Photon yields and decay times of cross luminescence in ionic crystals*

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Abstract

We have studied the scintillation properties of KMgF_3 , $\text{KYF}_4\text{:Rb}$, K_2YF_5 , KLuF_4 , RbMgF_3 , KZnF_3 , BaTm_2F_8 , $\text{LiYF}_4\text{:Nd}$ and $\text{BaF}_2\text{:Rb}$. The first four crystals produce cross-luminescence (CL) with a decay time of about 1.5 ns. CL was not observed for the other crystals.

behaviour. CL was already observed recently for KMgF_3 , KYF_4 , K_2YF_5 , and KLuF_4 crystals [3, 11, 12]. However, little is known about the absolute CL light output and its decay time on which we report in this work. The other crystals did not show evidence for CL. The Nd^{3+} doped LiYF_4 crystal is of interest because of a 5d-4f emission from Nd^{3+} at 185 nm.

Introduction

The discovery of a fast intrinsic luminescence with a decay time of 0.8 ns in BaF_2 crystals [1] started a renewed interest in scintillation materials. This so-called cross-luminescence (CL) is caused by transitions of electrons from the F^- 2p-valence band to holes, created by ionizing radiation, in the Ba^{2+} 5p-core band [2]. Since the discovery of fast scintillations in BaF_2 , other inorganic crystals were found to exhibit CL, e.g. the alkali halides CsF , CsCl , CsBr , RbF , RbCl , and KF and ternary compounds like KMgF_3 , KCaF_3 , KLuF_4 , K_2YF_5 , BaLiF_3 , BaMgF_4 , BaY_2F_8 , BaZnF_4 , CsCaCl_3 [3, 4, 5, 6, 7, 8, 9]. Extrinsic CL was reported for the cation doped alkali halides $\text{KF}\text{:Rb}$, $\text{KCl}\text{:Cs}$, and $\text{RbCl}\text{:Cs}$ [3, 10]. In these materials, electron transitions from the halogen-related valence band to holes at impurity cations produce CL. The CL decay time is usually very short (ns) which is a highly desirable property for fast timing purposes in radiation detection systems.

In this work, we report on the scintillation properties of the ternary inorganic crystals KMgF_3 , RbMgF_3 , $\text{KYF}_4\text{:Rb}$, K_2YF_5 , KZnF_3 , BaTm_2F_8 , KLuF_4 , $\text{LiYF}_4\text{:Nd}$ and $\text{BaF}_2\text{:Rb}$. The scintillation light output of these crystals was estimated from the X-ray induced emission spectra. Optical absorption spectra and decay time spectra were also measured and are presented. The Rb^+ doped materials are of interest because of possible extrinsic CL

Experimental details

The K_2YF_5 , $\text{KYF}_4\text{:Rb}$ and KLuF_4 single crystals were obtained by the hydrothermal synthesis method in the reaction of water solutions of KF with the corresponding oxides of rare earth elements. The synthesis was performed in a 40 cm³ autoclave with copper ampullae at ≈ 750 K temperature and 100-150 MPa pressure. The other crystals were grown by the conventional Bridgman method in graphite crucibles under protective atmosphere. The crystals were not analysed for the content of trace impurities. The authors are grateful to R. Yu Abdulsabirov, S.V. Petrov, V.M. Reiterov, N.I. Silkin, and H.W. den Hartog for some of the crystals studied in this work.

An ARC (Acton Research Corporation) vacuum monochromator (model VM-502) with a 1200 grooves/mm concave holographic grating blazed in first order at 250 nm was employed to measure the luminescence spectra of the crystals. The crystals were excited with X-rays from an X-ray tube with a copper anode operating at 35 kV. A diaphragm located between the crystal and the X-ray tube assured a well defined X-ray beam on the crystal surface. Several pure BaF_2 crystals with different dimensions showed within 5% the same luminescence intensity. The emission spectra recorded with our set-up is therefore rather independent on crystal dimensions.

