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ON THE REPRODUCIBILITY OF THERMOLUMINESCENT PROPERTIES IN X-RAY IRRADIATED KCl

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Résumé. — Les monocristaux de KCl de haute pureté ( exempts d'ions OH ) ont été irradiés par des rayons X par une série de la même petite dose à 80 K. Le changement d'intensité du pic principal à 125 K a été mesuré entre 80 et 300 K pendant des cycles constitus d'une irradiation et d'un chauffage. L'intensité commence par augmenter et atteint ensuite une valeur limite. Par comparaison avec des résultats antérieurs, on peut supposer que les centres F (et leurs agrégats) sont la cause de l'accroissement de l'intensité de thermoluminescence.

Abstract. — Extremely pure KCl single crystals (free from OH- ions) were irradiated with X-rays repeatedly by the same small dose at 80 K. The change of the main glow peak intensity at 125 K was measured in the temperature range 80-300 K during heating cycles consisting of an irradiation and heating out process. It was found that the main peak intensity first increases rapidly then smooths and at a higher number of cycles reaches a saturation value. Comparing these results to previous observations made by other authors, it may be supposed that the F centres (and their aggregates) are responsible mainly for the special feature of the thermoluminescent intensity increase.

1. Introduction. — Irradiation of alkali halides produces colour centers (mostly F centers), and the concentration of these centres can be measured easily by optical methods if the concentration is high enough. Since the thermoluminescent process is sensitive to smaller concentrations when the radiation recombination yield is equal to unity, the thermoluminescent method may provide information on crystal defects, concerning a low concentration of effective traps. This is the case when the irradiation doses are very low. In the present paper an attempt made to improve the thermoluminescent method. Thermoluminescence (TL) was used to investigate an early stage coloration of KCl at liquid nitrogen temperature.

2. Experimental procedure. — The samples of zone-refined KCl single crystals were kindly supplied by Voszka [1]. Their purity was very high, the divalent impurity concentration was $2 \times 10^{-8}$ mol/mol and they were free of OH- ions. The crystals were always freshly cleaved into samples of dimension about $0.8 \times 0.8 \times 0.07$ cm$^3$ and used without annealing. The effective sample area was 28 mm$^2$. The TL light emitted by the sample was detected by an EMI 9558 QA photomultiplier tube, cooled down to $-18^\circ$C by Peltier elements (Type EREPA, Alcatel) to reduce the dark current. The samples were irradiated at 80 K with X-rays by using a Liliput 120 type X-ray equipment giving 5 mA at 100 kV$_{ph}$. (A 2 mm thick aluminium filter was used.) Temperature was measured by an iron-constantan thermocouple. The heating was nearly linear with a heating rate of 0.6 K.s$^{-1}$. The dose rate at the sample was $2.2$ R.min$^{-1}$. The energy absorbed by the crystal was $4.6 \times 10^{15}$ eV.cm$^{-3}$ for an exposure time of one minute. The vacuum in the cryostat was better than $10^{-5}$ torr. The TL glow curves were registered between 80 and 300 K by means of the generally used method.

3. Results. — The TL peak at 125 K was the dominant glow peak in the sample as shown in figure 1. The measurements were carried out strictly under identical experimental conditions. The thermal history of the sample during the experimental cycles is shown in figure 2. According to the investigation of the reproducibility of the glow curves found in KCl it

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**Fig. 1.** — Thermoluminescent glow curve of KCl zone-refined by Voszka after 15 cycles and an exposure time of 10 minutes X-ray irradiation.
was observed, that the main glow peak intensity changes, i.e., first it increases rapidly, then smoothes, and at a higher number of cycles reaches a saturation value (Fig. 3). It must, however, be mentioned that these experiments were carried out with the same X-ray exposure times. This is also valid for the other glow peaks but the growth rates for these higher temperature peaks are much smaller than the peak at 125 K. The experiments were performed on a set of crystals of KCl from different sources (Voszka, ISOMET Co., and the Institute of Physics, Bucharest). The early stage coloration was studied with another type of experimental series. In this experiments the sample was irradiated by increasing doses and the coloration curve is shown in figure 4.

4. Discussion. — Halperin et al. [2] reported that after many TL cycles on the same crystal a change of intensity could be observed. Some of the glow peaks of annealed samples increased enormously. Jain and Mehendru [3] also mentioned this phenomenon. They used the TL method to clarify that F centers play a key role in the TL processes. Ausin and Alvarez-Rivas [4] also observed a strong correlation between the TL glow curves intensity and the F center annealing processes. In the present investigation the glow peak intensity changes were studied systematically. The evaluation was based on the data given by the manufacturer (EMI), and from combining with the experimental and geometrical conditions used follows that the total number of photons emitted during the thermoluminescence from the sample at the 125 K peak is between $1.1 \times 10^{11} - 208 \times 10^{11}$ photons cm$^{-3}$. It should be mentioned that the short X-ray irradiations had no detectable effect on the absorption spectra. The conclusions based on the measurements are as follows:

1) The results found were identical with those obtained in F center coloration studies. As it could be seen in figure 4, at liquid nitrogen temperature we detected a very short «first stage» coloration which has not been observed clearly like this by other authors, because their methods were sensitive only at higher F center concentrations. It is probable that the «late stage» coloration is identical with that received by absorption measurements on pure crystals.

2) The estimated values based on the knowledge of the absorbed energy were $1.74 \times 10^3$ eV/TL photon for the «first stage» and $11 \times 10^3$ eV/TL photon (the Ritz’s result [5] on KCl at LHeT is 2400 eV/F center) for the «second stage», by dividing the total energy absorbed from the X-ray beam by the total number of TL photons. These values are characteristic for the processes, and seem to be realistic.

3) The small depression of our coloration curve is thermally activated as well as radiation triggered back reactions [6] are operative in the first stage.

Finally, it can also be seen that the comparison of the classical glow curves of the X-ray irradiated KCl single crystals is possible only by using virgin samples of the same dimensions and under the same experimental conditions since reproducibility can only be realized in this way.

We hope that the TL method may be a possible means of investigating the early stage coloration processes at low temperature in the near future.
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References


DISCUSSION

W. A. SIBLEY. — In temperature cycling experiments it is sometimes possible to deform a sample due to the difference in the expansion coefficients between the sample and the bonding material or holder. It is possible that with your pure KCl (which is normally very soft, $\tau \approx 30$ g/mm$^2$) you produce dislocations by the cycling treatment?

J. FÉLISZERFALVI. — Yes, it is, but these new dislocations are relatively much lower in number than those present originally in a high purity crystal of KCl.

W. A. SIBLEY. — Did you measure the wavelength of the emitted light?

J. FÉLISZERFALVI. — We did not measure the wavelength of the emitted light. Our calculations were based on the data given by Sonder, Sibley, Mallard in Phys. Rev. (1967) and Ausín, Alvarez-Rivas in reference [4], concerning the wavelength of the emitted light from lightly irradiated KCl.

A. A. BRANER. — Similar results obtained a long time ago in our laboratory, were then interpreted as being due to diffusion of oxygen into the crystal during heating cycles. A KCl crystal heat-treated in air or poor vacuum showed this increase of the 150 K TL peak. This increase could be reversed by heating the crystal in our inert oxygen-free atmosphere. Pellets made from crystal powder behaved as aged crystals from the very beginning.

(See works by Braner, Kristianpoller, Schlesinger and Halperin in the years 1956-1962.)