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The photostimulation process in the x-ray storage phosphor KBr:In

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Abstract. The nature of the radiation damage centres generated by x-irradiation at 300 K, 100 K and 10 K and the photostimulation process are investigated in KBr:In. Measurements of the optical absorption, the magnetic circular dichroism of the optical absorption (MCDA) and the MCDA-detected electron paramagnetic resonance, as well as of the photostimulated In^+ emission, are utilized. At room temperature, F centres, In^{2+} centres and $\text{In}^0(1)$ centres are generated. In^{2+} centres are not produced at low temperature and therefore are not primary hole defects. It is shown that the photostimulation process does not occur within F- In^{2+} centre pairs, as previously proposed. It involves an aggregate between F centres, In^+ centres and an unknown, room-temperature-stable hole centre. The photostimulation process, including replenishment effects, seems very similar to the one found earlier for the well known x-ray storage phosphor BaFBr:Eu²⁺.

1. Introduction

X-ray storage phosphors are materials capable of storing images produced by the absorption of x-radiation. During image formation, radiation-induced defects are formed. It is generally believed that room-temperature-stable electron and hole trap centres are involved in the image formation. For the read-out process, usually the electron trap centres are photoexcited, and the luminescence of a doped activator is recorded. The x-ray storage phosphors can offer a number of advantages compared to conventional x-ray films: high sensitivity and a higher dynamical range (10^4 – 10^5) as well as the possibility of directly obtaining digitized x-ray images. Therefore, there is much interest in finding suitable systems. Several systems have been proposed (for a concise recent review, see Blasse 1993). The best known x-ray storage phosphor is BaFBr:Eu²⁺, which is already used commercially (Sonoda *et al* 1983). In spite of many efforts to understand the mechanism of the information storage and read-out process, no complete understanding has yet been achieved (see, e.g., Blasse 1993, Spaeth *et al* 1994a). In particular, the nature of the room-temperature-stable hole centres is not clear, nor the mechanism of energy transfer to the activator after photostimulated F centre electrons have recombined with the hole centres (Spaeth *et al* 1994a and references therein). It was shown recently that in BaFBr:Eu²⁺ an apparently unavoidable oxygen contamination greatly influences the formation of electron trap centres and provides hole trapping defects (Koschnick *et al* 1991, Eachus *et al* 1991).

It has been shown recently that KBr doped with In also has x-ray storage phosphor properties, whereby the efficiency of this material is of the same order of magnitude as that of BaFBr:Eu²⁺ (Kalnins *et al* 1991). It was postulated on the basis of optical investigations

that the x-ray energy storage is mainly due to the formation of F centre-In²⁺ centre pairs (Plavina *et al* 1991). If this were true, one would possibly have with KBr:In a simple model system to study the mechanism of the storage and read-out processes in detail.

It was the purpose of this investigation to use magneto-optical and optically detected electron paramagnetic resonance techniques to identify the nature of the radiation-induced defects in KBr:In and to study their role in the storage and read-out processes. The electron paramagnetic resonance (EPR) of the defects was measured via magnetic circular dichroism of the optical absorption (MCDA) (see e.g. Spaeth *et al* 1992) because with this method the high sensitivity necessary to detect the radiation damage centres was achieved as well as a correlation to their optical absorption bands. The development of the MCDA bands upon x-irradiation at various temperatures and their changes caused by the photostimulation process was studied.

It turned out that the postulation of In²⁺-Fe centre pairs as electron hole trap centres for the energy storage in KBr:In was not correct and that the storage and read-out mechanism seems to be very similar to the one thought to be present in BaFBr:Eu²⁺.

A preliminary but incomplete account of this work was presented elsewhere (Rogulis *et al* 1994).

2. Experimental details

The KBr:In single crystals were grown with the Bridgman-Stockbarger method. The KBr was treated prior to the crystal growth with NH₄ Br to minimize the oxygen contamination. The In doping level was $(1-2) \times 10^{17} \text{ cm}^{-3}$. This was determined using the optical absorption with the In⁺ oscillator strength (Smith and Dexter 1972).

