Creation of storage centers in CsBr:Eu²⁺ needle image plates by vacuum ultraviolet radiation

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SPECIAL TOPICS

Creation of storage centers in CsBr:Eu²⁺ needle image plates by vacuum ultraviolet radiation

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In this study, the photostimulated luminescence of the CsBr:Eu²⁺ needle image plates (NIPS) after vacuum ultraviolet (VUV) irradiation and their photoluminescence under VUV and UV excitation were investigated. It was shown that the photostimulable storage centers arise almost exclusively due to irradiation of the CsBr:Eu NIPs in the spectral lines of creation of the $4p^55s$ anion excitons, and not in the region of interband transitions of CsBr. The explanation of the results is based mainly on the radiative and nonradiative decay of the self-localized $e+\text{Br}_2^-$ excitons. © 2009 American Institute of Physics. [doi:10.1063/1.3224860]

I. INTRODUCTION

Because of their unique properties the needle image plates (NIPs) based on the CsBr:Eu²⁺ storage phosphor are increasingly used for digital x-ray imaging. The use of the CsBr:Eu NIPs in digital radiography offers better image quality and a potential for exposure dose reduction of up to 75% compared to the dose required when granular BaFBr:Eu²⁺ image plates are used.¹ These NIPs have the potential to replace the granular image plates in medical diagnostics.² Offering a high lateral resolution combined with a high sensitivity³ and a dynamic range of several orders of magnitude, the CsBr:Eu NIPs are especially useful for mammography.

The operation of the x-ray image plates relies on the occurrence of x-ray induced photostimulated luminescence (PSL) in storage phosphors.⁴ In general, the absorption of x-ray irradiation induces, after multistage Auger processes, the generation of charge carriers, which can be stored in traps formed by crystal defects. Stimulated by red light, the electrons are released from their traps and recombine with the localized holes. The released energy is transferred to an activator ion resulting in luminescence with an intensity which is proportional to the x-ray dose absorbed by the image plate. Scanning the stimulating laser and recording the luminescence signal enables to obtain a radiograph.

Since the discovery of CsBr:Eu²⁺ as a storage phosphor for digital x-ray imaging⁵ there have been speculations about the nature of the charge carrier traps in this material. It is known that the electron is trapped by a Br⁻ vacancy forming an F -center,^{6,7} whereas the nature of the hole trap is still under discussion. The assumption that it is a Eu³⁺ could not be verified and it was concluded that the hole trap consists of a Eu²⁺ with a hole nearby.⁷ Further, it was supposed that it is an Eu²⁺- V_K -complex,⁸ similar to the one in BaFBr:Eu²⁺, which is necessary for the PSL storage and readout processes.⁹ However, the Eu²⁺- V_K aggregates in CsBr:Eu²⁺ are more complex: the EPR studies provide evidence that no

isolated Eu²⁺ ions exist in CsBr:Eu at room temperature.^{10,11} There is a defect close to the Eu²⁺ in the $\langle 001 \rangle$ direction.¹¹ Very probably, this is an oxygen ion, so the Eu²⁺-O²⁻ dumbbells + V_K structure is built.⁸ The latter model of the hole center in CsBr:Eu was supported by the investigation of the role of water molecules for the PSL in the needle-structured and powder samples.^{12,13} Moreover, much speaks for it that V_K -centers, created after irradiation, migrate toward the (Eu²⁺-O²⁻)-complexes with water molecules in their neighborhood, and are captured there.¹³

The creation mechanism of the hole and electron trap centers in CsBr:Eu storage phosphor was discussed in the literature much less. It has been suggested that there are naturally occurring V_K -centers (hole which is shared by two adjacent halogen ions) following the irradiation in this material.¹³ The forming of F -centers as a result of creation of new Br⁻ vacancies as well as a result of possible trapping of electrons by the pre-existing Br⁻ vacancies has not been discussed for CsBr:Eu yet. However, in undoped CsBr, the problem of creation of the F - H pairs (an F -center along with an interstitial Br⁻ ion) following UV-radiation has been investigated in details.¹⁴⁻¹⁶ It was determined that the creation of the F - H pairs occurs mostly due to irradiation with photons having energies corresponding to the exciton generation spectrum.

We report the PSL of the CsBr:Eu²⁺ NIPs after vacuum ultraviolet (VUV) irradiation as well as their photoluminescence (PL) under VUV and UV excitation and discuss the possible mechanisms of the creation of storage centers in CsBr:Eu²⁺.

