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SIMULTANEOUS DETECTION OF THERMOSTIMULATED LUMINESCENCE AND EXOELECTRONIC EMISSION BETWEEN 77 AND 650 K: APPLICATION TO ALPHA ALUMINA

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Abstract — Detection apparatus which allows the simultaneous recording of TL and TSEE curves up to 650 K after X or UV irradiations at 77 K is described. Various samples of α-alumina were studied in these conditions. It is shown that this approach avoids some measurement errors and compares accurately the TL and TSEE peaks related to the same trapping centre.

INTRODUCTION

TSEE and TL measurements presented by various authors[1–7] show that the position of thermostimulated peaks exhibited by α-alumina are in good accord. This correlation allows us to interpret the exoelectronic emission mechanisms by a continuity of bulk defects towards the surface of this material. In order to develop a good experimental approach to various thermostimulated mechanisms of α-alumina, simultaneous measurements are required. They permit avoidance of some measurement errors and accurate comparison of the TL and TSEE peaks related to the same trapping centre. Some authors have described such experimental devices but the temperature range studied[1,2], the method adopted[5] or the lack of technical data forbids the use of their methods.

Here, the simultaneous detection apparatus that has been developed is described. It allows the simultaneous recording of TL and TSEE curves up to 650 K after X or UV irradiation at 77 K with various heating rates. Different α-alumina samples are studied. Thanks to a physical model of thermostimulated phenomena, these investigations lead to an estimation of 0.12 eV for the value of the effective electron affinity presented by an α-alumina sample.

EXPERIMENTAL SET-UP

The experimental set-up presented here has been obtained from the TSEE apparatus previously described[7]. The detection device and the data processing unit have been modified.

Silica and beryllium windows located on the ultra-high vacuum (UHV) chamber (5×10^{-6} Pa) permit irradiation of the sample by UV light or X rays, respectively.

Temperature controller

The sample holder has already been described[7]. Cooling is achieved by drawing up liquid nitrogen through the sample holder. Heating is achieved by a resistance element supplied by an electronically programmed device directly controlled by a microcomputer (Figure 1). The modulation of heating power is provided by a pulse width modulation signal (PWM) which drives a solid state relay followed by a step-down transformer. The PWM signal is calculated and generated every second by the microcomputer thanks to a like-PID regulation algorithm complemented by an open-loop action (static + first order dynamic).

A temperature limiting system and a 'watchdog' device are used to ensure the safety of heating equipment. Thus, the heating law is linear and its rate can be adjusted. The value adopted for this study is 20 $K.min^{-1}$.

Detection device

The exoelectrons are detected by a channel electron multiplier (CEM) operating in the pulse counting mode whereas the photons are led to a photomultiplier tube (PMT) thanks to optic fibre bundles followed by a focusing lens. The chosen silica optical fibres are UHV-resistant and have a good UV transmission.

In order to eliminate the infrared emission produced by the heating holder, appropriate optical filters are used.

After they have been recovered and shaped, the PMT and CEM information — transformed into TTL signals — is driven to a pulse counter and recorded by the microcomputer simultaneously with the sample temperature (Figure 2).

EXPERIMENTAL CONDITIONS

The TL and TSEE intensities depend on various parameters such as the nature of the sample, the type, energy and kerma of the radiation, and the heating rate. When these parameters are fixed, the number of photons and the number of electrons emitted per unit time at the maximum of TL and TSEE peaks related at a particular trapping centre are constant. A good simultaneous

measurement is obtained when these quantities are, at the same time, neither too weak nor too high in such a way that the detected signals are sufficiently important but not saturated. In the case of very disproportionate responses, some of these parameters can be adjusted so as to reduce this disequilibrium.

Choice of sample

In this study, results are presented obtained from three kinds of alumina samples: a single Baikowski crystal, a powdered Desmarquest sample and crushed α-Al_2O_3:C[6].

The choice of these samples was forced by a previous study[7] in which it was shown that the first two samples present TL and TSEE responses of same magnitude for a given air kerma whereas the as-received α-Al_2O_3:C single crystal is very thermoluminescent and very weakly exoemissive.