X-irradiation was performed with a fine-structure tube with either 50 kV, 30 mA or 60 kV, 15 mA. The irradiation times were 0.5-2 h. The samples could be x-irradiated *in situ* in the spectrometer to measure the MCDA and MCDA-detected paramagnetic resonance at 4.2 K. In this irradiation the x-ray tube was further away from the sample compared to the room-temperature irradiation.

The photostimulation was performed with an He-Ne laser (5 mW) in the ODEPR apparatus, while in the optical spectrometer a combination of halogen lamp (150 W) and a double monochromator were used. The photostimulation time varied between 1 min (RT) and 15 min (low temperature). The optical absorption and emission spectra could be measured between 10 K and 300 K with a computer-controlled custom-built optical spectrometer, using a 0.25 Spex double monochromator and a phototube for the visible spectral range.

The MCDA, which is the differential absorption for right and left circularly polarized light, is proportional to the population difference of the Zeeman levels of the ground state of a paramagnetic defect. Upon saturating microwave transitions between the Zeeman levels it decreases, which can be used to detect the EPR of the ground state of the paramagnetic centre (see e.g. Spaeth *et al* 1992). This MCDA-detected EPR was used to identify the various paramagnetic radiation-induced defects. The MCDA, its excitation spectra ('tagged' MCDA) and MCDA-detected EPR were measured in a computer-controlled custom-built K band (24 GHz) MCDA-EPR spectrometer, usually at 1.5 K. For the infrared spectral range, a Ge detector (Northcoast) was used.

3. Experimental results

3.1. X-irradiation at room temperature

KBr:In crystals were irradiated with x-rays at room temperature for approximately 2 h, then

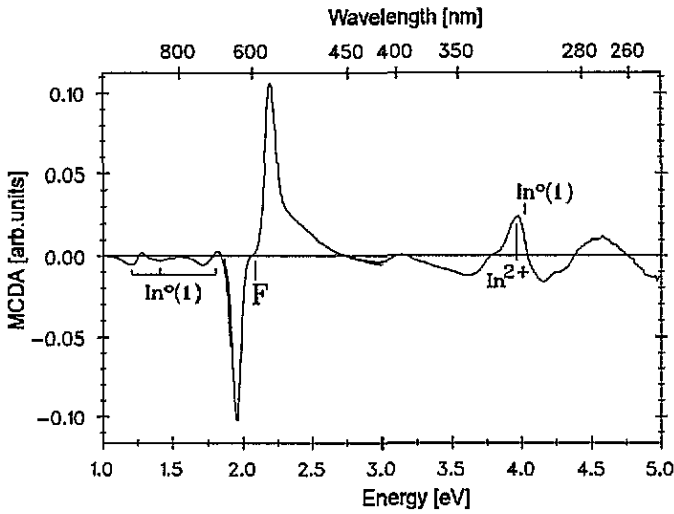


Figure 1. The MCDA spectrum of a KBr:In crystal x-irradiated at room temperature, measured at $T = 1.5$ K, $B = 2$ T. The MCDA bands of those paramagnetic defects identified by MCDA-detected EPR measurements are marked.

