

Thermoluminescence and structural characterization of new $\text{KCl}_x\text{Br}_{1-x}:\text{Eu}^{2+}$ sintered phosphors

R. Bernal and M. Barboza-Flores

*Centro de Investigación en Física de la Universidad de Sonora
Apdo. Postal 5-88, 83190 Hermosillo, Sonora, México.*

C. Cruz-Vázquez, K. R. Alday-Samaniego and F. Brown

*DIPM, Universidad de Sonora
Apdo. Postal 130, Hermosillo, Sonora, México*

Thermoluminescence properties of new europium doped $\text{KCl}_x\text{Br}_{1-x}:\text{Eu}^{2+}$ type phosphors were investigated as a function of the relative composition X. We show that these phosphors exhibit better thermoluminescence properties than those made of the alkali halides crystals with same composition, for the case of X and ultraviolet radiation. The characterized samples were made from a mixture of KCl, KBr and Eu_2O_3 high purity powders. The mixed powder were first pressed at an average pressure of 5.5 ton for 10 minutes, and then sintered five hours at 700 °C. In both cases, for X and UV radiation exposure of the samples, the thermoluminescence response depends strongly on the composition X, and a notorious enhancement of the thermoluminescence intensity is obtained around X=0.3. The samples exhibit promising properties as detectors and dosimeters for X and UV radiation.

Keywords: Phosphors; Thermoluminescence; Alkali halides

1. Introduction

Permanent monitoring of different types of ionizing radiation (dosimetry of ionizing radiation) is of great importance in scientific disciplines as well as in those that requires the use of radiations in a suitable and safe way, like in clinical applications and nuclear reactor facilities. Because the diversity of radiation sources, it is necessary to monitor different types and ranges of radiation, as well as a wide dose and energy interval. Thermoluminescence (TL) has demonstrated to be a trustworthy technique for dosimetry applications, and the alkali halides crystals exhibit remarkable properties as detectors and dosimeters. Recently, a lot of research has been focused on the luminescent, TL and dosimetry properties of the alkali halides crystals. Divalent europium doped alkali halides crystals present notable properties for the detection of ultraviolet light (UV) in the actinic region of the spectrum [1, 2], as well as for ionizing radiation of the type alpha, beta and gamma [3]. $\text{KCl}_x\text{Br}_{1-x}:\text{Eu}^{2+}$ mixed crystals have gained interest because they improved TL efficiency with respect simple ends components crystals, showing a maximum sensitivity for intermediate values of the relative composition X [3, 4].

The growth of crystals involves the use of expensive techniques that requires to take into account some special conditions if a good quality crystal is wanted. So it is important to investigate alternative easier and cheaper methods of making materials that present the noteworthy properties that alkali halides reveal. In this work, we report the process to manufacture and characterize materials of the $\text{KCl}_x\text{Br}_{1-x}:\text{Eu}^{2+}$ type, that can be used instead of bulk crystals of the same composition suitable for dosimetry purposes. This method is easier and cheaper than those commonly used to grow crystals. Moreover, its preliminary

characterizations indicate very promising properties to be used as detectors and dosimeters of UV and X radiation.

2. Experimental

Pellets of $\text{KCl}_x\text{Br}_{1-x}:\text{Eu}^{2+}$ were fabricated for various values of X, from a mixture of KCl (99.997 %), KBr (99.999 %) and Eu_2O_3 (99.9 %) high purity powders. The powders were carefully weighted in a balance Sartorius model R200D and then mixed by using an agate mortar. The mixed powder, containing 0.1 % of Eu_2O_3 per mol of $\text{KCl}_x\text{Br}_{1-x}$, was placed in a 7 mm diameter die, then pressed at an average pressure of 5.5 ton during 10 minutes using a Carver model C press to get pellets, that were subjected to a thermal process of 700 °C for 5 h using a Thermolyne model 48000 furnace. The pellets thickness measured around 1.09 mm (for X=0), and up to 0.81 mm (for X=1). The UV irradiation was performed with a 1000 W Oriol Hg-Xe high pressure arc lamp monochromated with a 0.25 m focal length Kratos monochromator, at room temperature and for 3 minutes of exposure. X irradiations were done using a Tel-X-Ometer type 580 M operated at 20 kV at room temperature avoiding the exposure of the samples to environmental light. All the TL measurements were performed under a N_2 atmosphere with a Harshaw 4000 TLD system using a linear heating rate of 5 °C s⁻¹. To perform structural characterization, scanning electron microscopy (SEM) images were obtained using a JEOL JSM-5410LV scanning electron microscope.

