained by evaporation of the molten mixture of RbI and PbI$_2$ powders of stoichiometric amounts onto quartz substrates heated up to $T_{\text{sub}} = 433$ K, followed by annealing the samples for 2 h at the same temperature. The synthesis of RbPbI$_3$ films without admixture of Rb$_4$PbI$_6$ requires careful selection of the substrate temperature and the rate of evaporation of the melt and is a great challenge.

Phase composition of the films was monitored via absorption spectra measured at 90 K. Such monitoring is possible because of the significant differences in the spectral position of long-wavelength exciton bands in RbPbI$_3$ (2.975 eV), Rb$_4$PbI$_6$ (3.41 eV), PbI$_2$ (2.5 eV), and Rbl (5.7 eV).

Absorption spectra of thin films were measured using a spectrophotometer SF-46 in the spectral region between 2 and 6 eV in the temperature range of 90–500 K. For the measurement, films 80–120 nm thick were used. The spectral width of the gap in the region of the most narrow long-wavelength exciton bands is 0.02 eV, i.e., much smaller than the half-width of the exciton bands ($\Gamma \sim 0.1$ eV at $T = 90$ K).

Parameters of the long-wavelength exciton bands (the position of $E_m$, the half-width $\Gamma$, and $\varepsilon_{2m}$) were selected in such a way that on the long-wavelength side the calculated and the experimental circuits were in good agreement.

**Absorption spectra of RbPbI$_3$ and Rb$_4$PbI$_6$ thin films**

In the absorption spectrum of the RbPbI$_3$ thin film an intense long-wavelength $A_1$-band is observed at 2.975 eV (90 K) (Fig. 2(a)), shorter-wavelength bands $C_1$, $C_2$, $C_3$, and $D$ (the spectral position of the bands is given in Table 1) are located amid interband absorption. In Rb$_4$PbI$_6$, the absorption edge is shifted towards the lower wavelength region of the
spectrum by 0.44 eV with respect to RbPbI₃ (Fig. 2(b)), the long-wavelength A₁⁻ band is located at 3.41 eV (90 K), and in the low-wavelength region intensive C₁⁻, C₂⁻, and DⅡ⁻ bands are observed (see Eₚ in Table 1).

With increasing temperature, the A-, C-, and D-bands in the absorption spectra of both compounds are shifted to longer wavelengths, broadened, and weakened due to the exciton-phonon interaction (EPI), which indicates their relationship with the exciton excitations.

After the separation of the A₁, A₁⁻ and A₁⁻ bands by a symmetric contour on the long-wavelength side of C₁⁻ and C₁⁻ in RbPbI₃ and PbPbI₆, weak A₂⁻ and A₂⁻ bands are observed, which we attribute to the excitation of 2×excitons. We estimate the exciton binding energy based on their spectral position in the Wannier-Mott approximation of excitons we estimate the exciton binding energy Rₚ = 4/3(Eₑₑ₂ – Eₑ₁) = 0.153 eV, Rₑₑ = 0.133 eV and the width of the band gap Eₑₑ = Eₑ₁ + Rₑₑ, equal to 3.14 eV in RbPbI₃ and 3.54 eV in PbPbI₆.

For the interpretation of absorption spectra of RbPbI₃ and PbPbI₆ thin films we compare them with the spectra of the previously studied compounds CsPbI₃ and CsₓPbI₆ (Fig. 2). As seen in Fig. 2, the absorption spectra of CsPbI₃ and PbPbI₆, CsₓPbI₆ and PbₓPbI₆ are similar in structure and with respect to the position of absorption bands (see Table 1), indicating the localization of excitons in the cation sublattice of the compounds, which contains lead ions. Also in favor of this localization, we observe a linear dependence of the spectral position of the long-wavelength exciton bands on the molar concentration x of Pbl₂ in a line of compounds PbI₂, PbPbI₆, PbPbI₆, PbI₂, CsPbI₃, CsₓPbI₆, converging for x → 0 to E = 3.6 eV, which is significantly less than the spectral position of admixture bands of Pb⁵⁺ in RbP²⁺ (Fig. 2, inset). According to the concept developed for multicomponent compounds and solid solutions, in the case of localization of excitons in the sublattice of one of the components, with its decrease the low-frequency bands converge to the spectral position of the impurity bands of the metal in the lattice of the second component.