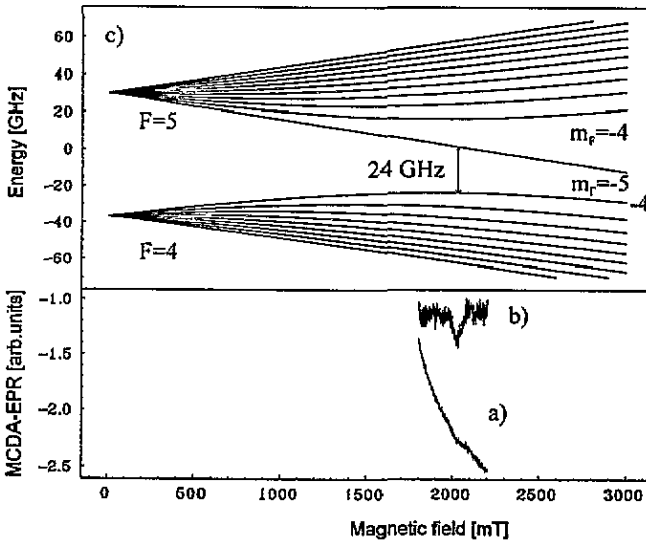


Figure 2. Lower part, EPR spectrum of In^{2+} centres in KBr:In measured as a microwave-induced change of the MCDA spectrum of figure 1 at 3.95 eV. $B_0 \parallel [100]$, $\nu_{\text{ESR}} = 24.21$ GHz, $T = 1.5$ K. Curve a, as measured; curve b, after subtraction of the MCDA background signal. Upper part, c, calculated Breit-Rabi diagram for the In^{2+} centre.

cooled to 1.5 K for magneto-optical measurements. Figure 1 shows the MCDA measured between 1.0 and 5.0 eV. The strongest MCDA band with a derivative-like structure centred at 2.06 eV is from F centres. The three bands at the low-energy side are due to $\text{In}^0(1)$ centres, i.e. In^0 atoms on cation sites next to a halogen vacancy (Ahlers *et al* 1984). The MCDA

bands at about 4.0 eV are due to a superposition of bands from $\text{In}^0(1)$ and In^{2+} centres as well as some unknown defects. Figure 2 (lower part) shows the MCDA-detected spectrum of In^{2+} centres measured at 3.95 eV. A weak EPR line is seen at 2040 mT. The spectrum is isotropic. In figure 2 (upper part) the Breit–Rabi diagram shows that only one EPR transition could be recorded with the microwave frequency of 24 GHz. The analysis yields that the In^{2+} hyperfine constant is $A = (13.2 \pm 1)$ GHz and $g = 2$. A hole is trapped at the In^+ ion and forms the paramagnetic In^{2+} ion, which has a $5s^1$ electron configuration (Rogulis *et al* 1994). In figure 3, the ‘tagged’ MCDA spectrum, which is an excitation spectrum of the MCDA-detected EPR line (see e.g. Spaeth *et al* 1992), shows that the In^{2+} MCDA band strongly overlaps one MCDA band of the $\text{In}^0(1)$ centres at 3.99 eV (figure 3, curves b and c). Thus, upon room-temperature x-irradiation, the electron traps $\text{In}^0(1)$ and F centres as well as the hole traps In^{2+} are produced. Apart from those MCDA bands identified by their MCDA-detected EPR spectra, there are other bands that are temperature dependent and therefore are caused by paramagnetic defects. The MCDA in the spectral range between 4.3 and 5.0 eV coincides with an absorption band associated with the V_2 centres, which are thought to be due to halogen di-interstitials (Ishii 1966). However, we could not observe any MCDA-detected EPR spectra. In the spectral range between 2.5 and 3.5 eV, the weak MCDA bands are also from paramagnetic centres. Their appearance depends on the crystal purity. In some crystals, a perturbed In^0 centre was identified ($\text{In}^0(\text{X})$) with MCDA bands at 2.3 and 3.0 eV (to be published). In particular, we did not find the MCDA-detected EPR of O^- centres in spite of a careful search.

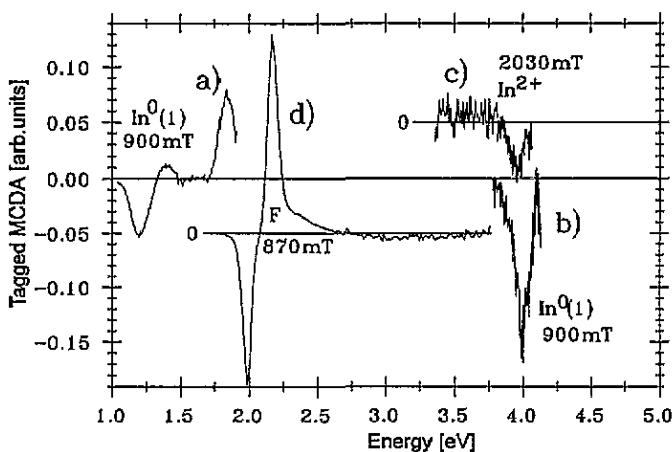


Figure 3. The tagged MCDA spectra of $\text{In}^0(1)$ centres (curves a and b, measured at 900 mT), F centres (curve d, measured at 870 mT) and In^{2+} centres (curve c, measured at 2030 mT) in KBr after x-irradiation at room temperature.

