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Spectral and kinetic characteristics of luminescent converters on the bases of cadmium iodide

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The results of a comprehensive investigation of spectral and kinetic characteristics of CdI_2, CdI_2:$CdCl_2$, CdI_2:PbI_2, CdI_2:$SnCl_2$, CdI_2:$MnCl_2$, CdI_2:$PbCl_2$,$MnCl_2$, and CdI_2:$SnCl_2$, $MnCl_2$ crystals grown by the Bridgeman-Stockbarger technique are presented. The chlorine admixture has been found to enhance the scintillator radiation resistance while influence only slightly the emission spectra and kinetic parameters of x-ray luminescence. Cadmium iodide doped with manganese exhibits the highest x-ray luminescence yield peaked in the red spectral area. The prepared CdI_2:PbI_2,$MnCl_2$, and CdI_2:$SnCl_2$,$MnCl_2$ systems are characterized by high-efficient luminescence of Mn^{2+} centers within a wide temperature range. Photoluminescence of those centers is observed both under intracenter excitations associated with the *d-d* transitions and in the sensitized process as a result of the energy migration from the crystal matrix and the extrinsic tin and lead centers. Basing on the results obtained and literature data, the nature of the luminescence centers is discussed as well as the possibilities of practical use of the studied crystal systems.

Приведены результаты комплексного исследования спектрально-кинетических характеристик кристаллов CdI_2, CdI_2:$CdCl_2$, CdI_2:PbI_2, CdI_2:$SnCl_2$, CdI_2:$MnCl_2$, CdI_2:PbI_2, $MnCl_2$ и CdI_2:$SnCl_2$,$MnCl_2$, выращенных методом Бриджмена-Стокбаргера. Установлено, что примесь хлора повышает радиационную стойкость сцинтилляторов, при этом слабо влияет на спектры излучения и кинетические параметры рентгенолюминесценции. Йодистый кадмий, с примесью марганца при комнатной температуре имеет наибольший выход рентгенолюминесценции с максимумом в красной области спектра. Полученные системы CdI_2:PbI_2,$MnCl_2$ и CdI_2:$SnCl_2$,$MnCl_2$ характеризируются эффективной люминесценцией Mn^{2+}-центров в широком температурном интервале. Фотолюминесценция этих центров наблюдается как при внутрицентровых возбуждениях, связанных с *d-d* переходами, так и в сенсибилизированном процессе в результате миграции энергии от основы кристаллов и примесных центров олова и свинца. На основе анализа полученных результатов и литературных данных обсуждается природа центров люминесценции и возможности практического использования исследованных кристаллических систем.

The layered ion-covalent cadmium iodide crystals are considered as perspective materials for optoelectronics and holographic data recording [1, 2]. Before, it has been shown [3–5] that it is possible to obtain short-decay scintillators based on CdI_2 and the advantage thereof has been established as compared to plastic scintillators in registration of short-time photon beams with quantum energy exceeding 200 keV. It has been shown [6] that the combined detectors (CD) comprising the layered CdI_2:Mn scintillators and the industrial silicon photodiodes exceed by 2 to 3 times the x-ray sensitivity of similar detectors based on CsI:Tl scintillators and are of a practical interest in ionizing radiation recording under stationary and low-frequency excitation regimes. In this work, the spectral and kinetic characteristics of cadmium iodide crystals (of special purity grade as well as single- and polyactivated) are studied comprehensively to obtain information on the effects of synthesis conditions, temperature, and co-activators on the luminescence parameters and to estimate the possibilities of the practical use of the studied systems.