An XP2020Q photomultiplier (PM)-tube operating in current mode was mounted behind the exit slit of the monochromator and measures the transmitted light. Since the PM-tube is insensitive at wavelengths smaller than 170

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nm, a Na-salicylate coated glass window with an XP2020 PM-tube was used for measuring emission spectra near these wavelengths. The emission spectra presented in this work were corrected for the quantum efficiencies (QE) of the photon detectors. We employed the QE of the PM-tube as specified by the manufacturer. The QE of Na-salicylate at wavelengths between 170 and 240 nm was determined by comparing the spectrum of a deuterium lamp recorded with Na-salicylate with the one recorded with the PM-tube; below 170 nm a constant extrapolated value was assumed. An estimate for the relative photon yield was obtained by comparing these corrected emission spectra with the one of a pure BaF_2 crystal recorded under identical conditions. Our pure BaF_2 reference crystal, if excited with 662 keV gamma rays, has a total light yield of 10000 photons per MeV absorbed gamma ray energy of which 1800 ph/MeV are created by core-valence transitions. With these values and the assumption that the light yield/(unit energy) for about 10 keV X-rays is the same as for 662 keV gamma rays, an absolute estimate for the photon yield was obtained. The, in this work reported, photon yields of emissions observed at wavelengths below 200 nm can have a systematic error of possibly 30% because the quantum efficiency of Na-Salicylate and the XP2020Q PM-tube are not accurately known below 200 nm. Furthermore, the results were not corrected for the transmission of the monochromator. In addition, there is a stochastic error of about 15% in the reported photon yields.

The optical absorption of the crystals between 200 and 600 nm was measured by means of a Perkin-Elmer spectrophotometer (model lambda 9). An ARC (model DS-775) deuterium lamp was used to determine the optical absorption of the crystals at wavelengths between 110 and 240 nm. Decay time spectra of the scintillation emission from the crystals were determined by means of single photon counting techniques. We employed a modified version of the method described by Bollinger *et al.* [13]. The decay time spectra were corrected for dead time and counts caused by random coincidences.

Results

A. KMgF_3 . CL in KMgF_3 between 140 and 190 nm was reported some years ago by Jansons *et al.* [3]. Buzlutskov *et al.* [11] reported an intensity about 43% of that of pure BaF_2 . We studied two cylindrical KMgF_3 crystals with dimensions $\varnothing 6.7 \times 3.1$ mm and $\varnothing 6.5 \times 1.6$ mm containing traces of Eu^{2+} . The emission, optical transmission, and decay time spectra of the 3 mm thick crystal are shown in figures 1, 2 and 3 respectively. The CL can be observed between 140 and 190 nm and has a decay time of 1.5 ± 0.3 ns. The transmission of the crystal at 140 and 190 nm is 15% and 59%, respectively. From the integrated emission spectrum, we obtain a CL light yield of about 2400 photons/MeV. The 1.6 mm thick crystal had a CL light yield

of 3500 ph/MeV. This larger value is caused by a better optical transmission of the crystal near 170 nm.

Figure 1: X-ray induced emission spectra at room temperature. Spectrum 1) 3.1 mm thick KMgF_3 ; 2) $\text{KYF}_4:\text{Rb}$; 3) K_2YF_5 ; 4) KLuF_4 . For illustration purposes, the last three spectra are shifted vertically by multiples of -10 ph/(MeV.nm).

Optical absorptions between 220 and 300 nm, partly visible in Fig. 2, are caused by 4f-5d transitions in Eu^{2+} . The emission peak at 363 nm with a light yield of 675 photons/MeV must be attributed to a 4f-4f ($^6\text{P}-^8\text{S}$) transition in Eu^{2+} [14]. Since the 4f-4f emission is forbidden according to the electric dipole approximation, its decay time is very long (about 1 ms [14]) and therefore of no practical interest for scintillation techniques.

Figure 2: Optical transmission spectra not corrected for Fresnel reflections of 1) 3.1 mm thick KMgF_3 ; 2) $\text{KYF}_4:\text{Rb}$; 3) K_2YF_5 ; 4) KLuF_4 .

