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THERMOLUMINESCENT RESPONSE OF TLD-100 TO LOW ENERGY ELECTRONS (5 keV to 30 keV)
AND THE EFFECT OF ANNEALING IN VARIOUS ATMOSPHERES

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Abstract

Previous investigations of the thermoluminescent response of the dosimetry phosphor LiF (TLD-100) to low energy electrons have given varied estimates of its sensitivity. In addition, several investigators have noted that the glow curve shape of TLD-100 irradiated with low energy electrons differs from that obtained after irradiation with ^{137}Cs gamma rays or high energy electrons. The work reported here, for single crystal TLD-100, yields the following main conclusions:

(1) If annealings are done carefully in vacuum, then the sensitivity of TLD-100 single crystals to 5-30 keV electrons and the resultant glow curve is essentially the same as when irradiations are carried out with ^{137}Cs gamma rays.

(2) All discrepancies in sensitivity can be accounted for by the higher Linear Energy-Transfer (LET) of the electrons.

(3) The "standard annealing" of 400°C for one hour commonly used, produced a change in the glow curve shape and a loss in sensitivity in contrast to the vacuum anneal results. Diffusion of hydroxyl ions into the sample during air annealing was isolated as the primary cause for this change.

(4) These results also explain the cause for the existence of the 0.4 micron "dead layer" which previous investigators have proposed to explain the variation with particle size of the luminescent efficiency of TLD-100 powder irradiated with x-rays.

The glow curve obtained from virgin TLD-100 extruded ribbon samples varied, but in general lay between the glow curve obtained from single crystal samples annealed in vacuum and single crystal samples annealed in air.

With the use of the annealing procedure presented here TLD-100 may immediately be applied to the dosimetry of low energy electrons and other shallowly penetrating radiation. With this proposed annealing procedure the same sensitivity and reproducibility can be achieved as is currently achieved for the dosimetry of x-rays.

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Thermoluminescent Response of TLD-100 to Low Energy Electron (5 keV to 30 keV)
and the Effect of Annealing in Various Atmospheres

J.B. Lasky and P.R. Moran

1. INTRODUCTION

The measurement of dose absorbed from low energy electrons is of widespread practical interest. Health physicists, for example, attempt environmental monitoring of tritium, and the electron microscopist uses electron probes in elemental microanalysis of biological tissue. Previous investigations of the thermoluminescent response of the dosimetry phosphor LiF (TLD-100) to low energy electrons have given varied estimates of its sensitivity (Kasner et al., 1967, Barber et al., 1973.) In addition, Kastner has noted that the glow curve shape of TLD-100 irradiated with low energy electrons differs from that obtained after irradiation with ^{137}Cs gamma rays or high energy electrons. The work reported here, however, yields the following main conclusions for experiments done with single crystals:

- (1) If annealings are done carefully in vacuum, then the sensitivity of TLD-100 single crystals to 5-30 keV electrons and the resultant glow curve is essentially the same as when irradiations are carried out with ^{137}Cs gamma rays.
 - (2) All discrepancies in sensitivity can be accounted for by the higher Linear-Energy-Transfer (LET) of the electrons.
 - (3) The "standard annealing," as suggested by Cameron and Harshaw Chemical Co. and commonly used in previous experiments, is carried out in air at 400° for one hour. This standard anneal, in contrast to the vacuum anneal, produced a change in the glow curve shape and a loss in sensitivity. Diffusion of hydroxyl ions into the sample during air annealing was isolated as the primary cause for this change.
 - (4) These results also explain the existence of the 0.4 micron "dead layer" which previous investigators have proposed to explain the variation with particle size of the luminescent efficiency of TLD-100 powder irradiated with x-rays.
- The glow curve obtained from virgin TLD-100 extruded ribbon samples varied but in general lay between the glow curve obtained from single crystal samples annealed in vacuum and samples annealed in air.

2. EXPERIMENTAL

Samples measuring $1/8" \times 1/8" \times .040"$ were cleaved from a large single crystal of TLD-100 purchased from the Harshaw Chemical Co. (Cleveland, Ohio) in 1975. TLD-100 extruded ribbon samples purchased from Harshaw Chemical Co. were also used. Virgin samples were read out once and then irradiated. Single crystal samples were annealed for one hour at 400°C in a vacuum of 5×10^{-6} Torr and allowed to cool in the vacuum to room temperature. This vacuum annealing was crucial to obtaining the results to be described since it minimized the diffusion of impurities into the sample. Another batch of single crystal and extruded ribbons were annealed in air at 400°C to duplicate the usual laboratory procedure.