The crystals to be studied were grown using the Bridgeman-Stockbarger technique from the raw material pre-purified by zone melting [1]. The admixtures were introduced into the melted raw materials as $CdCl_2$ (0.5 mol.%), PbI_2 (0.1 mol.%), $SnCl_2$ (0.1 mol.%), $MnCl_2$ (0.5 mol.%). The luminescence spectra were measured in a vacuum cryostat using the monochromator of a SF-4A spectrophotometer on the samples prepared by cleaving along the cleavage planes and shaped as about 15×15×1.5 mm^3 plates. The samples were optically excited using a LGI-21 N_2 laser. When studying the x-ray luminescence (XRL) and thermostimulated luminescence (TSL) spectra, the crystals were excited using an URS-55A x-ray unit (BSV2-Cu tube, $U = 45$ kV, $I = 12$ mA). The crystals were irradiated with X-quanta through an about 0.5 mm thick beryllium window. The crystal temperature was measured using a copper-constantan thermocouple. The emission spectra and TSL curves were measured using a FEP-51 sensors, the sensor signals were amplified by DC amplifiers and applied to two-coordinate PDA1 plotters, thus providing the simultaneous recording of the emission intensity and the wavelengths or the emission intensity and temperature. When measuring the optical excitation and photoluminiscence PL spectra, a DksEl-1000 xenon lamp was used as the excitation source. The necessary exciting spectral band was provided by a ZMR-3 monochromator. When measuring the excitation spectra, the constant intensity of the exciting light quanta at different frequencies was provided. The luminescence kinetic parameters were studied under pulse x-ray excitation (the pulse half-width $\Delta t = 0.5$ ns) using the statistical single-quantum counting, similar to the procedure described in [7]. For high-energy excitation, an URS-55 x-ray unit and a home-made pulse x-ray source comprising a RTI-0.05 tube were used. The setup provided the study of emission processes within the time range of 10^{-10} to 10^{-4} s. When estimating the luminescence decay time of the crystal, the kinetic curves obtained at 295 K were re-plotted in $\lg I - t$ coordinates, the exciting X-pulse duration being not taken into account.

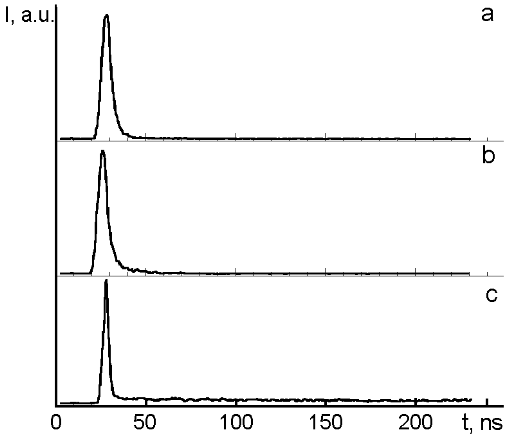

Fig. 1. XRL kinetic curves of CdI_2 (a), CdI_2:$CdCl_2$ (b) and BaF_2 (c) crystals at 295 K.

The XRL spectra of non-activated CdI_2 crystals at 85 K contain an intense non-elementary 540–560 nm band. At 295 K, those are characterized by a weak luminescence peaked in the 490 to 510 nm range. At room temperature, the light yield of the CdI_2 scintillator is 0.5 to 0.8 of that of polystyrene plastic scintillators [3, 4]. The XRL kinetics of CdI_2 is characterized by a very short (less than 0.3 ns) pulse rise and a rapid glow dropping described by an exponential dependence with $\tau_1 \approx 2.8$ ns (Fig. 1,a). In the samples obtained using the lower purity salt, a slow component $\tau_2 \approx 10^{-7}$ s and a long-duration component $\tau_3 \approx 10^{-4}$ s are also observed, the contribution of both to the decay kinetics amounts 1 to 10 %. Independent of the growing method, the crystals are characterized by almost the same kinetics of the XRL flaring-up.

Cadmium iodide activation with the homologous Cl^- admixture does not influence essentially the light yield and spectral parameters of CdI_2 scintillator both at low and high temperatures. At room temperature, the scintillation pulse of the material contains a weak component with the decay time $\tau_2 \approx 15$ ns is observed along the main one with $\tau_1 \approx 3.8$ ns (Fig. 1,b). For comparison, (Fig. 1,c) presents the XRL kinetic curve of a BaF_2 crystal. The integral glow decay kinetic of that scintillator is characterized by fast ($\tau_1 \approx 1.8$ ns) and slow ($\tau_2 \approx 300$ ns) stages. As to the fast component, the en-

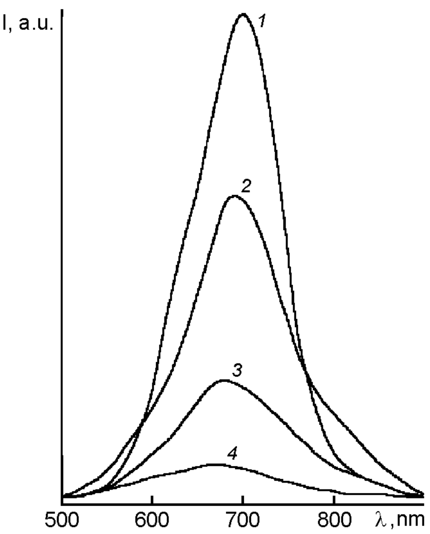

Fig. 2. XRL spectra of CdI_2:$SnCl_2$ crystal at temperatures (K): 85 (1), 185 (2), 235 (3), and 295 (4).