Upon photostimulation into the F band at room temperature, two luminescence bands of In^+ centres are observed around 2.9 eV and 2.4 eV (Kalnins *et al* 1991, Plavina *et al* 1991, Trinklere *et al* 1993, Fukuda 1970) (figure 4). The intensity of the photostimulated luminescence (PSL) bands cannot be compared with those measured at low temperature because of the fast decay of the PSL as shown in figure 5 (see below). After exhaustion of the PSL, the crystal was cooled to 1.5 K, and the MCDA was again measured. After a strong bleaching at room temperature all F centres have disappeared, as well as all In^{2+}

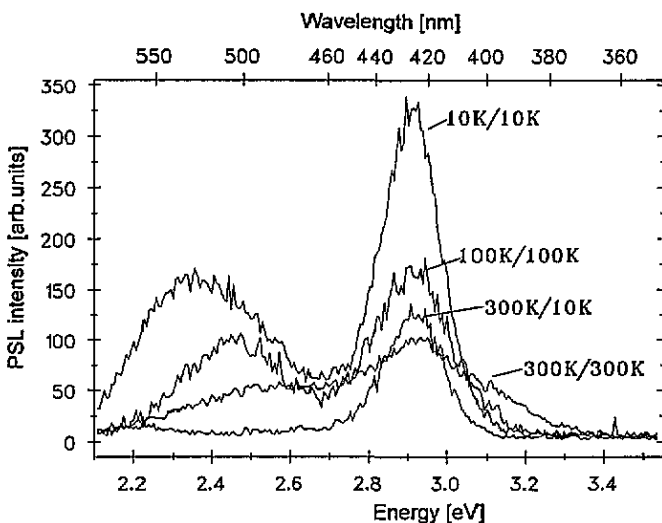


Figure 4. Photostimulation spectra of KBr:In after x-irradiation at 300 K, 100 K and 10 K (first temperature at curves) and measurement at 300 K, 100 K and 10 K (second temperature at curves). The photostimulation was performed at 604 nm with very weak light, such that only a very low loss of luminescence, i.e. of photostimulable centres, occurs during the measurement. For the low-temperature photoexcitation, the different luminescence intensities can be compared, because the conditions (exciting light intensity and decay times) are the same. For the room-temperature luminescence, the intensity is not comparable to the other ones because of the much faster decay that occurred during the measurement.

centres, whereas the $\text{In}^0(1)$ centres remained unchanged. If the bleaching is finished when over 95% of the PSL has occurred, then about one-third of the initial F centre band remains. The disappearance of the In^{2+} centres could lead to the conclusion that F centres and In^{2+} centres are the correlated electron-hole pairs responsible for the energy storage and photostimulation. However, as is seen from the following experiments, this is too simple a picture.

When x-irradiating a crystal at room temperature and subsequently cooling it down to 10 K, a photostimulation in the F band at 10 K also generates the In^+ luminescence band at 2.9 eV. At low temperature, only the band at 2.9 eV is observed (Fukuda 1970). The luminescence intensity is shown in figure 4 in comparison to that measured after low-temperature x-irradiation (see below). Its intensity is smaller than that found for low-temperature irradiation. Figure 5 shows the PSL intensity as a function of time for continuous bleaching. It is seen that, when exciting at room temperature, the PSL decays much faster than when exciting at 10 K. In contrast to the photostimulation at room temperature, only a small fraction of the F centres has disappeared. The MCDA of In^{2+} centres has not changed measurably, nor that of $\text{In}^0(1)$ centres. The MCDA between 4.3 and 5.0 eV does not change either. Thus, this MCDA is not from hole centres involved in the PSL process, and F centres and In^{2+} centres are not spatially correlated such that they can easily recombine at low temperature. However, the following experiments show that at higher temperatures dynamical effects are involved in the PSL mechanism. We have observed a replenishment effect similar to the effect observed previously in the storage phosphor BaFbr:Eu^{2+} by Hangleiter *et al* (1990, 1991).