With the localization of excitons in PbI₂ sublattice of compounds PbPbI₃ and PbPbI₆, the top of the valence band in them, as in PbI₂, is formed by Pb 6s-states mixed with some I 5p-states, and the conduction band—by Pb 6p-states, and excitons, as in PbI₂, are cationic in nature. Therefore, absorption spectra of PbPbI₃ and PbPbI₆, like the spectra of CsPbI₃, CsₓPbI₆, and PbI₂, should be treated on the basis of electronic transitions in the (PbI₆)₄⁻ octahedra, the structural elements of the crystal lattices of the compounds similarly to the spectra of Pb⁵⁺ impurity ions in alkali-halide crystals. For the Pb²⁺ impurity ions in a lattice of D₃d symmetry the following transitions are characteristic: A → A₁g band → 3T₁g, (S₀ → 3P₁ in the free Pb⁵⁺ ion), B → A₁g band → 3T₂g or 3E_u (S₀ → 3P₂), C → A₁g band → 3T₁u, (S₀ → 1P₁). The sharp long-wavelength edge and the high intensity of A- and C-bands in the RbPbI₃ and PbPbI₆ spectra indicate their relation to direct allowed transitions in the (PbI₆)₄⁻ structural elements of the lattices of the compounds and, as in RbI: Pb²⁺, they apparently correspond to the transitions A₁g → 3T₁u (the A-band) and A₁g → 1T₁u (the C-bands). The B-band corresponding to the forbidden transition in Pb²⁺ impurity centers does not appear in our spectra.

Temperature dependence of the long-wavelength parameters of exciton absorption bands in RbPbI₃ and PbPbI₆

The absorption spectra of RbPbI₃ and PbPbI₆ thin films in the long-wavelength exciton band (2.4–3.7 eV) were measured in the temperature range from 90 to 500 K.

With increasing temperature the A₁⁻ band in RbPbI₃ shifts linearly to longer wavelengths with dEₑₑ/dT.

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<td>0.133</td>
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FIG. 3. Temperature dependence of the spectral position Eₑₑ(T) (a) and half-width T(T) (b) of the long-wavelength exciton band A₁ in RbPbI₃ (I = 80 nm) and A₁⁻ in RbPbI₆ (2) (I = 120 nm): dots represent experimental data, solid lines (b) were calculated from Eq. (1) for d = 3 (1) and d = 2 (2).
$dT = -1.73 \times 10^{-4} \text{eV/K}$ (Fig. 3(a)), close to the value of $dE_{\text{md}}dT = -1.6 \times 10^{-4} \text{eV/K}$ in PbI$_2$ thin films. With respect to the order of magnitude, such a shift is typical for most ionic crystals, including the compounds studied here. In ionic crystals the interaction of excitons with longitudinal optical (LO) phonons and the greatest temperature-dependent changes of the exciton band parameters occur at $\hbar\omega_{\text{LO}} \leq kT$. Unknown values of $\hbar\omega_{\text{LO}}$ in RbPbI$_3$ and Rb$_2$PbI$_6$ can be estimated from the known values of $\hbar\omega_{\text{LO}} = 13.7 \text{meV}$ in PbI$_2$, and $\hbar\omega_{\text{LO}} = 13.2 \text{meV}$ in RbI. Given the molar composition of the compounds, in RbPbI$_3$ $\hbar\omega_{\text{LO}} \sim 13.45 \text{meV}$, and in Rb$_2$PbI$_6$ $\hbar\omega_{\text{LO}} \sim 13.5 \text{meV}$.

Temperature dependence of the half-width of the $A_1^-$ band in RbPbI$_3$ $\Gamma(T)$ is shown in Fig. 3(b). The dependence $\Gamma(T)$ for excitons of different dimension $d$ ($d = 1, 2, 3$) according to theory is defined as

$$\Gamma(T) \approx \left[ \frac{\pi D^2}{\gamma(d/2)(2\pi B)^{d/2}} \right]^{1/2},$$

where $\gamma(d/2)$ is the gamma function, $B$ is the exciton band width, $D^2 = 0.5 C^2 h \omega_{\text{LO}} \text{cth} (h \omega_{\text{LO}}/kT)$, and $C^2/2$ is the energy of lattice relaxation in the exciton. The contribution of the residual broadening $\Gamma(0)$ to the total half-width of the exciton band $\Gamma$ due to the lattice defects should also be taken into account. In RbPbI$_3$ the exciton band $A_1^-$ has a Gaussian shape in the entire investigated temperature range. In this case, the total half-width of $\Gamma$ is defined as

$$\Gamma = [\Gamma^2(0) + \Gamma^2(T)]^{1/2},$$

where $\Gamma(T)$ obeys Eq. (1) with an unknown factor $A$, independent of $T$.

