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TL Behaviour of Some Limestone Rocks

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Introduction

As part of a study of several limestone rocks from an archaeological site (Ganj Dareh, Iran) dating back 8-10 ka, we used an unorthodox method of preparing fine grains for TL analyses. The method was chosen to minimize the time required to produce a few hundred mg of sample. The purpose of this account is to describe our sample preparation, and the TL behaviour of different grain sizes for two of these rocks as observed through two spectral emission windows - the yellow-green (~ 550 nm), and the blue (< 500 nm).

The TL of calcite (our samples were largely pure calcite, as verified by X-ray diffraction and chemical analyses) has some attractive characteristics from the point of view of dating. In particular, there are usually two distinct peaks, both of which are normally free from detectable anomalous fading, and both of which are probably thermally stable at $< 15^\circ\text{C}$ for $\geq 10^6$ years (e.g. Wintle 1978, Debenham 1983). However, difficulties have been encountered with stalagmitic and flowstone calcites because of non-uniformity of TL sensitivity and of radioactivity (e.g. Wintle 1978, Walton and Debenham 1982). Furthermore, calcite is especially susceptible to spurious TL attributed to mechanical and thermal disturbances during cutting and crushing. This spurious signal can overwhelm the high temperature peak and seriously affect the other. To avoid this spurious TL, workers have resorted to the slice technique, with its attendant disadvantages (e.g. Aitken and Wintle 1977), or the use of large (~ 100 μm) grains prepared by crushing in a vice (e.g. Wintle 1978). The use of fine grains (2-10 μm , say) has been avoided because for them spurious TL apparently has been more difficult to control. Wintle (1974) did use 1-8 μm grains for soft limestones with apparent success, but this has not been followed up.

Notwithstanding problems with inhomogeneity (which might be expected to be less for some types of massive limestone than for travertines), we think the use of fine grains of calcite offers some advantages over the use of coarse grains. In particular, it is relatively easy to prepare many uniform subsamples.

Sample Preparation

All steps were performed in dull red or in amber light. Our samples were hard, most having a Mohs index of 3-4 1/2, and one (not discussed here) had an index of 5 1/2 (it also contained ~ 2-3% of α - SiO_2). We used a water-cooled, high-speed, 10-inch carborundum blade to cut away the outer 2 mm of each rock. Each sample was hand-held against the cutting edge and a slight pressure was applied, just sufficient to overcome "chattering". Following this cutting, approximately 200 μm was removed from the surfaces by hand lapping with a wet 400-grade emery paper. Then each piece was placed in 1N HCl for 1-3 minutes and rinsed in distilled water.

Crushing was done in a way to minimize the number of impacts received by individual small grains, as follows: a cleaned piece of limestone was placed in a steel percussion mortar, a single sharp blow was given, and the resulting debris was dry sieved at 1 mm; only fragments > 1mm were returned to the mortar, and the crushing and sieving was thus repeated until a few grams of < 1 mm material were obtained. The < 1 mm grains produced in this way were wet sieved at 250 μm and 88 μm . The 88-250 μm grains then were soaked in 0.5% acetic acid for 1 minute (Wintle 1975) and washed in distilled water. The < 88 μm grains were sized at 11 μm by Stokes settling in water and at 2 μm by timed centrifugation. The resulting 2-11 μm grains were washed in 0.5% acetic acid for ~ 20 seconds and recovered by centrifugation, washed in water and then methanol.

For each sample, about 60 aluminum discs, each holding ~ 1 mg of 1-11 μm grains, were prepared from methanol suspensions, with ~ 5 minutes of ultra-sonic agitation just after pipetting. For each of three samples about 25 discs of 88-250 μm grains were prepared by first wiping each disc with a tissue "wetted" with a drop or two of silicone oil, weighing, sprinkling the grains from a small spatula, and weighing again. In this way ~ 5-10 mg of material were deposited fairly evenly onto each disc.

This method of sample preparation yielded very reproducible TL signals with low or insignificant levels of spurious TL in both the 2-11 μm and 88-250 μm size fractions. The essentially identical TL behaviour for the large and small grains confirmed that our preparation procedures did not significantly disturb the natural TL. In what follows, we describe the TL behaviour of only two samples, 060 and 061L, which were examined in most detail.