II. EXPERIMENTAL

The experiments were carried out at the SUPERLUMI station of the HASYLAB, synchrotron center DESY in Hamburg, Germany, and were performed on the samples of CsBr:Eu²⁺ image plates produced by the Agfa Gevaert Company. The CsBr-layer thickness was about 320 μm and the samples had a Eu²⁺ dopant level of about 300 ppm. The emission and the excitation spectra as well as the formation

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FIG. 1. (Color online) Excitation spectra of the PL of a CsBr:Eu²⁺ NIP detected at 440 nm. The zero levels are shifted for clarity. The identification of the excitonic peaks is based on the data from the Table I and is explained in Sec. IV.

spectra of the PSL were recorded by an experimental setup consisting of the primary and the secondary monochromator, a liquid He cryostat, and a detecting system. The primary monochromator was working with a resolution of 0.1–0.3 nm providing the beam of about 10^{10} photons/s at 0.3 nm.

The PL spectra were measured at 9.8 K and at room temperature with the resolution of about 1 nm provided by the secondary monochromator. The excitation spectra of the samples were measured in a time integrated regime at 9.8 K and at room temperature in the range between 50 and 334 nm (resolution 0.1 nm, 1 nm step). For detection, a secondary monochromator (2 mm slit) was fixed either at 260 or 350 nm, or at the maximum of the PL emission of Eu²⁺ at 440 nm. A photon counting setup was employed for signal detection.

The formation spectrum of the PSL emission was taken at room temperature after exposing the samples for 15 min to the VUV irradiation at fixed wavelengths, in order to activate the samples. For the stimulation of the trapped electrons, a 650 nm diode laser was applied. The secondary monochromator was set to 440 nm, the maximum of the PSL emission. The decay of the PSL during the read-out was recorded, after the red laser was turned on. The dark count rate was about 30 photons/s and the background signal with the red laser turned on was about 70 photons/s. The count rate at the maximum of the PSL signal after activation of the samples amounted to several thousands of photons per second.

III. RESULTS

The formation of *F*- and *H*- centers in CsBr occurs very efficiently via the nonradiative decay of excitons. Consider-

ing this and the fact that the decomposition of excitons at existing defects such as *F*⁺-centers (Br[−]-vacancy) and/or Eu²⁺ ions is possible as well, one can suggest an important role of the excitons in the formation of storage centers in CsBr:Eu²⁺. Therefore, the UV and VUV excitation spectrum of the Eu²⁺ emission was measured, including the spectral region where the generation of excitons was expected. The resulting spectra at 9.8 K and at room temperature are shown in Fig. 1.

Between 3.7 and 6.0 eV, the T_{2g} and E_g absorption bands of the $4f^65d$ level of Eu²⁺ are excited (9.8 K). Irradiation between 6.0 and 6.6 eV leads to Eu²⁺ emission as well. We suggest that the origin of these bands is related to the intrinsic lattice defects, which are not related to Eu²⁺ ions. From other experiments (not reported here) it has been found that these states are also present in undoped CsBr NIPs.

Close to the band gap in CsBr (7.30 eV at 4.2 K),¹⁶ the excitation maxima at 6.80, 7.04, and at 7.29 eV were detected. The first maximum is assigned to the creation of $4p^55s$ anion excitons. The maxima at 7.04 and 7.29 eV we interpret as a creation of $4p^54d$ excitons. The energetic positions of the excitonic absorption in NIPs measured in this work via the luminescence of Eu²⁺ differ insignificantly from the values of the absorption maxima for the thin films given by Teegarden *et al.*¹⁷ at 10 K and from the excitation maxima of the self-trapped dihalide exciton luminescence at 3.5 eV measured on single crystals at 4.2 K by Alexandrov *et al.*¹⁶ (see Table I).

For the identification of the excitation maxima near the band gap, the data on absorption and excitation were decisive.^{18,19} The interpretation of the reflection spectra is more complicated due to the conventional shift between the corresponding reflection and absorption/excitation maxima of several millielectron volts.

