Ancient TL

Isolation of the rapidly bleaching peak in quartz TL glow curves

A. D. Franklin and W. F. Hornyak
Physics Department, University of Maryland, College Park, Maryland 20742, USA.

Introduction

Natural archaeological and geological quartz, without additional laboratory doses, often exhibits two major TL peaks: a rapidly bleaching peak (RBP) at about 325 °C, for a ramp rate of 20 °C/s. (Fleming, 1979; Spooner et al, 1988). In the work reported here the ramp rate was considerably lower at 1°C/s. This difference in ramp rate is accompanied by a downward shift in TL peak temperatures, so that in our data the RBP occurs at about 275 °C and the SBP at 310-330 °C, depending on the optical filter used. Expected plateau ranges are likewise shifted downward. The RBP is currently the basis for work on optically stimulated luminescence dating (Huntley et al., 1985; Smith et al., 1986; Godfrey-Smith et al., 1988; Rhodes, 1988).

To make possible direct study of the RBP we have devised a method of isolating this peak by combining use of optical filters during measurement with a subtraction technique, in which a TL glow curve taken after apparently complete removal of RBP by optical bleaching is subtracted from the corresponding glow curve obtained before bleaching to yield the RBP by difference.

To obtain a pure quartz, sand was extracted from samples drawn from the relic dunes at Dobe - toa in the northwest Kalahari desert of southern Africa (Helgren and Brooks, 1983; Helgren, 1982), part of a site of considerable archaeological interest. The sediment was treated at room temperature with 3N HCl to remove carbonates, 48% HF followed by 3N HCl to remove alkali feldspars and fluorides, and H_2O_2 to remove organics. The 90-150 μm fractions was selected by sieving, and then density separated to isolate the quartz component with density <2.70 g/cm^3. The resulting freely-flowing powder was light buff in color and was composed of rounded transparent grains, most of which were shiny and clear. The x-ray powder diffraction revealed only excellent α-quartz patterns.

The RBP has in the past for the most part been observed using broad banded blue (e.g. Corning 5-60) or violet (e.g. Corning 7-59) optical filters in conjunction with a PM tube such as the EMI 9635Q. In order to separate the RBP and SBP as much as possible we have examined the spectral output of our quartz after laboratory irradiation, across the near u.v. and visible regions. In keeping with data in the literature (Schlesinger, 1964; Medlin, 1968; Zimmerman, 1971; Fuller and Levy, 1977, 1978; David et. al., 1977) we find emission peaks centered around 490 nm with glow peaks at 190 and 330 °C and perhaps at higher temperatures, using a ramp rate of 1°C/s. In the wavelength region below about 400 nm major glow

peaks occur at 70, 180, 275 and 310 °C, and minor peaks are seen at ~125 and ~240 °C.

The peak at 275 °C, strongly emitting below 400 nm, is the RBP. It cannot be completely isolated by the use of filters but it can be maximized relative to the SBP by using a uv filter such as the UG11. Spooner et. al. (1988) showed that RBP can be rapidly and completely bleached with long wavelength (e.g. green ~2.3 eV) light with essentially no effect on the SBP. Thus (fig. 1) the RBP can be obtained by subtraction of the glow curve after the green-light bleach from the glow curve with no bleach.

A subtraction technique demands the utmost in precision, with respect to both TL intensity and temperature. Weight-normalized glow curves obtained with the sample essentially as described earlier (Franklin et al., 1987) are averaged over up to 16 runs. A temperature marker is placed on each by exposing the sample to a very small standardized beta-dose, the ZG dose, using a Sr-90 source prior to the run but after a brief (three minute total, including 1 °C/s rise time) preheat to and hold at 150 °C. The purpose of the preheat is to remove low-temperature TL induced in artificially irradiated samples. The ZG dose generates an easily measurable peak at 70 °C which is used to normalize temperatures.

The resulting averaged glow curves still contain uncertainty in TL intensity and in temperature. The uncertainty in TL of the unbleached curve relative to the bleached curve can be reduced by normalizing the former to the latter using the ratio of intensities of the average 70 °C peaks. Some further adjustment along the temperature axis is necessary because the use of the Au/Ni plate Cu bowl as a specimen holder introduces a small variable effect on the rate of the rise of the temperature of the sample. This adjustment was made in present data by maximizing the coincidence of the unbleached and bleached glow curves on the high temperature side of the SBP. The results of these procedures are data such as illustrated in figure 1 for laboratory-irradiated samples.

First Glow Growth Curve

RBP glow curves for laboratory doses from 0 to 300 Gy added to the natural dose are shown in figure 2. Experimental standard deviations for the TL value range from 6% to 13%. The curve for 300 Gy is apparently saturated, higher laboratory doses up to 1 kGy yielding essentially the same curve. The first glow growth curve for the temperature at the glow curve peak (275 °C) is shown in figure 3a. Because if the apparent sudden

onset of saturation at 300 Gy no attempt has been made to fit with a saturating exponential equation. These data can be used to obtain an estimate of the intercept on the dose axis. A simple unweighted linear squares fit to the first four data points produced a value for the intercept of 40 Gy. The 1σ confidence interval (treating the four fitted data points in figure 3a as single values) is shown as a small heavy line on the abscissa at the intercept. The intercepts, from several such glow growth curves, produce a reasonable plateau from 240 to 290 °C, shown in figure 3b. This figure suggests a mean intercept of 42 ± 1 Gy.