TL Behaviour at Yellow-Green Wavelengths

Since some calcites have their most intense emission around 550 nm (e.g. Medlin 1968, Debenham et al. 1982), we chose an interference

filter with a transmission peak at 550 nm and a "window" of ~ 100 nm width (Spectracoat Monopass, No. 546 of set 246, Optics Technology Inc., Belmont, Calif.). This was used together with our usual infrared rejection filter. All samples were glowd using the low-pressure flow of Ar and hot gas-purifier described elsewhere (Berger et al. 1982).

The additive-dose glow curves for the 2-11 μm grains of sample 061L are shown in Figure 1. Here it is seen that the dominant peak is

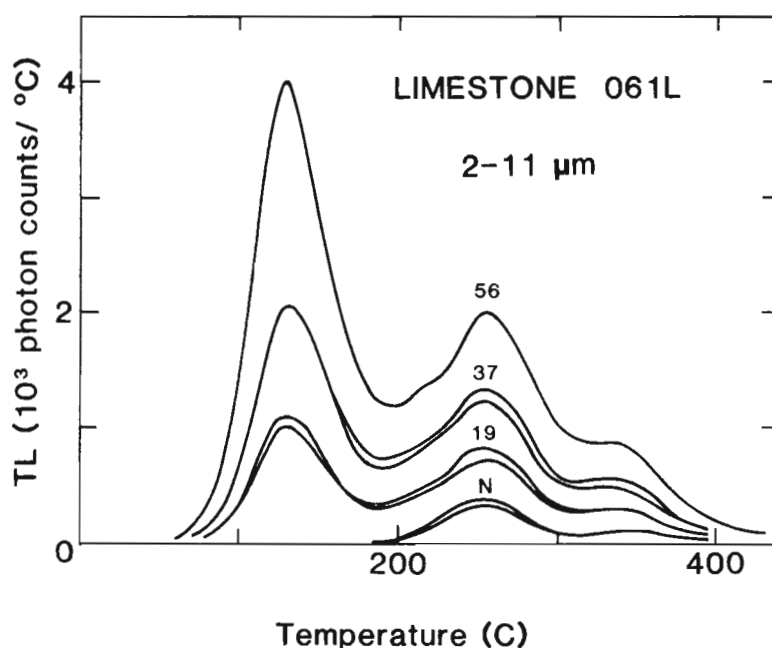


Fig. 1 Smoothed glow curves for the 2-11 μm fraction of sample 061L observed at the yellow-green (~ 550 nm) wavelengths (filter 546). There are 2 curves at each dose (in Grays) except for the unirradiated or natural (N) where there are 3. Reheats have been subtracted. The heating rate was $5^{\circ}\text{C}/\text{sec}$. See Wintle and Huntley (1980) for equipment descriptions.

at 260°C with a lower signal at higher temperatures. The ratio of natural TL peak heights is ~ 3.1 ($260/330^{\circ}\text{C}$). Similar sets of glow curves were obtained for the 2-11 μm and 88-250 μm fractions of sample 060, in which the ratio of peak heights was $\sim 4:1$.

We were surprised to observe prominent supralinearity in the growth of TL at both 260°C and 330°C for samples 060 and 061L. An example is shown in Figure 2 for 061L. This behaviour is reminiscent

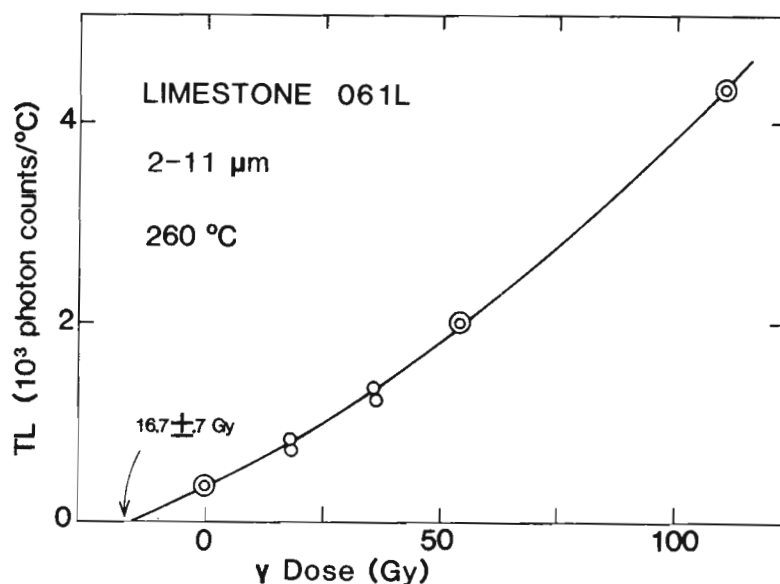


Fig. 2 Supralinear growth in TL at the 260°C peak of Figure 1.