Generally, the single crystals, which have a relatively weak specific surface, present a TSEE phenomenon weaker than the TL one, which is essentially a bulk phenomenon. This effect can be counterbalanced by an appropriate grinding which augments the specific surface value and results in an intensification of TSEE. It is for this reason that the α-Al_2O_3:C sample was crushed.

The Desmarquest alumina is a commercial powdered alumina. It is prepared by electrofusion and its granularity is between 5 and 20 μm. The Baikowski single crystal is a disc of 8 mm in diameter and 0.8 mm thick synthesised by the Verneuil method. The α-Al_2O_3:C sample is a single crystal which has been crushed for this study. It has been obtained in very reducing conditions[6].

Experimental parameters

The number of photons and the number of electrons emitted in unit time can be indiscriminately changed by variation of the heating rate, q, and by modification of the air kerma of X irradiation.

The use of a convenient optical filter allows attenuation of the TL intensity without modification of the TSEE response. In order to lessen the infrared radiation, an interferential filter has been interposed between the sample and the PMT, centered on the emission of F (410 nm) or F^+ (320 nm) centres of α-Al_2O_3.

Often, when the samples are powders, it is the TSEE signal which must be attenuated. In this case, a grid maintained at a weakly negative potential (Wehnelt) can be placed in front of the CEM cathode so as to reduce the number of detected exoelectrons. In order not to alter the TL detection, this possibility has not been

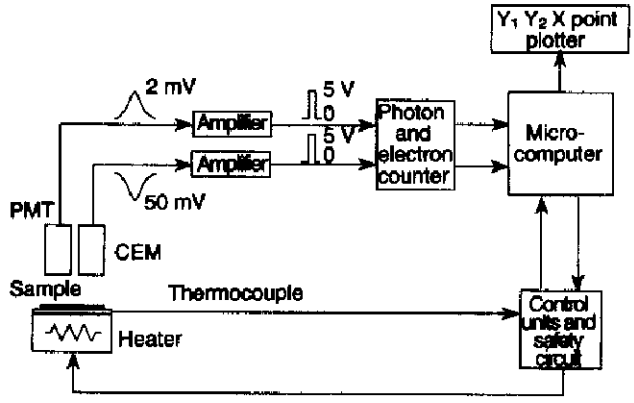

Figure 2. Diagram of detection device.

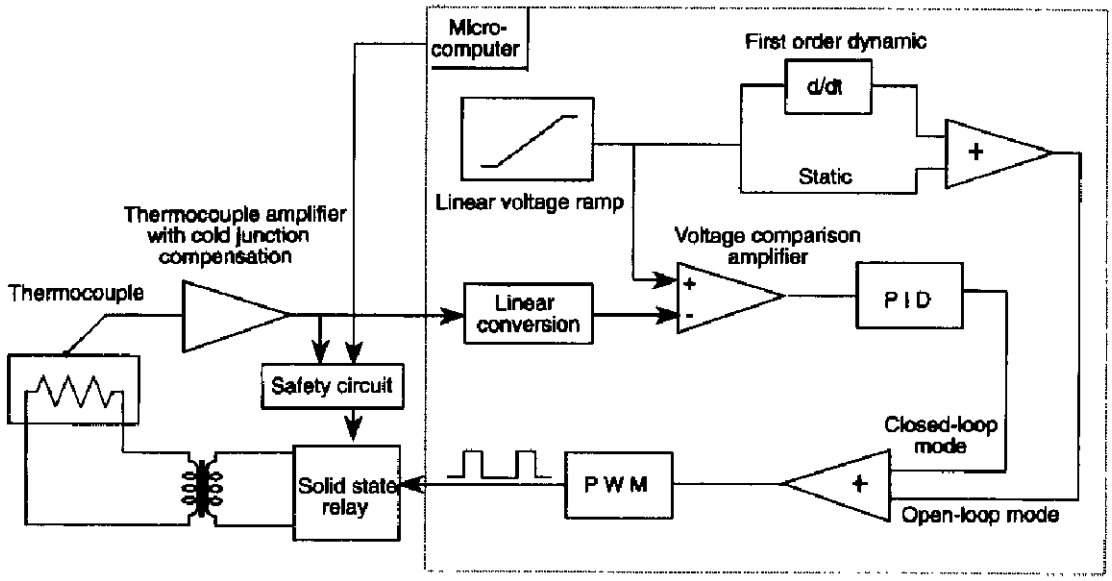

Figure 1. Diagram of temperature controller.

explored but the potential of the CEM cathode has been changed.