After exhausting the PSL at 10 K, warming the crystal to room temperature and again cooling to 10 K, the crystal regained photostimulability. Again, the In^+ PSL can be generated

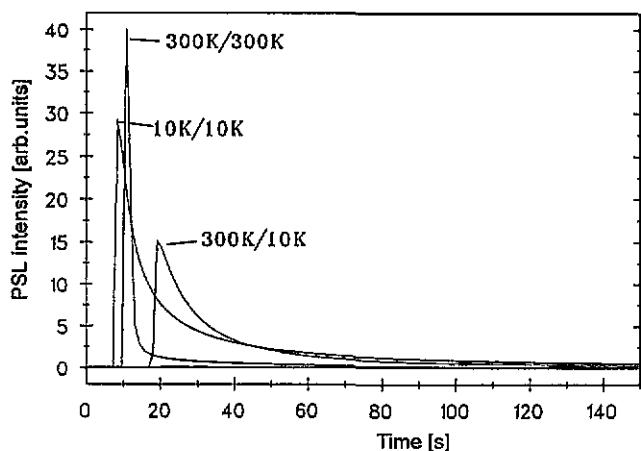


Figure 5. PSL measured at 2.9 eV as a function of time during continuous photoexcitation at 2.05 eV. Given by the curves are the x-irradiation temperature (the first temperature) and that of the PSL measurement (the second temperature). The three starting times of the measurements are shifted with respect to each other to enable a clearer presentation of the results.

by bleaching into the F band, although weaker compared to the first time. About one-third to one-half of the first PSL intensity could be observed again. Thus, a thermally activated process occurs, which, after exhaustion of the photostimulation centres, apparently produces new ones. We have performed a series of similar experiments, i.e. x-irradiation at room temperature, a first and second photostimulation at 10 K with intermediate annealing and variation of the annealing temperature. Annealing up to about 280 K did not cause a replenishment effect. To observe it, we had to anneal above 280 K. This result differs from what was found for BaFBr:Eu²⁺ previously (Hangleiter *et al* 1991). There, a small replenishment was observed even for 200 K.

It was further observed that after exhausting the PSL at 300 K and waiting for a long time (for about 1 d), again a PSL effect could be observed. It is, however, only very small, below 1% of the PSL intensity observed during the first reading. Such an effect was also observed in BaFBr:Eu²⁺ (unpublished). For an explanation, we propose that after photostimulation at 300 K there are still F centres and hole centres that can aggregate by thermal activation at 300 K to form photostimulable centres, a feature we have denoted as the 'replenishment' effect above. We cannot, however, exclude that further unknown defects may play a role. For example, during the photostimulation electrons may be captured by other thermally unstable traps and then be thermally released and form photostimulable centres. However, we have not found experimental evidence for such traps.

After x-irradiation at 300 K, about 20–30% of the generated F centres decay within about 20–30 min.

3.2. X-irradiation at helium temperatures

The MCDA measured in a crystal x-irradiated at 4.2 K contained the MCDA bands of F centres, V_K and H centres, the latter two having been identified recently by Spaeth *et al* (1994b). No In⁰(1) centres, nor any In²⁺ centres could be found.