Treatment of the experimental dependence $\Gamma(T)$ using Eq. (1) for different values of $d$ gives the best agreement of the calculation with experiment for $d = 3$. In this case

$$\Gamma(T) = A \text{cth}^2 (\hbar\omega_{\text{LO}}/kT)$$

and the dependence $\Gamma(T)$ in the coordinates $\Gamma^2$ from $\text{cth}^4 (\hbar\omega_{\text{LO}}/kT)$ is linear. Treatment of this relationship by least squares gives the values $\Gamma(0) = (0.126 \pm 0.004) \text{eV}$ and $A = (0.78 \pm 0.006) \times 10^{-2} \text{eV}$. Calculated using Eqs. (2) and (3), temperature dependence $\Gamma(T)$ with the found values of $\Gamma(0)$ and $A$ is in good agreement with the experimental one (Fig. 3(b)).

In Rb$_2$PbI$_6$ the temperature dependence of the spectral position $E_m(T)$ in the range of 90 K $\leq T \leq 440$ K is linear (Fig. 3(a)): $E_m(T) = E(0) - aT$, where $E(0) = (3.428 \pm 0.02) \text{eV}$, $a = dE_{\text{md}}dT = (2.27 \pm 0.06) \times 10^{-4} \text{eV/K}$, and this is due to EPI. The temperature shift coefficient $dE_{\text{md}}dT$ is characteristic for ionic crystals in terms of order of magnitude. At $T > 430$ K, a kink in the dependence $E_m(T)$ is observed with a stronger shift of the spectral position of the exciton band towards lower frequencies. Since in the temperature dependence of the half-width of $\Gamma(T)$ (Fig. 3(b)) near 430 K no features are observed, we do not link changes in $E_m(T)$ to a phase transition. At $T > 430$ K in Rb$_2$PbI$_6$ films there is slight light scattering, pointing to their destruction. In particular, at high temperatures the film loses its continuity and transforms into an insulator. This effect was observed, for example, in CuBr films. During high-temperature annealing of continuous films ($T_{\text{ann}} \sim 423$ K) they turned into insulae, and their absorption spectrum changed significantly: spectrum inversion occurred in the region of exciton absorption, and near the exciton absorption bands rather intense transmittance bands appeared. In the initial stages such a process can lead to a significant change in the spectral position of the exciton bands. Perhaps the deviation of the spectral position of the long-wavelength exciton band in Rb$_2$PbI$_6$ at $T > 430$ K from a linear dependence is due to the above.

The shape of the exciton band $A_1^-$ is a Gaussian in the entire range of temperatures under investigation, and its total half-width $\Gamma$, with the contribution of residual broadening $\Gamma(0)$, is determined by Eq. (2). Treatment of the temperature dependence of the half-width $\Gamma$ with Eq. (1) gives the best agreement with experiment for $d = 2$ (Fig. 3(b)). In this case, $\Gamma(T) = A \text{cth} (\hbar\omega_{\text{LO}}/kT)$ and in the coordinates $\Gamma^2$ from $\text{cth}^4 (\hbar\omega_{\text{LO}}/kT)$ the dependence is linear. Its treatment by the method of least squares gives the values $\Gamma(0) = (0.114 \pm 0.001) \text{eV}$ and $A = (0.34 \pm 0.003) \times 10^{-2} \text{eV}$.

Thus, it follows from the analysis of the temperature dependence of $\Gamma(T)$ that the excitons in RbPbI$_3$ have three-dimensional character, and in Rb$_2$PbI$_6$—two-dimensional. Perhaps the crystal structure of Rb$_2$PbI$_6$ is layered, which results in two-dimensional character of excitons, but to reach the final conclusion it is necessary to study the crystal structure.