Electron irradiations were carried out in an evacuated pyrex chamber. A vacuum of 10^{-5} Torr was sufficient to prevent ionic currents. The source of electrons is a tungsten filament whose potential can be varied from 0 to minus 30 keV. A stainless steel paddle may be rotated between the filament and irradiation port to stop irradiations. The electrons pass through an irradiation port consisting of two aligned 1 mm holes in stainless steel discs to strike the sample (see Fig. 1.) The "Source-side" disc is grounded and the "Sample-side" disc is at minus 45 volts. The samples rest in shallow wells drilled in a rotatable stainless steel holder. These wells may be precisely positioned under the irradiation port. The sample holder is insulated electrically from the chamber and returned to ground through a Keithley 616 electrometer; this measures the total charge striking the sample. A fine tungsten mesh was spot welded over the irradiation port in the sample-side disc to minimize perturbation by this port of the electric field near the crystal. Geometrically, all electrons which pass through the irradiation port strike the sample. Secondary electrons produced at the sample are repelled by the sample-side stainless steel disc and are collected by the sample holder (Kazan et al., 1968.) All secondary electrons produced at the top of the source-side disc are repelled by the sample-side disc and do not reach the collecting sample holder (See Fig. 1)

The possibility that the dielectric sample would charge while being irradiated was considered. The secondary emission coefficient is greater than 1 for incident electrons with energies greater than 22 eV. (Bruining, 1954.) Samples should thus reach electrical equilibrium with the most positive electric potential that they sense, in this case the rotatable sample holder which is grounded through the electrometer (Kazan et al., 1968.) The secondary emission coefficient is usually measured in ultra-high-vacuum on vacuum-cleaved crystals or vacuum-evaporated samples. To verify that the secondary emission coefficient was greater

than one for the samples used here, the potential on the sample-side disc and cylindrical housing was changed to +45 V to collect all secondary electrons produced at the sample. The current measured by the electrometer attached to the sample holder was positive for all beam energies between 100 eV and 30 keV indicating that the secondary emission coefficient was greater than one. As a final check that the beam was not changing shape after passing through the irradiation port the thermoluminescence image produced by crystals irradiated with 30 and 10 keV electrons was photographed and the size of the luminescent spot measured. Its size was the same as the irradiation port to within 20%, the accuracy of this method.

The volume used to calculate the electron dose in rads (100 ergs/gm.) delivered to the sample, was the product of the irradiation port area and the depth of penetration of the electrons. This depth was found by scaling Kobitch's data for the range of electrons in Al to the relative stopping power of LiF (Berger, 1966.) Cameron's (1968) value for the density of LiF (2.64 gms./cm.³) was used to convert the volume irradiated to grams. The energy deposited in the sample was the product of the total charge which struck the sample, recorded by the Electrometer attached to the sample holder, and the acceleration voltage of these electrons. This voltage was checked with a Keithley model 6103B Divider Probe attached to a Keithley 616 Digital Electrometer. The same Electrometer was used to integrate the charge collected by the sample holder. The Electrometer had been calibrated by Keithley Instruments before the experiment and its accuracy in both models was 5%.

Samples were read out in a Harshaw 2000 A reader which uses a photomultiplier with a S-11 response. A helium gas flush was added to the TL reader sample chamber. This improved reproducibility of glow peak positions by increasing thermal contact of the heater with the sample. The helium flush also inhibited chemoluminescence and diffusion of impurities into samples while they were read out. A linear heating rate of 70°C/sec. was used. The glow curves were recorded on a x-y recorder and simultaneously a Norland 2001 programable calculating oscilloscope. The latter instrument carried out numerical time integration of selected portions of the glow curve. In general the area under peaks 3-5 (Cameron et al., 1968, p. 39,) the main dosimetry peaks, is reported as the measure of TL response. To eliminate errors caused by apparent wandering peak positions in time-temperature, due to varying thermal contact between samples and the heater pan, the area under peaks 3-5 was evaluated by taking a cut of fixed time width keyed about peak 5.