EXPERIMENTAL RESULTS

Simultaneous TL and TSEE curves of the three samples

The results obtained with the three samples, after X irradiation at 77 K and heating at $q = 20\ K.min^{-1}$, are reported on Figures 3–5. TL is registered through a 410 nm interferential filter which corresponds to the emission of F centres. In these conditions, the temperature difference, ΔT, between the TSEE and TL peaks B observed with the Baikowski sample is near to zero. For the peaks situated above peak B, ΔT is all the greater as the temperature of the involved peaks is high (Table 1).

If the TL curves are recorded through a 320 nm filter (emission of F^+ centres), an enhancement is observed of the intensities of TL peaks B and D′ and a disappearance of TL peak B′ (Figure 6). Nevertheless, the ΔT values remain unchanged.

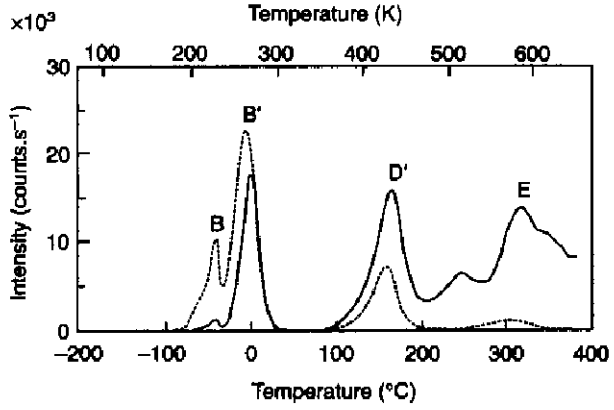

Figure 3. TL and TSEE curves of Baikowski single crystal sample after X irradiation at 77 K (50 kV, 5.1 Gy); filter 410 nm; $q = 20\ K.min^{-1}$. (—) TSEE, (----) TL (×10).

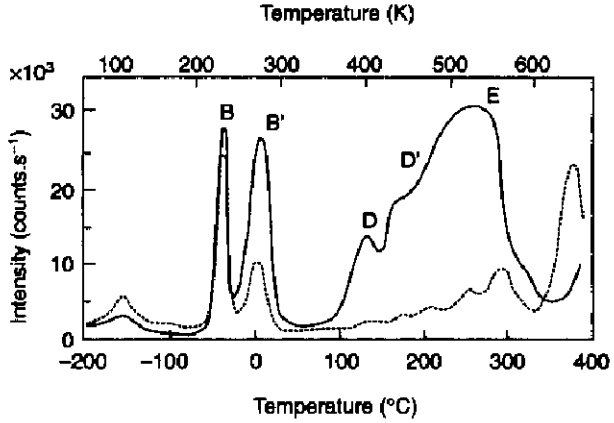

Figure 4. TL and TSEE curves of Desmarquest powdered sample after X irradiation at 77 K (50 kV, 5.1 Gy); filter 410 nm; $q = 20\ K.min^{-1}$. (—) TSEE, (----) TL (×100).

Heating rate and air kerma effects

A variation of the air kerma from 0.34 to 10.2 Gy does not notably modify the ΔT values observed with the Baikowski sample (410 nm filter). On the other hand, the ΔT difference relative to peak B′ increases from 3 to 12 K as a function of air kerma. Additionally, no significant variation is observed for the values of ΔT when the heating rate is changed from 15 to 30 $K.min^{-1}$.

DISCUSSION AND CONCLUSION

The various models used to interpret the thermostimulated phenomena show that there is a temperature difference between the homologous peaks of TL and TSEE.