B. K_2YF_5 and $\text{KYF}_4:\text{Rb}$. The emission, optical absorp-

tion, and decay time spectra of these crystals with dimensions $6 \times 8 \times 3.1$ mm and $5 \times 5 \times 0.75$ mm, respectively, are also shown in figures 1, 2, and 3. CL in K_2YF_5 with an intensity about 17% of that of pure BaF_2 was reported by Buzulutskov *et al.* [11]. We observe CL in the 140–220 nm region with a light yield of 510 ph/MeV and with a decay time of 1.3 ± 0.3 ns. A significant fraction of the CL is probably absorbed in the crystal because of the rather poor optical transmission of 7.5 % near 170 nm. The emission band at 320 nm might be caused by 5d-4f luminescence of Ce^{3+} impurities. There is slight evidence of a decay time component of about 20 ns and two optical absorption bands at 205 and 288 nm which correspond with 4f-5d transitions in Ce^{3+} . The narrower peaks near 378 nm, 415 nm, and 435 nm are possibly caused by 4f-4f luminescence of Nd^{3+} impurities.

Figure 3: Decay time spectra at room temperature and plotted on a $^{10}\log\text{-}^{10}\log$ scale. 1) 3.1 mm thick $KMgF_3$; 2) $KYF_4:Rb$; 3) K_2YF_5 ; 4) $KLuF_4$. The small peaks at 15 and 30 ns are artifacts caused by after pulses in a photomultiplier tube.

The KYF_4 crystal was doped with Rb^+ in order to obtain extrinsic CL. Initially holes are created by ionizing radiation in the K^+ 3p-shell, and it was anticipated that they would be trapped by Rb^+ ions. A transition of an electron from the F^- 2p-valence band to the relaxed hole would then produce extrinsic CL near 225 nm similar as it was observed in $KF:Rb$ crystals [3]. The $KYF_4:Rb$ crystal shows CL in the 140–190 nm region with a yield of 1800 ph/MeV, i.e. equal to that of pure BaF_2 , and has an optical transmission of 28% at 160 nm. The larger CL light yield as compared to K_2YF_4 is probably caused by the better optical transmission of the crystal. The emission lines in the 300 to 500 nm region, with a total light yield of 840 ph/MeV, are very similar to those of the K_2YF_5 crystal.

A faint emission band can be observed near 225 nm where the extrinsic CL was anticipated. Whether this is caused by extrinsic CL due to Rb or some other defect is not clear.

C. $KLuF_4$. Buzulutskov *et al.* [8] reported that $KLuF_4$ crystals show, like $KMgF_3$, CL between 140 and 200 nm. We studied a crystal with dimensions $6 \times 8 \times 1.2$ mm, its spectra are again shown in Figures 1, 2 and 3. Contrary to Buzulutskov *et al.* [8], we did not observe CL below 170 nm. There is, however, a fast component of 1.3 ± 0.3 ns in the decay time spectrum, possibly caused by CL between 170 and 200 nm with a yield of about 170 ph/MeV. The absence of CL below 170 nm can not be explained by optical self absorption in the crystal because the optical transmission at these wavelengths is reasonably good, see Figure 2. The origin of the faint broad emission band extending from 200 to 500 nm is not known. Its total light yield is about 540 ph/MeV.

D. $KZnF_3$, $BaTm_2F_8$ and $RbMgF_3$. These crystals did not show any evidence of CL. The $KZnF_3$ crystal has an emission band at 260 nm with a yield of about 2300 photons/MeV and a decay time larger than 5 microseconds. This emission is probably caused by Pb^{2+} contamination of the crystal. Optical absorption bands at 191, 176, and 155 nm also indicate the presence of Pb . The absence of CL is most likely caused by the overlapping of the Zn^{2+} 3d-core band with the F^- 2p-valence band. The authors are grateful to Dr. Yu. P. Kostikov for the measurements of XPS (X-ray photon spectroscopy) spectra on this crystal which revealed the overlap of the energy bands. The $BaTm_2F_8$ crystal showed between 180 nm and 600 nm optical absorption peaks and emission lines characteristic for the 4f-4f transitions in Tm^{3+} ions. The emissions were very weak with light yields smaller than 20 photons/MeV. Fast emissions, characteristic for CL were not observed, most likely because of the partly filled 4f shell of Tm^{3+} . For the same reason $KErF_4$, $KTmF_4$, and $KYbF_4$ do not emit a CL component [12, 8]. The emission spectrum of $RbMgF_3$ showed a weak (400 photons/MeV) broad emission band with some structure between 200 nm and 450 nm. An emission line with a width of about 60 nm and a total intensity of 1800 photons/MeV was observed near 575 nm. Their decay times were not measurable with our set-up and must be longer than 0.1 ms. This material is because of this long decay time not a practical scintillator. The absence of an expected measurable CL in $RbMgF_3$ can be caused by a too high concentration of Pb and other impurities.