3. RESULTS

A. Single crystals annealed in vacuum

Single crystals, annealed in vacuum were given 10³ rads of 5, 10 and 30 keV electrons. The resulting glow curve shapes are similar to those obtained when the samples are irradiated with cesium γ -rays. This is illustrated in Fig. 2. It should be remembered that cesium emits .662 MeV gamma rays. These penetrate the 1 mm sample with little attenuation and deposit their energy via Compton-recoil electrons, uniformly throughout the sample. The recommended buildup of 2.5 mm Lucite (Cameron 1968, p. 27) required for electronic equilibrium was used for all γ -ray irradiations. Low energy electrons, on the other hand, deposit their energy near the surface. The ranges of 5, 10, 20, and 30 keV electrons are .31, .90, 2.8 and 5.8 microns respectively. Fig. 3 shows the crystal's response to 5, 10, and 30 keV electrons as a function of dose delivered to the irradiated volume. This response bears many resemblances to the dose-response characteristics obtained when irradiations are carried out with cesium γ -rays. The response exhibits supralinearity above 10³R and saturates at 10⁵R. The supralinearity and sensitivity for electron irradiations are observed to decrease with decreasing electron energy. The response to cesium γ -rays was measured at 1 R and found to be 1.29×10^2 in the units used in Fig. 3. It maintains this value until the onset of supralinearity at 10³R (Cameron et al., 1968, p. 156)

B. Single crystals annealed in air

Figures 4 and 5 show the glow curve obtained from single crystal samples annealed in air at 400°C for one and twenty-four hours, rapidly cooled to room temperature and irradiated to 10³ rads of 5, 10, 20 and 30 keV electrons. The height of peaks 3-5 are reduced while the height of peak 2 is unchanged. This change takes place first near the surface (5 and 10 keV response) and moved to progressively greater depths (response to 20 and 30 keV electrons) with further annealing. After 24 hours annealing, the crystals response to electrons (i.e., within 10 μ of the surface) was about one third its initial response. The response to γ -rays (i.e. bulk response) remained unchanged. Since no changes occurred in the glow curve when samples were annealed in vacuum for similar periods, it is clear the change is caused by impurities diffusing into the

sample.

C. Single crystals annealed in H_2O atmosphere.

DeWerd and Stoebe (1971) have reported glow curve changes similar to those described in section 3B above for the γ -ray response of TLD-100 diffused with hydroxyl ions. To verify that hydroxyl ions were responsible for the altered response to electrons, freshly cleaved samples were sealed in a thoroughly cleaned, evacuated quartz tube along with ice made from deionized water and heated at $700^\circ C$ for four hours. The results are shown in Fig. 6. The glow curve changes produced are similar to the changes produced by annealing in air and to DeWerd's results for hydroxyl ions diffused into the bulk of TLD-100.

D. TLD-100 extruded ribbons.

Harshaw Chemical Company has heated loose powder of TLD-100 and extruded it through special dies to form "ribbons", 7 mm by 7 mm by 0.8 mm. This form of TLD-100 is used for routine dosimetry of γ -rays since the response uniformity of a batch of these extruded samples to γ -rays is very good, about 4%. However, the TL response of virgin TLD-100 extruded ribbon to electrons varied from sample to sample. In general the glow curves showed some attenuation of peak five. This was more pronounced when lower energy electrons (5 and 10 keV) were used. Annealing in air (Fig. 7 and 8) produced changes similar to those observed in single crystals but these changes occurred more rapidly in the extruded ribbons. This is probably because extruded ribbons have a more porous structure than single crystals.

4. DISCUSSION

A. Single crystals annealed in vacuum.

The Linear-Energy-Transfer (LET) of charged particles is the amount of energy locally imparted to the medium (dE) in an elemental distance along the particle's track (dL), and can be expressed as the quotient of dE/dL. For γ -rays the charged particle is the primary Compton-recoil electron. The Dose Averaged LET stated in Table 1, column (2), was obtained by scaling Cole's (1969) expression for the LET of electrons in plastic as a function of the electron's energy to the relative stopping power of LiF and integrating this expression with respect to energy from the electron's incident energy to

zero. This weights the LET of the electron at each energy by the amount of energy it loses at that LET. Table 1 shows the relative sensitivity of TLD-100 to radiations with different Dose-Averaged LET; column 4 lists the sensitivity observed in the present work and column 5 that observed by Wingate et. al. (1967).