Conclusion

By use of an ultraviolet band pass filter, careful TL measurements, and appropriate data manipulation, the rapidly bleaching TL peak in quartz for doses in the range 0 - ~200 Gy can be isolated and studied independently of the nearby slowly bleaching peak. For a sample of the average radioactivity discussed by Aitken (1985, p. 68), this suggests and upper limit to the age determination of about 70 ka. However many samples exhibit much less radioactivity and the age limit could be much higher, limited eventually by the lifetime of the RBP, estimated at ~10^8 years (Aitken, 1985, p. 272).

Figure 1.
TL glow curves for Kalahari dune sediment, 90-150 μm quartz fraction given 100 Gy Co-60 gamma dose: a) without light exposure; b) with 4 hour exposure to solar simulator with Chance-Pilkington 26-3350 green filter; c) difference curve.
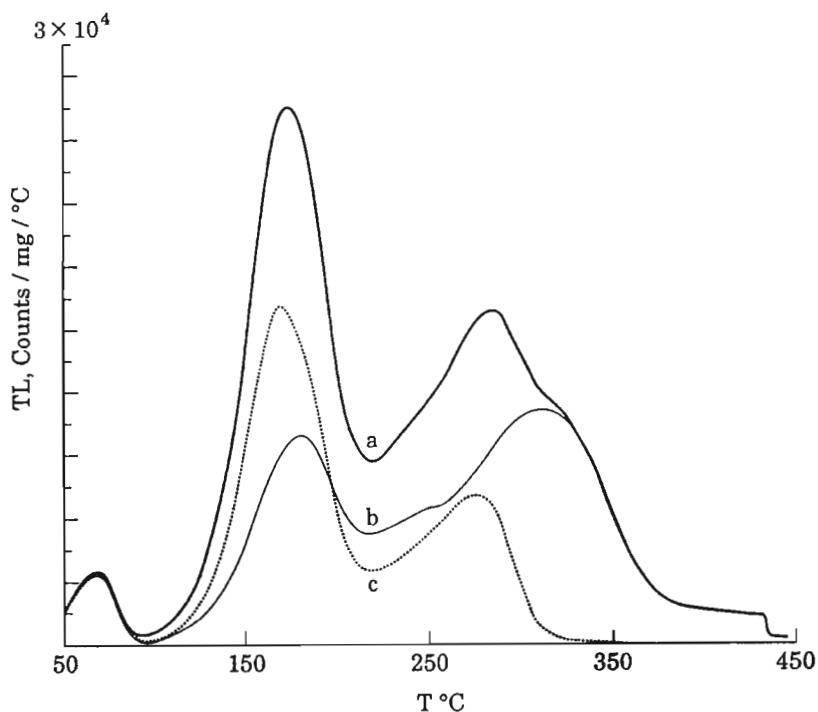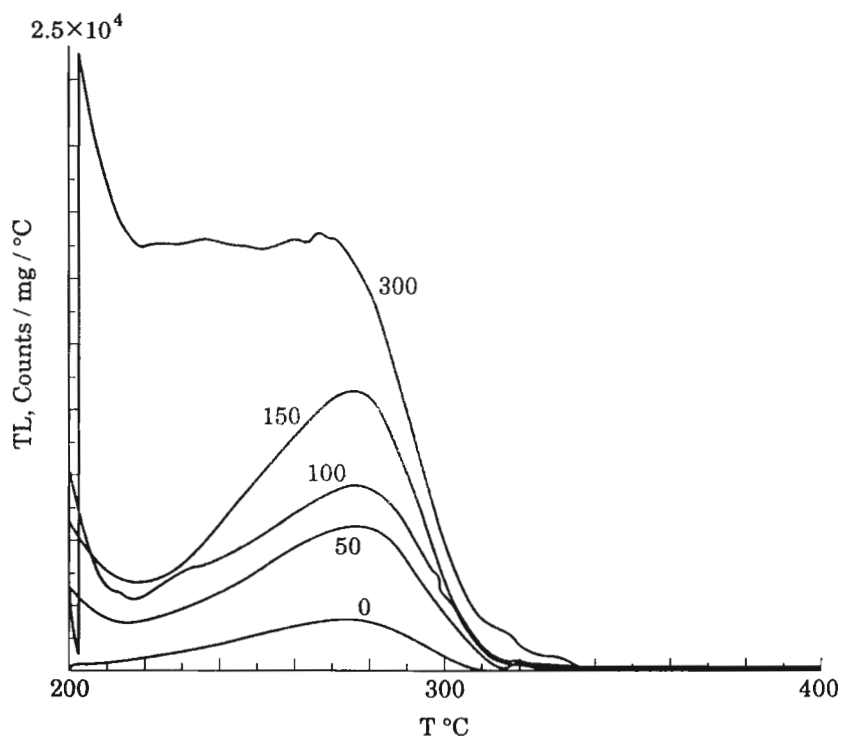