At energies higher than the band gap, the observed maxima correspond to electron excitations involving the outer shell of the Br[−] ion, and to the electron excitations involving $5p^6$ shell of Cs⁺, as interpreted by Alexandrov *et al.*¹⁶ At room temperature, the T_{2g} and E_g bands of the $4f^65d$ level of Eu²⁺ as well as the maxima at 6.39 eV (194 nm) and 6.93 eV (179 nm) were detected. We suggest that the excitation peaks at 179 nm (6.93 eV) and 194 nm (6.39 eV) belong to the generation of anion excitons in CsBr lattice. The line at 6.39 eV is interpreted as the p^5s exciton, which at 9.8 K peaks at 6.80 eV (182.4 nm) and is shifted to 6.39 eV (194 nm) at 300 K. This shift (0.41 eV) corresponds roughly to the thermal shift in 0.53 eV of the CsBr band gap, estimated

TABLE I. Excitonic peaks in CsBr and CsBr:Eu (this work) in absorption, reflection or excitation spectra (in electron volt) and their assignment.

Reference 15	Reference 16		Reference 18 and 19	Reference 17	This work
$T=4.2$ K	$T=4.2$ K		$T=80$ K	$T=10$ K	$T=9.8$ K
Reflection	Excitation	Reflection	Absorption	Absorption	Excitation
6.85 (p^5s)	6.88 (—)	6.90 (p^5s)	6.8 (p^5s)	6.81 (p^5s)	6.80 (p^5s)
7.11 (p^5d)	7.07 (—)	7.11 (p^5d)	7.11 (p^5d)	7.09 (p^5d)	7.04 (p^5d -doublet)
7.17 (p^5d)	7.16 (—)	7.19 (p^5d)	7.18 (p^5d)	7.17 (p^5d)	
	7.25 (—)	7.27 (p^5d)			7.29 (probably p^5d)
		7.42 (—)	7.4 (p^5s)	7.4 (p^5s)	7.55 (probably p^5s)

FIG. 2. (Color online) Formation spectrum of the PSL, stimulated at 633 nm (squares) and excitation spectrum of the Eu^{2+} luminescence. Both are detected at 440 nm at room temperature.

from the data presented in Refs. 16 and 17 and using the Varshni's²⁰ equation. The band at 6.93 eV (179 nm) belongs, very probably, to the creation of the p^5d exciton. Similar peaks (at 178.5 and 189.5 nm) were found by Zorenko *et al.*²¹ in the RT excitation spectrum of the Eu^{2+} emission in a CsBr:Eu single crystal and were assigned to the anion excitons as well.

The spectral dependence of the PSL activation was determined as follows. Knowing the exact spectral positions of the excitonic absorption, the PSL signal of a CsBr:Eu NIP (Fig. 2, dashed line, squares) was measured after activation at different photon energies, especially at the energies (wavelengths) corresponding to these distinctive wavelengths (Fig. 2, solid line) at room temperature. Charging at the wavelengths of the excitation maxima of Eu^{2+} was checked as well to prove a possible ionization of the Eu^{2+} ions due to UV irradiation and following trapping of escaped electrons by existing defects in the lattice. The experiment was repeated twice using two different sites on the NIP.

As can be seen in Fig. 2, the most efficient way to create the PSL active centers is the irradiation with photon energies in the excitonic absorption area. Irradiation with UV-light between 220 and 330 nm (excitation of Eu^{2+}) causes no observable storage effect. Surprisingly, activation energies exceeding the band gap (E_{bandgap}) do not lead to the PSL. Exclusively the direct irradiation in the excitonic absorption bands results in the creation of the storage centers.

Further, the measurements of the excitonic luminescence were carried out in order to prove the manifestation of this competition process with the nonradiative decay with possible formation of the F - H pairs or electron-hole pairs. Such as in undoped CsBr,^{15,16} luminescence of the dihalide self-localized excitons (DSLE) with the emission maxima at 4.7 eV (264 nm) and 3.5 eV (354 nm) was expected at 4.2 K. For excitation, photon energies which exceed the band gap (8.86 eV, 140 nm) as well as energies corresponding the creation of the $4p^54d$ excitons (7.04 eV, 176.2 nm) and the $4p^55s$ excitons (6.80 eV, 182.4 nm) were chosen and the emission spectra were recorded at 9.8 K (Fig. 3).