of that observed by Debenham et al. (1982) for a Holocene travertine for which the natural TL emissions occurred above ~ 500 nm while the radiation induced TL had a broader emission, extending down to ~ 450 nm. This spectral distinction of the natural TL from the TL induced by ionizing radiation, coupled with their observation of supralinearity over the ~ 400 -575 nm range (they used the Corning filter 4-96), suggested to us that the supralinearity we observed (e.g. Figure 2) might be eliminated if we were to block the > 500 nm emissions and examine the TL at the blue wavelengths. Unfortunately, second-glow growth curves at ~ 550 nm were uninformative because of poor reproducibility.

TL Behaviour at Blue Wavelengths

We repeated some experiments on 060 and 061L using a Corning CS5-58 filter (transmission window ~ 375 -475 nm) in place of the Spectraccoat No. 546 filter. The effects were of two kinds. Firstly, there was a dramatic increase in the 330°C peak relative to the 260°C peak, such that the ratio of intensities (260/330) was < 0.5 . An example of the glow curves obtained for the 88-250 μm grains of sample 061L is shown in Figure 3. Secondly, the growth curve at each peak for sample 060 was linear, although for 061L the supralinearity remained.

It is heartening to see that the supralinear behaviour of calcite can be defeated in some cases; however, the differences between these two samples are not obvious, though 061L does contain a few per cent magnesium carbonate, while 060 does not.

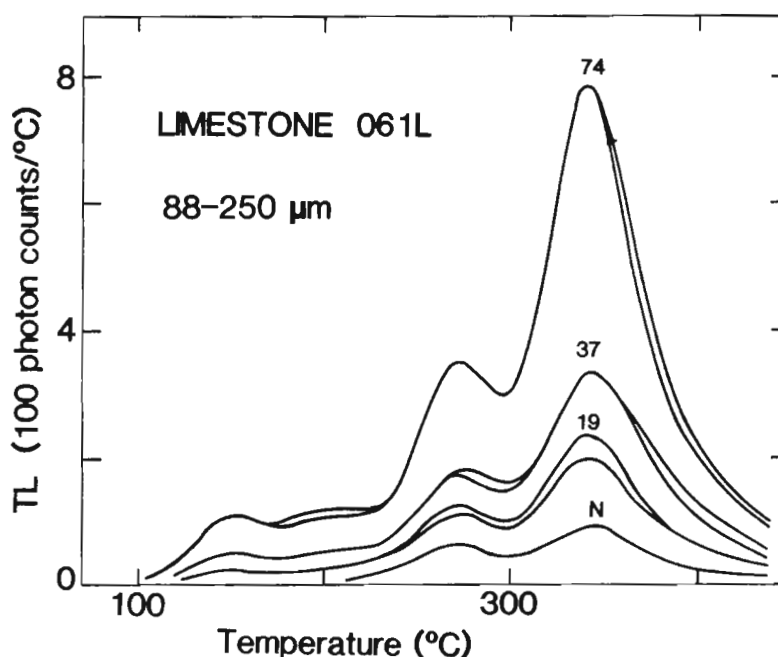


Fig. 3 Smoothed glow curves for the 88-250 μm fraction of sample 061L at the blue ($< 500 \text{ nm}$) wavelengths. There are two curves at each dose. All data have been mass normalized.

Implications

We speculate that if the cause of this supralinear behaviour can be determined and its effects controlled, then the fine grains of limestones should be useful for dating both cultural and geological heating events. For pre-Holocene ages it is not clear that supralinearity will be significant (e.g. Debenham et al. 1982). Even the sublinear behaviour of the high temperature peak from older calcite (Debenham 1983) is no obstacle to determination of EDs from first glow growth curves, because it is easy to prepare large numbers of uniform discs of fine grains in order to carefully define growth curves.

Summary

We prepared 2-11 μm grains of calcite without disturbing their natural TL by using an inexpensive, high-speed, water-cooled carborundum blade such as is present in most geology departments, and by crushing the sample in a steel percussion mortar. The fine grains prepared in this way showed little spurious TL, given a low flow rate and low pressure ($\sim 0.3 \text{ l/min}$ at $\sim 150 \mu\text{m}$) of purified argon.