One of the experimental approaches intended to test if a proposed model is fully justified consists in comparing the theoretical ΔT differences with the experimental ones. In order to obtain a more accurate evaluation of the ΔT differences, the latter must be measured on one apparatus only able to detect the TL and the TSEE

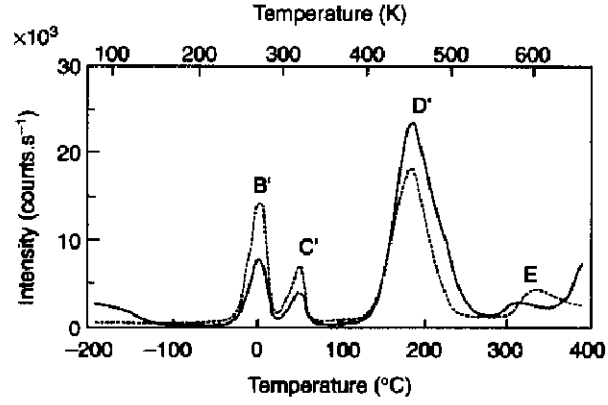

Figure 5. TL and TSEE curves of α-Al_2O_3:C crushed sample after X irradiation at 77 K (50 kV, 0.085 Gy); $q = 20\ K.min^{-1}$. (—) TSEE, (----) TL (×30).

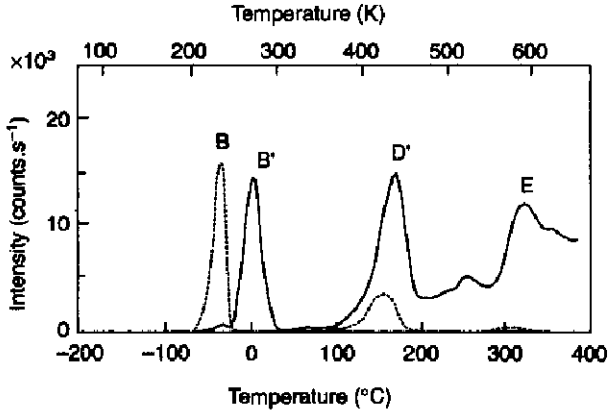

Figure 6. TL (----) and TSEE (—) curves of Baikowski single crystal sample after X irradiation at 77 K (50 kV, 5.1 Gy); filter 320 nm; $q = 20\ K.min^{-1}$.

phenomena simultaneously. In such a way, the values obtained for ΔT are not altered by the thermal gradient which exists between the real sample temperature and the temperature of the thermocouple junction. Nevertheless, the problem related to the thermal gradient which exists between the heated side of the sample and the TSEE emitting side remains. In fact, since the TSEE is a surface phenomenon whereas TL affects the bulk of sample, we expect to observe a ΔT difference all the greater as the thickness of the sample is large. This is the reason for the important ΔT values observed for the Baikowski sample (Table 1), which is thicker than the powdered or crushed sample layer. Furthermore, the surface state which can produce an energy band bending and modify the temperature of TSEE peaks is not taken into account.

For all these reasons, the ΔT values are not thought to be suitable for evaluating the electronic affinity of the alumina and a method based on the form of thermostimulated curves is used for preference.

In a previous study[8] it has been shown that the application of the thermoionic model described by Holzapfel[9] to TSEE peak B′ (270 K) permits us to obtain an estimation of the effective electron affinity of alumina: $\chi = 0.12$ eV.

It is not certain whether the thermoionic model fully describes all the other TSEE peaks of alumina. In fact, the thermostimulated peaks at 60 K[10], 100 K[7] and 230 K[4,7] have been interpreted by the use of a model involving the Auger effect.

Table 1. Experimental $\Delta T = T_{TSEE} - T_{TL}$ differences observed for some thermostimulated peaks of alumina, filter 410 nm.

Peak	Mean temperature	Sample	Irradiation	ΔT (K)
B	230 K	Baikowski	X (50 kV, 5.1 Gy)	0 ± 1
		α-Al_2O_3:C		—
		Desmarquest	X (50 kV, 5.1 Gy)	0 ± 1
B′	280 K	Baikowski	X (50 kV, 5.1 Gy)	6 ± 2
		α-Al_2O_3:C	X (50 kV, 0.085 Gy)	0 ± 1
		Desmarquest	X (50 kV, 5.1 Gy)	3 ± 2
C′	325 K	Baikowski		—
		α-Al_2O_3:C	X (50 kV, 0.085 Gy)	0 ± 1
		Desmarquest		—
D′	440 K	Baikowski	X (50 kV, 5.1 Gy)	8 ± 2
		α-Al_2O_3:C	X (50 kV, 0.085 Gy)	4 ± 2
		Desmarquest		—

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