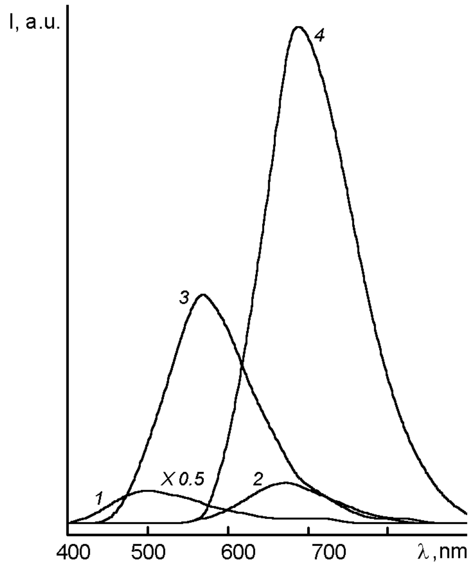

Fig. 3. XRL spectra of CdI_2 (1), CdI_2:$SnCl_2$ (2), CdI_2:PbI_2 (3), and CdI_2:$MnCl_2$ (4) crystals at 295 K.

ergy yield for CdI_2 is somewhat higher than that of BaF_2 [8].

Spectroscopic studies of CdI_2 crystals activated from the melt with Sn, $SnCl_2$, and SnI_2 admixtures have shown that the presence of tin ions in the CdI_2 lattice results in an extrinsic absorption in the near-edge area, activator bands in the excitation spectra of photoluminescence, photoconductivity, and the spectral sensitivity of the electret state [9]. Under excitation of those crystals with light from the intrinsic and extrinsic absorption areas as well as with x-ray quanta at 85 K, an emission peaked near 690 nm is observed. The crystal temperature rise up to 295 K results in a weakened luminescence intensity and the spectral maximum shifting to 680 nm. The temperature effect on the XRL spectra of CdI_2:$SnCl_2$ is shown in Fig. 2. At room temperature, the XRL pulse of the system at the decay stage is characterized mainly by two exponential processes with $\tau_1 \approx 20$ ns and $\tau_2 \approx 540$ ns. Along with the fast kinetic components of the flaring-up and decay, the emission of CdI_2 with Sn^{2+} admixture contains also a weak millisecond range component. It has been found that it is just CdI_2 crystals activated from the melt with low (about 0.03 mol.%) concentrations of Cd metal that show similar spectral and kinetic parameters when being excited with x-rays.

At 85 K, the CdI_2:Pb emission spectrum contains the bands peaked at 530–540 and 650–660 nm. In the XRL spectrum of that crystal luminophor at 295 K, a non-elementary band at 570 nm is observed [5]. The luminescence flaring-up kinetics in that band is similar to that in CdI_2, but the decay proceeds according to a more complex law, like to the case of CdI_2:Sn^{2+}. The XRL pulse shape for CdI_2:Pb at the initial and far decay stages is mainly exponential with constant decay times $\tau_1 \approx 10$ ns and $\tau_2 \approx 250$ ns.

The comparative study results of the luminescence intensity and spectral composition for pure and activated CdI_2 crystals at room temperature under the same experimental conditions are presented in Fig. 3. Consideration of these data shows that CdI_2 is characterized by a relative low XRL yield and that the emission has the form of a broad asymmetric band peaked in the 490–500 nm area (curve 1). CdI_2 activation with tin admixture results in an enhanced (by several times) light yield and the spectral maximum shifted to 680 nm (curve 2). For CdI_2:PbI_2, typical is an approximately one order of magnitude higher XRL yield in the band maximum as compared to CdI_2 (cf. curves 1 and 3). Manganese activated CdI_2 shows the highest XRL yield with a maximum located near 700 nm (curve 4).