3. Results and discussion

Figure 1 shows the SEM images of a pellet of composition $\text{KCl}_{0.3}\text{Br}_{0.7}:\text{Eu}^{2+}$, before the thermal treatment. Some clear zones and remarkably dark zones can be

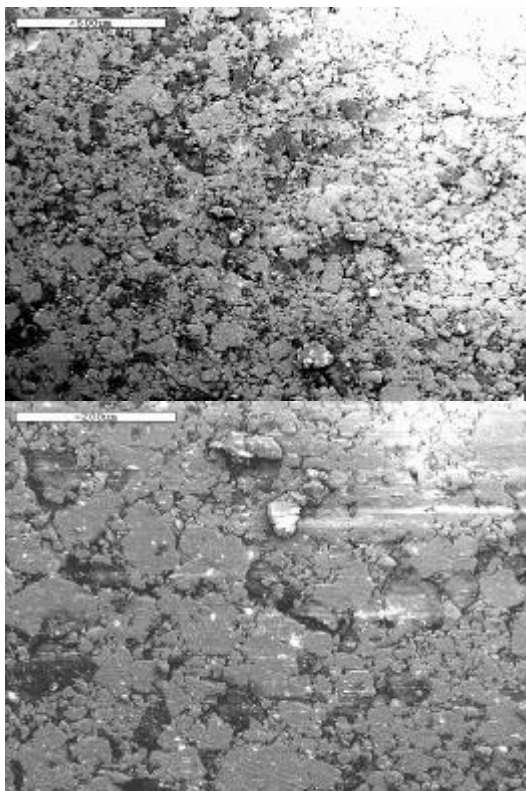


Figure 1. Scanning electron microscopy images of a pellet with composition $KCl_{0.3}Br_{0.7}:Eu^{2+}$, before being subjected to annealing.

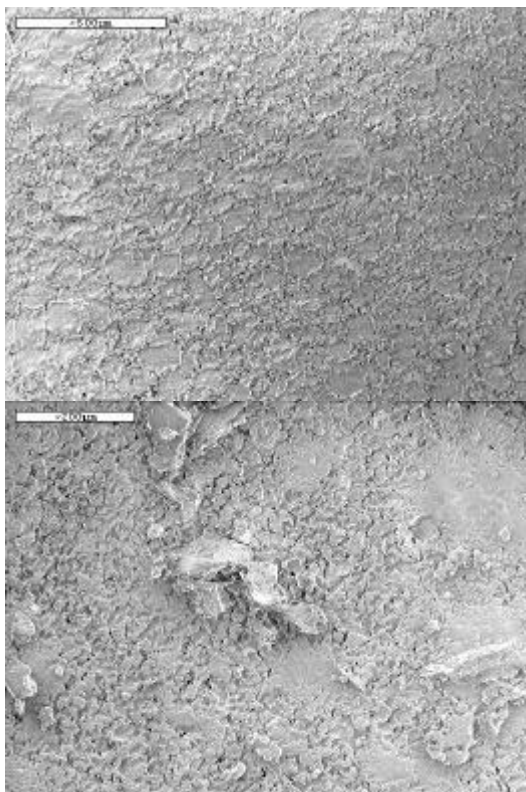


Figure 2. Scanning electron microscopy images of a pellet with composition $KCl_{0.3}Br_{0.7}:Eu^{2+}$, after being subjected to annealing.