E. $BaF_2:Rb$. Crystals of BaF_2 doped with Rb were studied because of a possible extrinsic CL. However, we did not observe any luminescence related to Rb^+ , and the anticipated extrinsic CL was therefore not observed. The only effect of Rb^+ doping was a reduction of the intensity of the STE emission of the host lattice.

F. $LiYF_4:Nd^{3+}$. The emission spectrum of a 7 mm thick $LiYF_4$ crystal doped with 0.9 mol% Nd is shown in figure 4a. The properties appear quite similar to those of Nd^{3+} doped LaF_3 crystals [15]. For comparison, the emission

spectrum of a LaF_3 crystal doped with 1.2 mol% Nd^{3+} is shown in figure 4b. The emission lines between 340 and 500 nm, with a total light yield of 250 and 1600 photons/MeV for $\text{LiYF}_4:\text{Nd}$ and $\text{LaF}_3:\text{Nd}$, respectively, are caused by 4f-4f transitions in Nd^{3+} ions. The 4f-4f luminescence decay times are usually very long (ms) and therefore not suitable for scintillation techniques. The emission lines at 182, 230, and 260 nm for $\text{LiYF}_4:\text{Nd}$ are caused by 5d-4f emissions. The optical absorption edge caused by 4f-5d absorption is located at 180 nm. The position of the 5d-4f emission differs somewhat from those in Nd^{3+} doped LaF_3 crystal because of a different interaction between the 5d shell of Nd^{3+} with the host lattice. The 184 nm emission in $\text{LiYF}_4:\text{Nd}$ and the 173 nm emission in $\text{LaF}_3:\text{Nd}$, both about 200 photons/MeV, are of interest because of detection with photosensitive gasses. The decay time was expected to be short since the 5d-4f transition is an allowed transition. We observed a decay time of about 40 ns for $\text{LiYF}_4:\text{Nd}$ and 6.3 ns for $\text{LaF}_3:\text{Nd}$ [15].

Figure 4: X-ray induced emission spectra. a) $\text{LiYF}_4:0.9$ mol% Nd^{3+} . b) $\text{LaF}_3:1.2$ mol% Nd^{3+} ; the intensity above 300 nm is divided by 5 for illustration purposes.

Discussion

Table 1 compiles the CL data obtained in this work. In order to interpret the photon yield/MeV one should consider the optical transmission of the crystals. The exciting X-rays are absorbed in the top layer of these crystals, and the created CL-photons have to transmit through the bulk material in order to be detected. Part of the photons will then be absorbed. If one corrects the photon yields for this self absorption, we obtain for KYF_4 , K_2YF_5 , and both crystals of KMgF_3 a photon yield/MeV of roughly 5000 at a bulk transmission of 100%. In the correction, we have assumed a linear dependence of the light yield on the transmittance. It seems therefore that these crystals,

Table 1: Compilation of cross-luminescence data obtained in this work. d = crystal thickness; λ = CL-emission wavelength; ph/MeV = photon yield per MeV absorbed X-ray energy with a possible (except for BaF_2) systematic error of 30%; τ = CL-decay time.

crystal	$d(\text{mm})$	$\lambda(\text{nm})$	ph/MeV	$\tau(\text{ns})$
BaF_2	5	195,220	1800 ± 100	0.8 ± 0.1
KMgF_3	3.1	140-190	2500 ± 400	1.5 ± 0.3
KMgF_3	1.6	140-190	3500 ± 500	—
KYF_4	0.75	170	1800 ± 300	1.9 ± 0.4
K_2YF_5	3.1	170	510 ± 80	1.3 ± 0.3
KLuF_4	1.2	170-200	170 ± 40	1.3 ± 0.3

if optically pure, have about equal CL-light yields which are about twice as large as that of pure BaF_2 .

Except for BaF_2 and CsF , little is known in the literature about the absolute CL light output. In many cases the intensity is reported relative to the intensity of some other crystal showing CL without a correction for the quantum efficiencies and transmissions of the photon detection systems. Sometimes, e.g. for KF and RbCl , only an order of magnitude is given. Furthermore, little is known about the optical quality of the crystals and the light collection efficiency. Nevertheless, we have tried to obtain an estimate for the photon yield/MeV of most CL-crystals reported in the literature up to date and compiled them in Table 2.