CdI_2:Mn^{2+} crystals under x-ray excitation are characterized by an efficient activator luminescence within a wide temperature range. At 85 K, the CdI_2:$MnCl_2$ XRL is presented by a weak matrix emission in the 400 to 600 nm range and an intense emission of Mn^{2+} centers near 735 nm (Fig. 4,

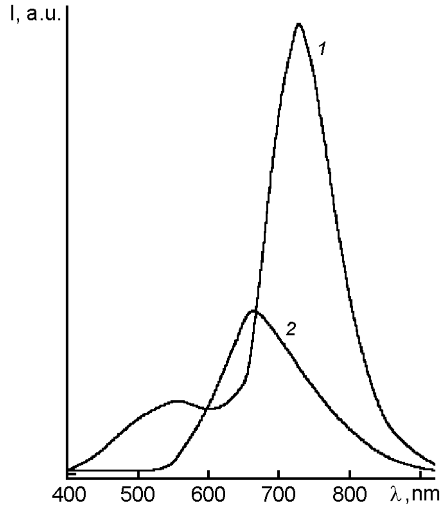

Fig. 4. XRL spectra of CdI_2:$MnCl_2$ crystal at temperatures (K): 85 (*1*) and 450 (*2*).

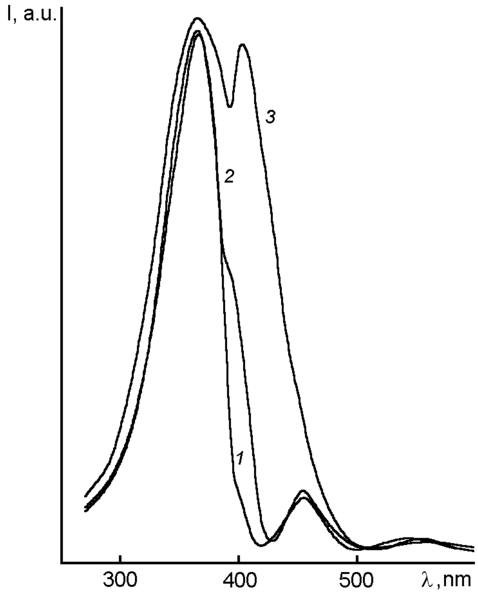

Fig. 5. Excitation spectra of Mn^{2+} center luminescence in CdI_2:$MnCl_2$ (*1*), CdI_2:PbI_2,$MnCl_2$ (*2*), and CdI_2:$SnCl_2$,$MnCl_2$ (*3*) crystals at 295 K.

curve 1). As the temperature rises up to 450 K, the emission yield drops more than by half, the activator band maximum being shifted to 660 nm (Fig. 4, curve 2). In the scintillation pulse of integral CdI_2:Mn emission at 295 K, the fast component ($\tau_1 \approx 3.4$ ns) of the matrix emission is observed and an inertial component ($\tau_2 \approx 450$ µs) associated with intracenter transitions in Mn^{2+} [4, 5]. The additional doping of CdI_2:$MnCl_2$ with PbI_2 has been found to do not influence essentially the XRL yield and spectral composition at 295 K. As the temperature is lowered down to 85 K, the manganese emission intensity is increased twice. In the XRL spectrum of the polyactivated crystal, along with the intense activator band at 735 nm, weak 540–550 and 640–660 nm bands are observed typical of CdI_2:PbI_2 [5]. The presence of tin admixture in CdI_2:$SnCl_2$,$MnCl_2$ influences slightly also the luminescence intensity and spectral composition of Mn^{2+} centers under x-ray excitation.

The excitation spectra of manganese PL in single- and polyactivated CdI_2 crystals are shown in Fig. 5. It is seen (curves 1 and 2) that in the excitation spectrum of Mn^{2+} center luminescence in cadmium iodide activated simultaneously with manganese and lead ions, there is a shoulder in the 390–415 nm area besides of the broad intense band in the intrinsic absorption range (315–390 nm) and weak bands in the 420–600 nm region typical of CdI_2:$MnCl_2$ excitation spectrum. The shoulder is due to Pb^{2+} center absorption [1]. The excitation spectrum of manganese PL in CdI_2:$SnCl_2$,$MnCl_2$ at room temperature contains also an additional intense band in the 385–460 nm range that is associated with Sn^{2+} ions [1, 9] (cf. curves 1 and 3, Fig. 5). The intense excitation of Mn^{2+} ions in the intrinsic absorption region and in the co-activator absorption bands evidences a sensitized mechanism of the emission excitation of those centers. The energy transfer from the matrix and mercury-like ions to manganese has been also revealed in polyactivated crystals at 85 K.