The number of F centres produced by the same x-ray irradiation dose is about half that found for room-temperature x-irradiation. Photostimulation into the F band produces also an In⁺ luminescence band at 2.9 eV and another one at 2.3 eV, which is from the self-trapped

excitons formed by recombination processes between the F centres and the intrinsic hole centres (Song and Williams 1993). Figure 5 shows the decay time of the PSL at 2.9 eV after x-irradiation under continuous excitation at 10 K. It is approximately the same as that after room-temperature x-irradiation when stimulating 10 K. The F centres bleach by about 50%. H centres and V_K centres vanish together with the F centres, both by about 50%. Upon annealing to 60–70 K, the H centres thermally decay; the number of F centres decreases to about one-third of the initial value. Upon further annealing to 200 K, the V_K centres also thermally decay, and there remains about 10% of the initial number of F centres.

At 4.2 K, clearly no In^{2+} centres are created by x-rays that would recombine with F centres to give the PSL emission at 2.9 eV. Only a very weak replenishment effect was observed after warming to 300 K. This effect is only $\sim 1/100$ that observed when x-irradiating at room temperature.

3.3. X-irradiation at 100 K

Upon x-irradiation for the same time as usual (2 h) at 100 K, F centres are generated, approximately as many as after x-irradiation at 4.2 K. The MCDA shows a strong band from F centres, a very weak one from V_K centres and around 3.5–4.0 eV two weak bands, which are paramagnetic, but no MCDA–EPR could be detected in those bands.

Upon photostimulation into the F band, two In^+ PSL bands appear: one at 2.9 eV and one at 2.4 eV (Fukuda 1970) (figure 4). The integral In^+ luminescence is about the same as that of the 2.9 eV luminescence measured at 10 K, thus the In^+ luminescence is the same for x-irradiation at 10 and 100 K (the precision of the luminescence intensity measurements is about $\pm 20\%$). The F centres did not bleach measurably during the photostimulation. After annealing to 300 K, the number of F centres decreased by approximately 20%.

4. Discussion

The failure to detect In^{2+} centres after x-irradiation at low temperature in spite of generating F centres demonstrates that In^{2+} –F centre pairs are not produced as primary radiation defects. The doped In^+ impurities do not act as primary hole traps. The observation of In^{2+} centres at room temperature, however, shows that the formation of In^{2+} centres is a thermally activated process. From a mobile hole centre of unknown nature, the In^+ must have captured the hole to form In^{2+} . The MCDA-detected EPR measurements of In^{2+} showed an isotropic spectrum (isotropic In hyperfine interaction and an isotropic g factor), suggesting that no other defect such as a nearest-neighbour vacancy is associated with the In^{2+} centre. Possibly, a V_K centre annihilates at the In^+ to form In^{2+} .

The observation that, after room-temperature x-irradiation and subsequent photostimulation at 10 K, the MCDA of the In^{2+} centres was not affected by the photostimulation shows that In^{2+} is not primarily involved in the photostimulated luminescence process. The In^+ luminescence is not a radiative decay of an F–electron– In^{2+} recombination process. Thus, we must conclude that the electron–hole recombination involves another hole centre. It is thought that, upon recombination of that hole centre with the photostimulated electron from the F centre, the recombination energy is transferred to a nearby In^+ centre, which is then excited and subsequently emits the characteristic In^+ luminescence. This would be very similar to what is thought to happen also in $BaFBr:Eu^{2+}$. There, upon x-irradiation, no Eu^{3+} is formed, and the PSL is thought to involve a triple aggregate between a hole centre, the Eu^{2+} activator and an F centre (Hangleiter *et al* 1990, 1991). The recombination between the excited F electron and the hole centre takes place via tunnelling within that

aggregate (de Leeuw *et al* 1987, v. Seggern *et al* 1988, Spaeth *et al* 1994a). It seems that also in KBr:In such a triple aggregate is formed. In BaFBr:Eu²⁺ the nature of the hole centre is not yet clear. It was speculated that the oxygen contamination (O_F⁻ centres) plays an important role. Here, there is no evidence for any oxygen contamination and, because of the careful growth process, there should not have been much oxygen incorporated. We have no information on the nature of the room-temperature-stable hole centre.