Principally, the CL light yield/MeV is determined by the number of holes/MeV created in the core band and the competition between radiative and non-radiative decay of the holes. The low light yield of KF , RbCl , and CsBr at room temperature and the extremely short decay time of 70 ps reported for CsBr [5], for instance, are caused by competing Auger decay of the holes [5, 6]. The crystals RbF , KMgF_3 , KCaF_3 , CsBr at 80K, and probably CsCl at 80K, have light yields comparable with those of BaF_2 and CsF . The CL photon yields reported by Buzulutskov *et al.* [11, 8] of the potassium compounds KMgF_3 and K_2YF_5 are significantly smaller than observed by us, compare Table 1 with 2. It is not known to what extent these yields are affected by the optical properties and the shape of the crystals. Melchakov *et al.* and Rodnyi *et al.* [7, 9, 17] report for CsCaCl_3 , CsSrCl_3 , RbCaF_3 , BaLiF_3 , BaMgF_4 , BaZnF_4 , and BaY_2F_8 CL photon yields comparable with that of pure BaF_2 and decay times of the order of 1 ns. It appears from the results in Table 2 that, generally, the extra metal ion in the ternary compounds causes a small shift of the CL emission band to larger wavelengths. In addition, the Auger decay which sometimes dominates the radiative decay at room temperature is often of less importance in the ternary compounds. E.g. CL in KF is quenched at room temperature whereas Auger quenching is not observed for the ternary potassium compounds; the

Table 2: Compilation of CL data at room temperature reported in the literature. λ = CL-emission wavelength; ph/MeV = estimated photon yield per MeV absorbed energy. This number is in many cases a rough estimate and must be interpreted accordingly; τ = CL decay time.

crystal	λ (nm)	ph/MeV	τ (ns)	ref.
KF ^{a)}	156	50-200	—	[3]
RbF	203,234	1700	1.3 ± 0.1	[4, 5]
CsF	390	2500	2.9 ± 0.1	[5, 16]
RbCl	190	0.5-2	—	[3]
CsCl ^{b)}	245,270	900	0.88 ± 0.07	[5]
CsCl	236,276	—	1.5 ± 0.5	[7]
CsBr	250	18	0.07	[5]
CsBr ^{c)}	250	1800	1.34 ± 0.05	[5]
KCaF ₃	155,171	1800	< 2	[3]
KMgF ₃	152,171	800 ± 250	< 2	[11, 8]
K ₂ YF ₅	180	200 ± 60	1.3 ± 0.3	[8]
KLuF ₄	165	400 ± 120	< 2	[8]
KLu ₂ F ₇	165	270	< 2	[11]
RbCaF ₃	—	1800	≈ 1	[17]
KCaCl ₃ ^{c)}	—	1800	≈ 1	[17]
CsCaCl ₃	248,305	1800	< 1.0	[7, 17]
CsSrCl ₃	—	1800	≈ 1	[17]
BaF ₂	195,220	1800	0.8 ± 0.1	[1]
BaLiF ₃ ^{d)}	228,252	1800	< 1.0	[9]

^{a)} at 80K, the emission is almost entirely quenched at room temperature due to Auger processes [3]. ^{b)} The light yield of CsCl increases by a factor of two upon cooling the crystal to 80 K [7]. ^{c)} at 80 K. ^{d)} BaMgF₄, BaY₂F₈, and BaZnF₄ show, in this order, increasingly lower CL intensity than BaLiF₃ [9].

same holds for KCl and CsCl and the related ternary compounds. These properties are probably caused by a widening of the bandgap between the conduction band and the upper valence band due to the presence of the extra metal ions.

Summarizing, the CL decay time of crystals known so far is usually between 1 and 2 ns, and there appears little variation with crystal type. BaF₂ is still the fastest scintillator with a decay time of 0.8 ± 0.1 ns. It seems that the CL photon yield of all crystals showing CL is of the same order of magnitude; a few thousand photons/MeV. Accurate numbers for the photon light yield are for most crystals not yet known.

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