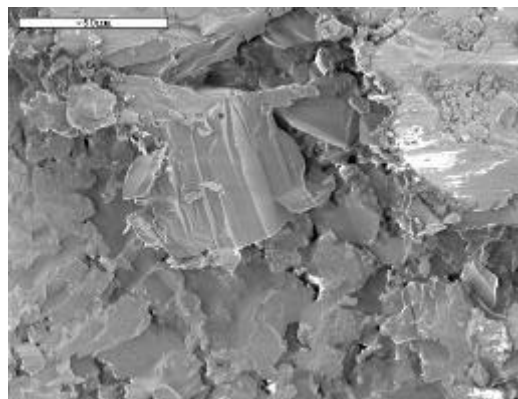


Figure 3. Scanning electron microscopy image of the same pellet as in figure 2

distinguished. The clearest zones correspond to the presence of KBr, whereas the darkest zones are due to the presence of KCl. In spite of the physical mixture of KCl and KBr salts, according to the SEM images, the salts of KCl and KBr coexists without losing its original phases. It is necessary to carry out the annealing of the sample in order to get a new mixed phase. The Eu_2O_3 appears as dispersed white centers. Figure 2 shows the SEM images of a pellet of the same composition after the sintering process of 700 °C during 5 h. As compared to figure 1, figure 2 instead clearly displays a new well homogeneous mixed phase. Figure 3 shows a crystalline $KCl_{0.3}Br_{0.7}:Eu^{2+}$ sample formed. The crystalline phase was confirmed to exist through X-ray diffraction data.

Figure 4 shows the thermograms of two $X=0.3$ composition pellets irradiated with X-rays for 1 min. The highest curve corresponds to a pellet sintered as described in the previous section, and the lower intensity curve is the TL emission curve corresponding to a pellet subjected to non-sintering process. The TL efficiency is greatly enhanced through the sintering process. Here, we recall the SEM images, and attribute the TL efficiency is related to the crystallinity of the samples. The 300 °C peak seems to be related to the new mixed crystalline phase, since it does not appears for the ends values of X, it arises only in sintered pellets.

Figure 5 shows the thermograms of sintered pellets with compositions $KCl_xBr_{1-x}:Eu^{2+}$, for $X=0.3, 0.5, 0.6$ and 0.85 , after being irradiated with UV light (235 nm wavelength) for 3 minutes. There is a strong dependency of the TL efficiency with respect to X. The curves seem to be composed of several peaks, showing in addition an optimal efficiency for $X=0.3$, decreasing for extreme values of X.

Figure 6 shows the thermograms of sintered pellets with compositions $KCl_xBr_{1-x}:Eu^{2+}$, for $X=0, 0.2, 0.3, 0.5$ and 0.7 after being X-irradiated for a minute. As in the case of UV irradiation, there is a strong dependence of the TL efficiency on the composition X. The whole shape of the thermograms seems to be composed of at least six peaks, showing a better TL emission for $X=0.3$, and decreasing TL efficiency for ends values.

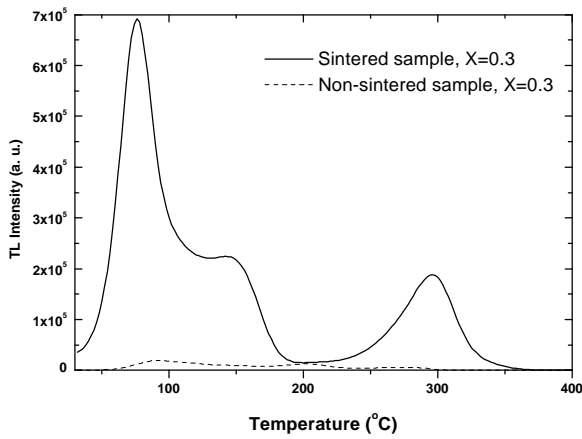


Figure 4. Thermoluminescence emission curve of a sintered pellet (solid line) and a non-sintered pellet (dashed line) after being irradiated with X rays for 1 min.