Figure 2.
Rapidly Bleaching Peak (RBP) TL glow curves for 90 - 150 μm quartz fraction of Kalahari dune sediment given Co-60 gamma doses shown (in Gy).
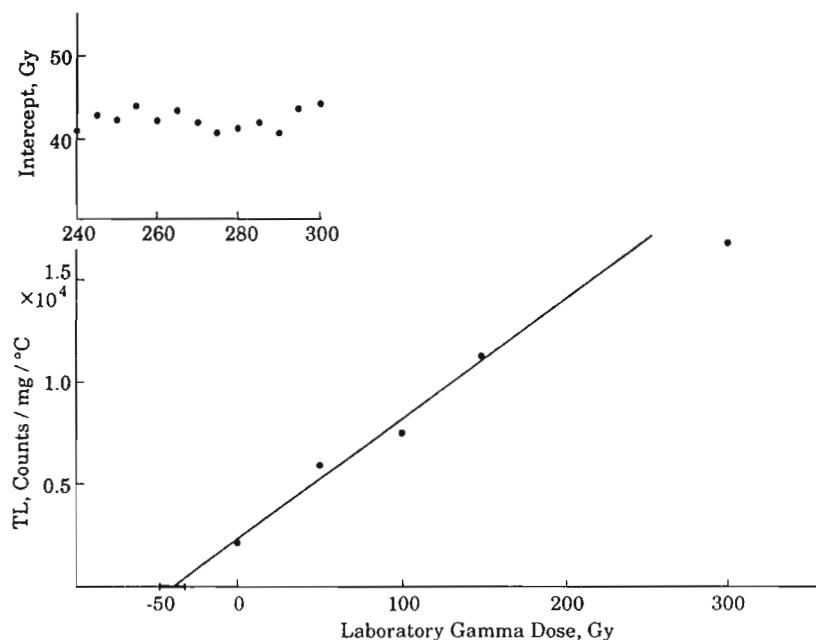
Figure 3.
First flow growth data from Rapidly Bleaching Peak (RBP) for 90 - 150 µm quartz fraction of Kalahari dune sediment: a) first glow growth curve using glow curve peak values (275 °C). Heavy line at intercept represents 1σ confidence interval; b) intercept at several temperatures.

References
Aitken, M. J. (1985) *Thermoluminescence dating,* Academic Press, London.

David, M., Sunta, C.M., and Ganguly, K. (1977) Thermoluminescence of quartz: Part1 - glow curve and spectral characteristics, *Ind. J. Pure and Appl. Phys.,* **15,** 201-4.

Fleming, S.J. (1979) *Thermoluminescence techniques in archaeology,* Clarendon Press, p46.

Franklin, A.D., Hornyak, W.F., and Tschirgi, A. (1987) Experimental TL Techniques for the inclusion method, *Ancient TL.,* **5,** 9-10.

Fuller, E.G. and Levy, P. (1978) Thermoluminescence of natural quartz, *Bull. Am. Phys. Soc.,* **23,** 324.

Godfrey-Smith, D.I., Huntley, D.J., and Chen, W.H. (1988), Optical dating studies of quartz and feldspar sediment extracts, *Quaternary Science Reviews,* **7,** 373-380.

Helgren, D.M. (1982) Edaphic context of the mongongo *(Ricinodendron rautanenii)* in the Northwestern Kalahari. *South African J. of Science,* **78,** 131-2.

Helgren, D.M., and Brooks, A.S. (1983) Geoarchaeology at Gi, a middle stone age and later stone age site in the Northwest Kalahari. *J. Archaeological Sciences,* **10m,** 181-197.

Huntley, D.J., Godfrey-Smith, D.E., and Thewalt, M.L.W. (1985) Optical Dating of Sediments, *Nature,* **313,** 105-7.

Medlin, W.L. (1968) Thermoluminescence in Aluminium-containing Quartz. *Physics Letters,* **10,** 49-50.

Rhodes, E.J., (1988), Methodological Considerations in the optical dating of quartz. *Quaternary Science Reviews,* **7,** 395-400.

Schlesinger, M. (1964) Thermoluminescence in quartz. *J. Chem. Phys.,* **38** , 1132-43.

Smith, B.W., Aitken, M.J., Rhodes, E.J., Robinson, P.D., and Geldard, D.M. (1986) Optical dating: Methodological aspects. *Radiation Protection Dosimetry,* **17,** 229-33.

Spooner, N.A., Prescott, J.R. and Hutton, J.T. (1988) The effect of illumination wavelength on the bleaching of the thermoluminescence (TL) of quartz. *Quaternary Science Reviews,* **7,** 325-9.

Zimmerman, J. (1971) The radiation-induced, increase of the 100 °C thermoluminescence sensitivity of fired quartz. *J. Phys. C. Solid State Physics,* **4,** 3265-76.

PR Helen Rendell