The dispersion of the refractive index $n(\lambda)$ in RbPbI$_3$ and Rb$_2$PbI$_6$ thin films in the transparency region was determined by the interference method. The obtained dependences $n(\lambda)$ (Fig. 4) are well described by the Vempala single oscillator model

$$\varepsilon_1 = n^2 = 1 + \frac{E_2E_0}{E_0^2 - E_2^2},$$

where $E = \hbar\omega$, $E_0$ and $E_2$—are the single oscillator model parameters. $E_0$ determines the spectral position of the effective

**FIG. 4.** Spectral dependence of the refractive index $n(\lambda)$ of RbPbI$_3$ (1) and Rb$_2$PbI$_6$ (2) thin films; dots represent experimental data, solid curves were calculated from Eq. (4).
oscillator associated with the interband optical transitions, the value of $E_0 > E_{ex}$, and $E_d$ is the dispersion energy, which characterizes the strength of interband transitions.

In coordinates $(n^3-1)^{-1} = f(E)$ the relationship (4) is linear. Treatment of the experimental data $n(\lambda)$ in these coordinates by least squares using the slope of the line allowed us to determine the value of $(E_d/E_{ex})^{-1} = 0.012 \pm 0.0002$ in RbPbI$_3$ thin films, $(E_d/E_{ex})^{-1} = 0.007 \pm 0.0002$ in Rb$_2$PbI$_6$ thin films, and the intersection with the ordinate axis value $E_d/E_{ex} = 0.262 \pm 0.0015$ and, therefore, $E_d = 9.76$ eV, $E_{ex} = 17.83$ eV in RbPbI$_3$ thin films, and $E_{ex} = 7.22$ eV, $E_d = 19.72$ eV in Rb$_2$PbI$_6$ thin films. The calculated dependence of $n(\lambda)$ according to Eq. (4) (Fig. 4, solid line) with such values of $E_0$ and $E_d$ is in good agreement with the experimental dependence $n(\lambda)$ (Fig. 4, point).

Approximating the dependence $n(\lambda)$ to the low energy limit according to Eq. (4) gives the value of the optical dielectric constant $\varepsilon_\infty = (1 + E_d/E_0) = 4.82$ in RbPbI$_3$ and $\varepsilon_\infty = 3.73$ in Rb$_2$PbI$_6$. The obtained values of $\varepsilon_\infty$ were used to estimate the exciton radius in both compounds

$$a_{ex} = a_B \frac{R}{R_{ex} \varepsilon_{eff}}, \quad (5)$$

where $a_B = 0.529 \times 10^{-8}$ cm is the Bohr radius, $R = 13.6$ eV is the Rydberg energy, $\varepsilon_{eff}$ is the effective dielectric permittivity, $\varepsilon_\infty < \varepsilon_{eff} < \varepsilon_0$. $\varepsilon_0$ is static dielectric permittivity, $R_{ex} = 0.153$ eV and $R_{ex} = 0.133$ eV are the defined above values of exciton binding energy in RbPbI$_3$ and Rb$_2$PbI$_6$, respectively. To estimate the radius of the exciton we used the lower limit of $\varepsilon_{eff}$, because in the low-frequency exciton band the main contribution to $\varepsilon_{eff}$ is determined by the value of $\varepsilon_\infty$. The obtained values of the exciton radius $a_{ex} = 9.76$ Å in RbPbI$_3$ and $a_{ex} = 14.49$ Å in Rb$_2$PbI$_6$ allow us to attribute the exciton states in both compounds to intermediate coupling excitons.

Conclusion

Using the absorption spectra the formation of RbPbI$_3$ and Rb$_2$PbI$_6$ in the Rbl-PbI$_2$ system has been determined. It is shown that in these compounds the long-wavelength exciton states are localized in the sublattices containing Pb$^{2+}$ ions, surrounded by I$^-$ ions, and refer to intermediate coupling excitons.

From the analysis of the temperature dependence of the exciton band parameters, the spectral position $E_m(T)$, and half-width $\Gamma(T)$ we conclude that there is no phase transition in RbPbI$_3$ in the studied temperature range.

The temperature dependence of $\Gamma(T)$ indicates a three-dimensional character of excitons in RbPbI$_3$ and a two-dimensional character in Rb$_2$PbI$_6$.

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Translated by D. K. Maraoulaite