The LET of a radiation influences its TL efficiency by determining the distance between electron-hole pairs it produces. According to Attix's "Track Interaction Model" the energy lost to the TL process through spontaneous recombination of electron-hole pairs will increase as the distance decreases. Although the "Track Interaction Model" is a semi-quantitative model and does not predict exact values for the extent of this effect, a calculation using the LET stated in Table 1 indicated that for a 30 keV electron there are 2 electron-hole pairs along the electron's track for every TL trap. Thus the LET of 30 keV and lower energy electrons is expected to be an important factor, a "dead layer" of the surface of LiF which absorbs energy but does not contribute to the TL process, has been postulated by several investigators to explain the low efficiencies they observed for shallowly penetrating particles to produce TL. This is discussed in more detail in the next section.

Wingate measured the relative sensitivity of TLD-100 as a function of LET using 30 - 910 MeV alpha particles whose range is two orders of magnitude greater than the ranges of electrons with comparable LET. The excellent agreement between the sensitivity measured here and the values measured by Wingate shows that the 20% loss in sensitivity of 5 keV electrons is strictly an LET effect and not due to a surface dead layer. A dead layer of $400^\circ A$ would have resulted in a loss of sensitivity of 10% in addition to the loss due to the LET effect. Detection of such a loss was within the sensitivity of the system used but was not observed.

The supralinearity factor is the ratio of the maximum TL per rad to the value at low rad levels, where the response varies linearly with dose. Figure 9 shows the decrease in the supralinearity factor with increasing LET observed by Suntharalingam (1969) using cesium γ -rays and 400-50 keV electrons and for 30-5 keV electrons in the present work. Extrapolation of the present work indicates there should be no supralinearity for radiation with LET greater than 25 keV/micron.

B. Single crystals and extruded ribbon annealed in air.

The observed loss of sensitivity for TLD-100 annealed in air explains the low sensitivities reported in the literature. It also explains why the dead layer hypothesized to explain the loss of sensitivity (Kastner et al., 1967; Zanelli, 1968) can be attributed to the presence of hydroxide ions introduced by air annealing. Kastner gave TLD-100 the standard annealing and found the efficiency of tritium β -rays to produce TL was 0.05 the efficiency of cesium γ -rays. He noted that peak 2 was unusually large compared to the higher temperature peaks and its persistence varied from batch to batch. Since tritium emits betas with 18 keV maximum and 5.7 keV average energy its efficiency to produce TL would be decreased by the standard air annealing. Powdered samples were not used in the present work, so it is not possible to give quantitative estimate of the effect of annealing on powders. However, the present results showed the effect was greater on the more porous extruded ribbon than for the single crystal material and, consequently, the effect on powder is expected to be even greater.

Barber et al. (1973) found the sensitivity of standard air annealed extruded ribbons exposed to 4 keV electrons was 0.4 of its sensitivity to cesium γ -rays. The relative sensitivity found here for 5 keV electrons in vacuum annealed single crystals is 0.80. The sensitivity suppression resulting from annealing in air can account for this difference.

Zanelli (1968) postulated the existence of a 0.4 micron "dead layer" in TLD-100 powder to explain its variation of luminescent efficiency with particle size after irradiation with x-rays. He also noted that the height of peak 5 decreased to a third of its original height when the particle size was reduced from 25 μ to 2.5 μ , while the height of peak 2 remained unchanged. These experimental results are in excellent qualitative agreement with the current findings, and can be attributed to the presence of hydroxide ions in the first micron. Again, only semi-quantitative comparison with the present data is possible since the present experiments were carried out only for crystals and extrusions. Moreover, Zanelli did not describe details of his annealing procedure. However, as shown in Fig. 4, the standard annealing results in a 25% decrease in TL efficiency for the first 3 microns. This would have a similar net effect on sensitivity as Zanelli's 0.4 micron "dead layer." He also noted a change in relative peak heights which makes it virtually certain that diffusion of hydroxyl ions also were responsible for his observed phenomena.