When excited with N_2 laser in the intrinsic absorption region, the CdI_2-based crystal luminophors are characterized by an efficient luminescence similar to XRL in spectral composition. In the course of optical and x-ray excitation at room temperature, both pure and activated crystals are not colored and do not store the light sum. In the TSL curves of single- and polyactivated CdI_2 crystals with chlorine admixture after x-ray excitation at 85 K, the high-temperature (>200 K) peaks are almost unobservable. It follows from the results obtained that the luminescence of the pure crystals is due mainly to the single emission center type with the decay time $\tau_1 < 5$ ns. In the activated crystals, an additional emission with a longer component ($\tau_2 > 10$ ns) is observed. The forward front of the scintillation pulse that is defined by the energy transfer processes to the emission centers depends slightly on the crystal structure perfection and the presence of Sn^{2+} and Pb^{2+} admixtures. The kinetic parameters of the CdI_2 and CdI_2:Pb^{2+} crystals are in agreement with the time parameters of those systems obtained under excitation with α particles [3] and electron beam [4].

The nanosecond scintillations in BaF_2 are due to the Rodny mechanism [10–12], i.e., to transitions between the anionic and cationic valence bands of the crystal. The energy yield of the core-valence luminescence (CVL) does not exceed 1 or 2 %. The CVL is characterized by very short-timed emissive relaxation being within limits of 0.8 to 3 ns. Unlike the usual activator or exciton luminescence, the CVL yield remains essentially constant within a wide temperature range. Its time parameters are also highly temperature-independent. The emission is not excited in the regions of the anionic exciton absorption and the fundamental absorption onset. In TSL and in the IR illumination stimulated luminescence, the CVL is not revealed [10, 13]. The short decay times and small amplitude of the scintillations allow to ascribe the "intrinsic" luminescence of CdI_2-based crystals with the decay time of 2.5 to 4 ns to the CVL. That luminescence, however, is excited effectively in the region of the long-wavelength intrinsic absorption edge and is quenched heavily as the temperature rises from 130 up to 295 K [1, 5]. These data evidence the absence of CVL in CdI_2. It has been supposed [3] that the short-decay emission of CdI_2-based crystals is defined by the non-equilibrium state of V_k centers that are formed and exist during the crystal irradiation.

The scintillation excitation mechanism in CdI_2 at 295 K may be due to migration processes involving the non-relaxed holes and excitons [14, 15]. The fast component of the emission pulse predominating in the perfect non-activated CdI_2 crystals at room temperature is likely associated with the V_F type centers [5]. The short-wavelength luminescence of CdI_2-based crystals at 295 K can be considered as the radiative decomposition of (V_k-e) excitons formed during the recombination and localized near the cationic vacancies, while the short decay time may result from the luminescence temperature quenching. This conclusion is confirmed by the fact that the emission peaked in the 490–510 nm region is quenched in CdI_2:Cd crystals while effective emission at 680–690 nm is observed [5]. Such changes in the luminescence spectra are caused by the fact that when cadmium iodide is activated with Cd from the melt, it is enriched in the metal component due both to appearance of the excess interstitial Cd in the lattice and to increased number of iodine vacancies, the concentration of cationic vacancies being decreased, too. The defects generated due to the crystal composition deviations from the stoichiometry define also the main spectral properties of ZnS [16] and BiI_3 [17]. The low-temperature emission of CdI_2, CdI_2:$CdCl_2$, and CdI_2:$CdBr_2$ in the 540–550 nm band is associated with autolocalized excitons [5, 18, 19].

The isovalent cationic impurities in low concentrations are arranged in the layered cadmium iodide crystals in two fashions. A fraction of Pb activator enters the CdI_2 lattice by substituting the cations thereof, while another one is distributed near the lattice defect sites forming cluster centers with the intrinsic point defects [20]. The emission peaked at 560–580 nm observed in CdI_2:Pb^{2+} luminophor at high temperatures may be due to the emission of near-activator anionic excitons characterized by a fast deactivation of the excited state. The slow component of the system luminescence is due likely to the charge carrier recombination at the emission centers that are associates of the intrinsic and extrinsic defects [5, 20]. The low-temperature luminescence of CdI_2:Pb^{2+} peaked at 640–660 nm is associated with the emissive annihilation of anionic excitons localized at the iodine vacancies associated with Pb^{2+} [5]. Taking into account the volume compensation factor [20], the formation of those centers is energy-favorable, since Pb^{2+} has larger ionic radius than Cd^{2+} [21].

Tin impurity does not form activator luminescence centers in CdI_2 but it reinforces the self-activator emission typical of low-doped CdI_2:Cd crystals [5, 9]. It is supposed [5] that the CdI_2:Cd luminescence peaked at 680–690 nm is associated with the crystal stoichiometry deviation towards enrichment in cadmium and appears as a result of the charge carrier recombination at halogen vacancies associated with the interstitial cadmium atoms. This is confirmed by the fact that the 680–690 nm emission is reduced at the subsequent heating of CdI_2:Cd crystals in iodine vapor.