Such as in the undoped CsBr single crystals, the exciton emission bands were observed. The high-energy excitation (140 nm) generates free electron-hole pairs and leads to the creation and subsequent emission of the singlet (λ_{em}

FIG. 3. (Color online) Emission spectra of the Eu^{2+} ions and DSLE of an optimally annealed CsBr:Eu NIP under excitation: (1) at 140 nm (band-to-band), (2) at 176.2 nm (p^5d -), and (3) at 182.4 nm (p^5s -excitons). $T = 9.8$ K.

$= 264$ nm) and triplet ($\lambda_{\text{em}} = 355$ nm) excitons. A part of the excitation energy is transmitted to the Eu^{2+} ions resulting in the blue emission. A similar spectrum was recorded for the excitation at 176.2 nm, but with a lower intensity in the 355 nm band. Surprisingly, the emission of the DSLE under excitation wavelength 182.4 nm corresponding to the generation of the p^5s -excitons is only slightly above the background level (Fig. 3, curve 3). The intensity of the Eu^{2+} emission is strongest for this excitation and the spectrum does not contain any components in the spectral region over 500 nm, as is the case for the excitation at 140 and 176.2 nm.

IV. DISCUSSION

The experiments demonstrate that storage centers in CsBr:Eu²⁺ are preferentially formed at wavelengths corresponding to the creation of excitons in the CsBr matrix: in the case of UV irradiation in the near band gap region of CsBr, the PSL can be detected only after direct irradiation in the excitonic absorption bands (Fig. 2). In addition, the significant difference in the creation efficiency of storage centers with the participation of the p^5s - and p^5d -excitons was found.

We suggest that the storage centers originate predominantly from the well-known "Itoh-process"^{14,15,22} which is valid for alkali halides. It is very probable that the creation of the F - and V_K -centers in Eu-doped CsBr occurs similarly to the undoped CsBr. The absorption of the UV-quanta generates free excitons, which can relax to $V_K + e^-$ -centers due to self-trapping of the hole component. The F - H -pairs can be created via a nonradiative decay of the self-trapped excitons. These excitons are $\text{Br}_2 + e^-$ -centers in CsBr, i.e., DSLE. If the F - H -pair receives some energy (e.g., via the excitation of a F -center in the read-out process), it can stimulate the transformation of the H - to the V_K -center and further to the DSLE,¹⁵ which can decay radiatively and transfer its energy to an activator, e.g., to the Eu^{2+} , giving rise to the PSL.

At the same time, free excitons as well as DSLE can decay at the existing defects in the crystal lattice, such as F^+ -centers (Br^- -vacancy) forming F -centers and V_K (Br_2)-centers. For the well-known storage phosphor BaFBr:Eu²⁺, this mechanism as well as the relative contri-

bution of the F - H -creation mechanism to the storage center formation have been described in detail.^{23,24} In CsBr:Eu²⁺ storage phosphor the issue has not been investigated yet.

The interpretation of the results depicted in Fig. 2 as the creation of new defects is strongly supported by the data of Alexandrov *et al.*¹⁶ showing that the creation of the Frenkel's defects in CsBr occurs exclusively after irradiation with UV photons in the spectral region of the exciton absorption. At 4.2 K, at high photon energies ($h\nu \geq E_{\text{bandgap}}$) when free electrons and holes are created, the formation of the Frenkel's defects and, subsequently, the F - H pairs is much less efficient than at photon energies in the region of creation of the anion excitons. Suggesting that this mechanism is valid at room temperature, and that some H -centers are stabilized in the neighborhood of Eu²⁺ ions, as was supposed in Ref. 25 for the V_K center in BaFBr:Eu²⁺, the creation of the stable F - H pairs and, accordingly, of storage centers should be expected in CsBr:Eu at excitonic energies.