Finally, two geologically old limestones, believed heated in the Holocene, exhibited supralinear TL growth curves. For one sample this supralinearity was eliminated by use of the $< 500 \text{ nm}$ emissions, rather than the dominant emission at $\sim 550 \text{ nm}$.

Acknowledgements

We are grateful for financial support received from an NSERC grant held by Professor F. T. Hedgcock of McGill, and a SSHRC grant held by Professor P. E. L. Smith at the Universite de Montreal, who also provided the samples. We also thank Professor D. J. Huntley for conducting the XRD analyses, for preparing the large-grain discs, and for his generosity in allowing us to use the TL facilities at S.F.U.

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Editorial Comment

Although in the present work the motivation for using a blue filter was to avoid supralinearity, in the case of Debenham et al. (1982) the motivation was to avoid zero-age TL which in young samples was liable to give rise to too great a TL age. This is discussed further in a recent article by Debenham et al. (Archaeometry vol. 26, no. 2, pp 155-170) who also report the use of fine-grains from calcite for determination of alpha effectiveness.

Rapid Thick Source Alpha Counting

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A difficulty often experienced when measuring dose rates of sedimentary samples is that low activities can be encountered. Thick source α counting using a 13.85 cm^2 screen is a common way of determining the uranium and thorium decay series contributions, however for low activity samples the time taken to achieve reasonable counting statistics may be very long, and discrimination between the series of pair counting would take even longer. To overcome this difficulty a simple inexpensive device can be constructed which increases the volume of material actually counted. Fig. 1 is a block diagram of the equipment used in our laboratory; Fig. 2 shows the counter head assembly in more detail.

The perspex sample holder is similar in design to the small ones supplied by ELSEC. As commercial α phosphorescent screens are expensive and have a high background we make our own. A sheet of Fascal 710 transparent adhesive is stretched over an aluminium ring and trimmed to size. Levy West G345 ZnS(Ag) is sprinkled uniformly over this and the excess is shaken off.

When counting a background the spacers are placed directly on the screen and the sealed sample holders are left for two days before counting to enable the short-lived daughters of radon already on the screen, and the ZnS(Ag) phosphorescence to decay. Before counting a sample it too should be left for 2 days after sealing, although it is preferable to leave it for a month so that ^{222}Rn will be in secular equilibrium with ^{226}Ra . Radon emanation can be corrected for later. To overcome problems of sample inhomogeneity common with many sediments, about 70g should be homogenized in a seed mill, and the required counting sample then be extracted from this. By comparing derived activities for crushed and uncrushed samples it has been possible to clearly demonstrate that for many aeolian sediments the radioactive nuclides are concentrated in crusts coating the matrix grains.

The sample is placed over the mouth of a plastic conical funnel which has its inner wall painted with white high gloss enamel paint to reflect the light pulses from the screen down to the photomultiplier tube (PMT) base. Using a pulser and a timer, a gating pulse of 0.4 sec. duration, but with a random delay of up to 20 msec., is used with a scaler to record the pair pulses. Another scaler records the total number of pulses.

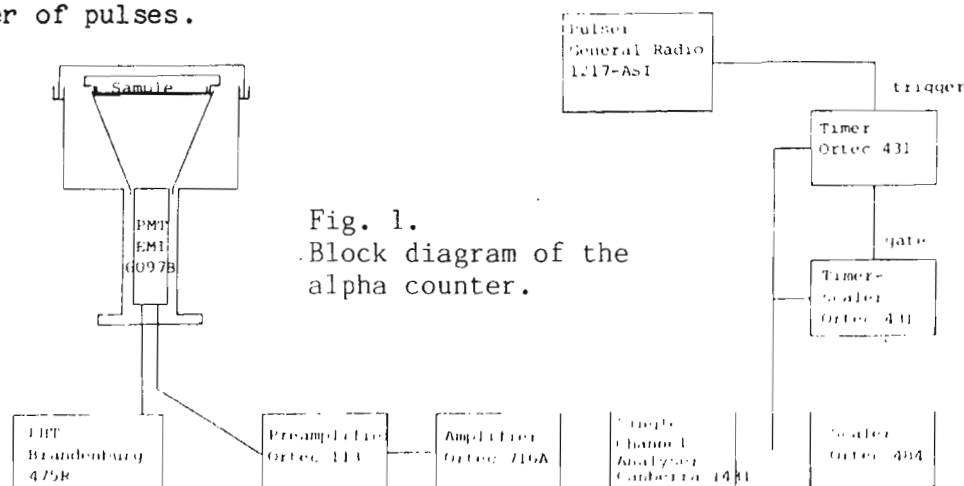
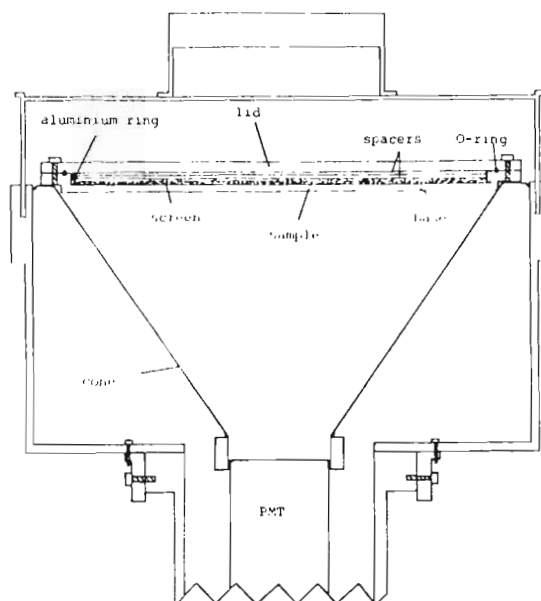