5. CONCLUSION

The excellent agreement between the TL from the first few microns of freshly cleaved single crystals and the glow curve from the bulk of these samples show that there is no intrinsic difference, as far as TL is concerned, between the surface of the crystal and the bulk. The change in glow curve shape and lower sensitivity reported by others for electron irradiations can be attributed to the samples receiving an anneal in air prior to irradiating. Diffusion of air-borne hydroxyl ions into the surface is the major impurity responsible for these air-anneal induced changes. The results of this work show that TLD-100 can be used for dosimetry of low energy electrons and that the same sensitivity and reproducibility can be achieved as is normally achieved for x-ray dosimetry, provided that annealing in air is avoided.

The observed, unaltered, glow curve shape at 5 keV leads one to wonder if changes in glow curve shape will be observed for even more shallowly penetrating, lower energy radiation. The Thermally Stimulated Exoelectron (TSEE) glow curve obtained from TLD-100 does not have the same shape as the corresponding TL glow curve (Zimmerman, 1971.) The TSEE signal electrons emanate from the first 200 $\text{\AA}$ of the sample (Holzapfel, 1969,) and the TSEE shape looks much like the TL glow curve obtained from air annealed samples irradiated with low energy electrons. Zimmerman cites damage of the surface as a possible cause for the lack of correspondence of the two glow curves. If it is found that the vacuum-annealed TL glow curve produced by radiation which deposits energy in the first 200 $\text{\AA}$ is unaltered from the bulk glow curve, then it seems probable that the diffusion of hydroxyl ions into the surface also is responsible for the altered TSEE glow curve. This line of reasoning and the experiments it dictates are explored in another report, (COO-1105-239.)

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Figures

- Fig. 1 Irradiation chamber.
- Fig. 2 Glow curves from TLD-100 single crystals exposed to 10^3 rads 5, 10 and 30 keV electrons and 1 rad cesium γ -rays.
- Fig. 3 Integrated TL of peaks 3-5 per rad-gram (arbitrary units) as a function of dose delivered to the irradiated volume. Error bars are not shown. The uncertainty in the TL measurement was $\pm 15\%$ for the first decade of data taken and $\pm 7\%$ above that rad level.
- Fig. 4 Response of TLD-100 single crystals annealed in air for 1 hour at 400°C to 5, 10, 20 and 30 keV electrons. Scales are multiplied by indicated factors.
- Fig. 5 Response of TLD-100 single crystals annealed in air for 24 hours at 400°C to 5, 10, 20 and 30 keV electrons. Scales are multiplied by indicated factors.
- Fig. 6 Response of TLD-100 single crystals diffused with OH at 700°C for 4 hours to 5, 10 and 30 keV electrons. Scales are multiplied by indicated factors.
- Fig. 7 Response of TLD-100 extruded ribbons annealed in air for one hour at 400°C . Scales are multiplied by indicated factors.
- Fig. 8 Response of TLD-100 extruded ribbons annealed in air for 60 hours at 400°C . Scales are multiplied by indicated factors.
- Fig. 9 Supralinearity of TLD-100 single crystals irradiated with cesium γ -rays and 400 to 5 keV electrons as a function of the LET of this radiation. The data for cesium γ -rays and 400-50 keV electrons is from Suntharlingham (1968).

Table

- Table 1 Supralinearity observed here and by Suntharlingham (1968) and response of peaks 3-5 observed here and by Wingate (1967) for single crystal TLD-100 irradiated with cesium γ -rays and 400-5 keV electrons.

Fig. 2

Fig. 1

T.L. (ARBITRARY UNITS)

Fig. 4

Fig. 3

Fig. 5

Fig. 6

Fig. 7

Fig. 8

Fig. 9

(1) Type of Radiation	(2) Dose Averaged L.E.T. (Kev./μ)	(3) Supralinearity Factor	(4) Relative Response (Observed Here)	(5) Relative Response (Wingate, 1967)
Cs^{137} x-rays (.662 Mev)	0.5	3.0	1	1
400 Kev. electrons	1.1	3.0		1
275 Kev. electrons	1.4	2.9		1
175 Kev. electrons	2.0	2.5		1
100 Kev. electrons	2.9	2.3		1
50 Kev. electrons	4.6	2.2		.96
30 Kev. electrons	6.4	2.0	.95	.93
10 Kev. electrons	12.7	1.5	.88	.84
5 Kev. electrons	18.7	1.2	.80	.77

Table 1