However, the creation of the F - H pairs as possible storage centers can accompany the above mentioned decay of the DSLE at existing defects such as F^+ -centers with the following formation of V_K^- and F -centers. The presented results and the discussed literature data cannot prove definitely the contribution of the two mentioned mechanisms at room temperature. Nevertheless, the DSLE emission at 9.8 K excited by photons with energies above the band gap and exactly in the excitonic maxima (Fig. 3) provides evidence of the significant contribution of the F - H creation mechanism to the storage centers formation in CsBr:Eu²⁺. We suggest that the different barriers¹⁵ of the self-localization for excitons created in CsBr with photon energies higher or lower than 6.9 eV at 4.2 K are the same in CsBr:Eu²⁺ at 9.8 K. The photon energy of 6.8 eV and the energies of 7.04 and 8.9 eV, which were used in our experiment for CsBr:Eu (Fig. 3), are situated at different sides of this 6.9 eV threshold. Therefore, a difference in the mean free path of the $4p^55d$ exciton (6.8 eV excitation, high barrier of the self-localization) and of the $4p^54d$ one (7.04 eV excitation, low barrier) up to self-trapping should be noticeable. Accordingly, one should expect a relatively strong DSLE emission under the excitation in the 7.04 eV (176.2 nm) band and at 8.9 eV (140 nm) due to the rapid self-trapping and the radiative decay of the DSLE at the regular sites of the crystal lattice. The Eu²⁺ emission is excited very probably via the dipole-dipole interaction due to the overlap of the DSLE emission spectrum (Fig. 3, curves 1 and 2) and the absorption spectrum of Eu²⁺ in CsBr.⁷ At the 6.8 eV (182.4 nm) excitation, free excitons migrate at higher distances, therefore the probability of self-trapping in the neighborhood of Eu²⁺ is higher than at the 176.2 nm excitation. As a result of the trapping near the Eu²⁺ center, the energy transfer from DSLE to Eu²⁺ is very effective, so practically no luminescence of the DSLE and only the Eu²⁺ emission could be detected (Fig. 3, curve 3). The narrow (in comparison with the other two excitation wavelengths) emission spectrum seems to be caused by the electronic transition in a certain kind of the of Eu²⁺-containing center only, probably in the PSL relevant Eu²⁺-O²⁻ complex.^{8,13}

If one suggests that the probability of the self-localization of the excitons at room temperature behaves in a similar manner and is lower for the $4p^55s$ excitons (194 nm) than for the $4p^54d$ exciton (179 nm), the formation spectrum of the PSL can be explained as well. Indeed, the self-trapping and the decay of the DSLE will occur mainly in the regular lattice at the irradiation energies higher than the band gap. Here it is not essential what kind of decay of the DSLE takes place: whether the creation of the new F - H pairs or the decay at the existing F^+ -centers. V_K -centers can migrate at room temperature to the electron centers and recombine with the electron centers giving rise to the 360 nm emission.¹³ After irradiating with 179 nm photons ($4p^54d$ -exciton), the probability of the self-trapping in the regular lattice is lower, and some part of the free excitons will reach Eu²⁺ and be self-localized and decay there. The hole will be stabilized as V_K -center by the Eu²⁺ ion and the electron will be trapped somewhere nearby, for example, at the F^+ -center. The appearance of storage centers leads to PSL. The storage effect should be much larger under the irradiation at 194 nm ($4p^55s$ -excitons) due to the lower probability of self-localization in the regular lattice. In this case the strongest PSL signal is detected (see Fig. 2). The significant effect of the relatively low Eu doping (300 ppm) on the exciton decay at sites close to Eu²⁺ can be explained with an enhanced exciton trapping cross-section due to the existence in CsBr:Eu of the (Eu²⁺-O²⁻)⁻ dipoles⁸ with the (Eu-water) complexes.^{12,13}

The experimental data presented above do not give evidence about the ratio of the DSLE decay at the existing defects and the forming of the F - H pairs. A possible way to determine this ratio is to find the fraction of the correlated electron-hole centers at irradiation in the excitonic bands. For the Eu²⁺ concentration of 0.1 mol % in CsBr and x-ray irradiation it was determined to be at about 50%.²⁶

V. CONCLUSIONS

The comparison of the excitation spectrum of the Eu²⁺ PL with the formation spectrum of the PSL shows that the creation of the storage centers in CsBr:Eu NIPs at room temperature occurs almost exclusively at photon energies corresponding to the creation of $4p^55s$ anion excitons and not to the interband transitions of CsBr. Moreover, a very low radiative decay rate of the DSLE was observed under excitation into the $4p^55s$ line at 9.8 K. It is concluded that the storage centers in the CsBr:Eu storage phosphor are formed via the nonradiative decay of the DSLE close to the Eu²⁺-containing emission centers. This conclusion is in good agreement with the literature data on the self-localization barrier of free excitons and with the formation spectrum of the F - H pairs in undoped CsBr. It is suggested that the DSLEs decay in two ways: (1) by creation of new F - H pairs in the neighborhood of Eu²⁺, (2) and by storage of holes as V_K centers close to the Eu²⁺ ions and trapping of electrons at existing F^+ centers at a distance.

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