Support for the assumption of the formation of an aggregate as the responsible defect arrangement is the observation of the 'replenishment' effect. A very similar phenomenon was observed in BaFBr:Eu²⁺ (Hangleiter *et al* 1991). The fact that, after exhaustion of photostimulable aggregates at low temperature, new photostimulable aggregates are formed after warming above 280 K suggests that F centres and possibly also the unknown room-temperature-stable hole centres become mobile near that temperature and that the formation of the aggregate is energetically favoured over the statistical distribution.

That radiation defects are mobile at room temperature is not only seen from the replenishment effect but also from the observation that, after x-irradiation at room temperature, about 20–30% of the initially generated F centres decay within a few minutes.

It seems that there is a thermodynamical equilibrium distribution of triple aggregates between F and hole centres and In⁺, which is restored after its perturbation by a PSL process, provided the temperature is sufficiently high for the mobility of aggregate partners. At room temperature, the PSL process must occur until all photostimulable centres have aggregated and recombined.

The question arises of why there is almost no replenishment effect when x-irradiating at 4.2 K, and why it is very similar when irradiating at 100 K and 300 K. For an explanation it may be speculated that the hole centre is mobile at 100 K and above and forms already a pair aggregate with In⁺ during the x-irradiation. After warming to 280 K, the F centres become mobile and join the pair aggregate to form the triple aggregate. For irradiations at 4.2 K, none of the centres is mobile, so that only those triple aggregates that are directly formed contribute.

It was argued previously from the kinetics of the PSL that F–In²⁺ pairs are the responsible defects for the PSL effect, since the decay curves were almost the same whether the pairs were created by UV or x-irradiation (Braslavets *et al* 1992). Of course, the same decay time is also observed if the recombination involves tunnelling within the triple aggregate.

The low-temperature PSL experiments show that only a fraction of the F centres are involved in the PSL process. This is best seen for x-irradiation at 100 K, where there is not the complication with a recombination between F centres and other hole centres as at helium temperatures. This conclusion is supported by an interesting observation made recently by Tale *et al* (1993), comparing the optical absorption and photostimulation spectra at room temperature and 80 K. It was found that the photostimulation band does not coincide with the F centre absorption band. Its peak is shifted to lower photon energy. The shifted photostimulation spectrum was observed also for very low x-ray doses (a factor of 50 lower than usually used here). The photostimulation spectrum changes during the course of the read-out process. The low-energy part is measured first, then the photostimulation spectrum resembles more that of the F centres (Springis 1994).

For an explanation it may be speculated that the major PSL effect is due to F centres perturbed by the nearby presence of the In⁺ and unknown hole centre, which shows up in the peak energy of the photostimulation band. The observation does give further support for our speculation that the PSL effect is primarily due to triple-centre aggregates.

This observation of the spectral shift of the photostimulation band may provide a key for a better identification of the photostimulable centres. Possibly with optically detected

electron nuclear double resonance (ODENDOR), one can investigate the microscopic structure of the perturbed F centres that appear after x-irradiation. In addition, the yet unidentified MCDA bands between 2.5 and 3.5 eV need to be investigated further. They are probably due to hole centres. The prospect of identifying the centres involved in the PSL mechanism is probably better in this system (KBr:In) compared to BaFBr:Eu, because it is easier to remove oxygen completely and because of the cubic structure of KBr, which makes an MCDA-detected EPR and ENDOR investigation feasible. In BaFBr such measurements are only possible for the magnetic field orientation along the *c* axis, which does not allow the measurement of the angular dependencies of the EPR/ENDOR spectra. These are necessary for an analysis that may lead to the determination of the microscopic structures.

It may be noted that in preliminary experiments with the x-ray storage phosphor RbBr:Tl, the replenishment effect was also observed similarly as here (to be published).

In conclusion, we have shown that the x-ray energy storage and the read-out process do not involve primarily $\text{In}^{2+}\text{-F}$ centre pairs. The In^+ luminescence is due to a recombination between an unknown hole centre and F centres, whereby the recombination energy is transferred to the In^+ centres. It is thought that triple aggregates between In^+ , F and hole centres are the responsible configuration within which the PSL process happens.

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