Fig. 1.
Block diagram of the
alpha counter.

As the light pulses from the screen generally suffer many reflections before reaching the photocathode, they are of weak intensity and so the PMT pulses have to be greatly amplified to raise them above the electronic noise background. For our combination of EHT and gain the background is typically 7.5 counts/hr total and 0.03

Fig. 2.
Cross-section
through the alpha
counter head
assembly.



pair counts/hr, of which the electronic noise contributes less than a third. As a comparison, a sample containing average ^{232}Th and ^{238}U decay series activities of 4.3 and 3.6 Bq/kg, respectively (equivalent to 1.1 ppm Th and 0.29 ppm U), yields 82.7 and 1.9 true total and pair counts/hr. At our operating efficiency of 85% for ^{232}Th decay series particles, less than 0.02% of β particles and $5 \times 10^{-6}\%$ of γ rays are detected.

The counting efficiency is less at the edges of the screen than in the centre, so calibration requires that a sample of known activity covers the whole screen. For this purpose $\text{Th}(\text{NO}_3)_4 \cdot 6\text{H}_2\text{O}$, which was refined in 1906 and hence is now in secular equilibrium, can be obtained from the Radiochemical Centre, Amersham, U.K., and can be diluted with silica and homogenized in a seed mill. The activity of the silica must be taken into account. When calibrating the counter it is generally easier to set the modules' parameters and then determine the relative efficiency rather than set the counter to record 85% of ^{232}Th decay series particles. The equations needed for conversion of count rates to specific activities can be obtained from the author.

Acknowledgements

The author wishes to thank D. M. Price and G. Pike for their assistance in the design and construction of the α counter head assembly and sample holders. The work was carried out under the supervision of Dr. A. J. Mortlock and while the author was in receipt of a Commonwealth Postgraduate Research Award.

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Editorial comment (MJA)

In response to the comment that use of sealed samples would accentuate error due to overcounting (such as reported in various articles of PACT 9) the author replied as follows:

Pair counting has shown that Rn-220 is largely responsible for the radon emanation observed with alpha counting; this is due to Th-232 and Th-228 being distributed on the crusts of grains. This inhomogeneity leads to much of the observed overcounting but very fine crushing of samples, as is done when preparing XRF pellets largely eliminates the problem.

Unusual Features of the Thermoluminescence Signal Profile for Sediments from beneath Lake George NSW

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The thermoluminescence signal as a function of depth has been measured on a core designated LG4 taken from the sediments beneath the northern end of the large, ephemeral fresh water lake, Lake George, New South Wales, which lies some 40 km to the north east of Canberra. The measurements were initially made using the inclusion method and the preliminary results reported by Mortlock and Price (1980). The TL signal at 375°C grew steadily with increasing depth and it was possible to make a provisional TL age determination at a depth of one metre (1.1×10^4 years) which compared well with a radiocarbon age of 0.9×10^4 years at the same depth. This age correspondence is important as it indicates that the TL signal is not spurious in its origin and that any surface residual signal must be negligible. These TL measurements were the first reported on sediments from within Australia. The annual radiation dose rates in all these cases were determined by XRF measurements of the contributing radioactive elements U, Th, K and Rb.