Basing on the data obtained, taking into account the studied spectral characteristics of CdI_2:MnI_2 and CdI_2:PbI_2,MnI_2 and the earlier study results [3, 4, 9], the chlorine admixture ($C < 0.5$ mol.%) has been established to do not effect essentially on the XRL and PL spectral composition of the studied cadmium iodide crystals activated with cationic impurities. The emission of manganese-activated CdI_2 in the red spectral region with the decay time of about 450 μs is due to electron transitions ${}^4T_{1g}({}^4G) \rightarrow {}^6A_{1g}({}^6S)$ in Mn^{2+} ions [1, 5]. It follows from the data obtained and spectral characteristics of $CdCl_2$:Mn and $CdBr_2$:Mn [1, 22, 23] that the specific features of those centers are defined by the local environment thereof and that the spectral position of the manganese emission bands is defined by the matrix anion. The existence of centers associated with substitutions in the anionic CdI_2 sublattice has been proved by optical methods [18] and the Raman scattering data [24]. Since chlorine impurity does not influence appreciably the spectral composition of $CdI_2:Mn^{2+}$ XRL and PL, it can be assumed that it is energy-favorable for the activator ions to be in iodine environment that has a higher polarizing ability as compared to chlorine ions [20].

The photoluminescence of Mn^{2+} centers in polyactivated crystals is observed both at intracenter excitations associated with *d-d* transitions [1] and in the sensitized process as a result of energy migration from the matrix and centers associated with tin and lead impurities. At high temperatures, the energy migration to manganese ions occurs mainly by the electron-hole mechanism [14] as a result of the electron excitation delocalization from Pb^{2+} and Sn^{2+} ions [1, 25]. At low temperatures, in addition, emissive and non-radiative energy transfer occurs from relaxed and localized excitons to Mn^{2+} centers [15]. Perhaps that is why the manganese PL in the crystals studied at 85 K is approximately twice as intense as that at 295 K.

It follows from the study results and literature data that the prepared CdI_2:$CdCl_2$ scintillator is suitable for use in thin-layer detectors of ionizing radiation [26] with a high time resolution. The CdI_2:PbI_2,$MnCl_2$, and CdI_2:$SnCl_2$,$MnCl_2$ crystals emitting in the manganese band and storing weakly the light sum at deep capture levels can be used as photoluminophors with enhanced light sensitivity in the 390–460 nm region. Those can be suitable also for thin-layer detectors [26] and the scintillator/silicon photodiode type detectors to record x-rays in stationary and low-frequency excitation regimes. The CdI_2:Pb crystals with a larger signal amplitude as compared to CdI_2 and short decay time can be used together with CdI_2:Mn ones to provide combined detectors of ionizing radiation [27].

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Спектрально-кінетичні характеристики люмінесцентних перетворювачів отриманих на основі йодистого кадмію

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Приведено результати комплексного дослідження спектрально-кінетичних характеристик кристалів CdI_2, $CdI_2:CdCl_2$, $CdI_2:PbI_2$, $CdI_2:SnCl_2$, $CdI_2:MnCl_2$, $CdI_2:PbI_2,MnCl_2$ і $CdI_2:SnCl_2,MnCl_2$, вирощених методом Бріджмена-Стокбаргера. Встановлено, що домішка хлору підвищує радіаційну стійкість сцинтиляторів, при цьому слабо впливає на спектри випромінювання і кінетичні параметри рентгенолюмінесценції. Йодистий кадмій, з домішкою марганцю при кімнатній температурі має найбільший вихід рентгенолюмінесценції з максимумом у червоній області спектра. Отримані системи $CdI_2:PbI_2,MnCl_2$ і $CdI_2:SnCl_2,MnCl_2$ характеризуються ефективною люмінесценцією Mn^{2+}-центрів в широкому температурному інтервалі. Фотолюмінесценція цих центрів спостерігається як при внутрішньоцентрових збудженнях, пов'язаних з *d-d* переходами, так і у сенсибілізованому процесі в результаті міграції енергії від основи кристалів та домішкових центрів олова та свинцю. На основі аналізу отриманих результатів і літературних даних обговорюється природа центрів люмінесценції і можливості практичного використання досліджених кристалічних систем.