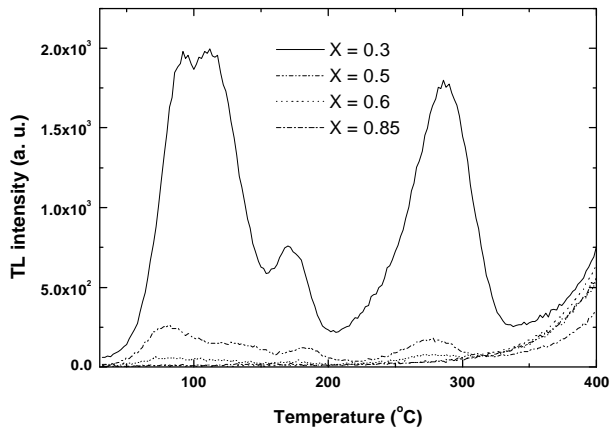


Figure 5. Thermoluminescence glow curves of $KCl_xBr_{1-x}:Eu^{2+}$ sintered pellets, for $X=0.3, 0.5, 0.6$ and 0.85 , after being irradiated with UV light (235 nm) for 3 minutes.

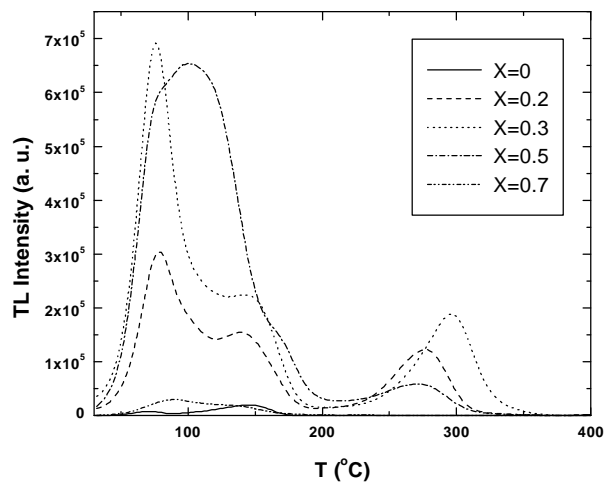


Figure 6. Thermoluminescence glow curves of X-irradiated $KCl_xBr_{1-x}:Eu^{2+}$ sintered pellets, for $X=0, 0.2, 0.3, 0.5$ and 0.7 .

4. Conclusions

We have provided some evidence to show that sintered phosphor materials exhibit promising features as detectors and dosimeters for UV and X radiation. These can be fabricated by a cheap and simple method. These materials exhibit improved TL features as compared to those bulk crystalline samples with similar composition. An enhanced TL efficiency is obtained for composition $KCl_{0.3}Br_{0.7}:Eu^{2+}$ ($X=0.3$) for both, UV and X irradiation.

Acknowledgements

This work is supported by CONACyT (México), Grants J35222-E and 32069-E, Universidad de Sonora Grant P100/29, FOMES, and DGICSA-SEP (México). The authors thank Japan International Cooperation Agency for donating the scanning electron microscope.

References

- [1] H. Nanto, K. Murayama, T. Usuda, F. Endo, Y. Hirai S. Taniguchi and N. Takeuchi, *J. Appl. Phys.* **74**, 1445 (1993).
- [2] I. Aguirre de Cárcer, F. Cussó and F. Jaque, *Phys. Rev. B* **38**, 10812 (1998).
- [3] B. Castañeda, R. Aceves, T. M. Píters, M. Barboza-Flores, R. Meléndrez and R. Pérez-Salas, *Appl. Phys. Lett.* **69**, 1388 (1996).
- [4] M. Barboza-Flores, R. Meléndrez, B. Castañeda, T. M. Píters, R. Pérez-Salas, R. Aceves, J. A. Muñoz and I. Aguirre de Cárcer, *Radiat. Meas.* **29**, 487 (1998).