Since that time more measurements have been carried out on the same core, not only extending to greater depths but also looking at the depth variation of the TL signal in greater detail.

The results obtained using the inclusion method on 100 μm separated quartz grains are brought together in Figure 1. The error bars correspond to standard errors of the means of the several individual readings taken at each depth. The numbers of these individual observations which go to make up one plotted point varied, but the average was six.

Two unusual features in the profile are immediately evident. The first is the apparent break between 5.5 and 8.2 m with the TL signal at depths below 8.2 m seemingly being translated by a fixed amount to

lower values than expected from an extrapolation of the earlier part of the profile. The second is an isolated high mean observation at a depth of 54 cm. These features cannot be explained by variations in annual radiation dose rate along the core. Such variations were relatively small, showing a standard deviation of 10% from the mean, and no correlation with the features themselves.

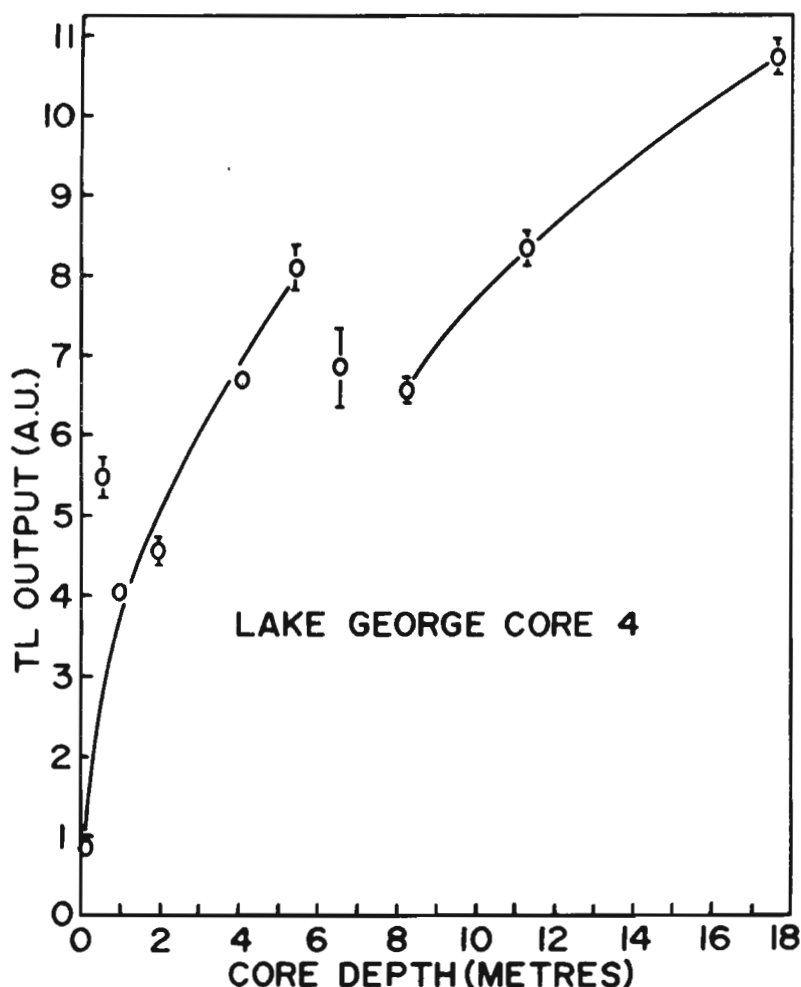


Figure I Variation of TL signal with depth beneath Lake George, N.S.W.

Before attempting to give other tentative explanations of these features some particular physical characteristics of Lake George must be set down. A full description of the Lake, its stratigraphy, palaeomagnetic chronology and vegetational history has been given by Singh, Opdyke and Bowler (1981) and the reader is referred to this for information too extensive to be repeated here.

Of importance is the fact that the Lake is not always full. It can in fact be totally dry and the water level is certainly subject to great variations over quite short periods of time. In essence it is a large relatively shallow ($\leq 5\text{m}$) pan which gains water from a number of sources and loses it by evaporation and seepage to depths below the floor.

Sediment is contributed to the Lake from four sources:

- (i) bedload and suspended sediment carried by the numerous creeks that empty into it.
- (ii) weathered material from the steep western escarpment accumulates as debris fans near the bottom edge of the slope near the Lake edge. Particularly during phases of high water such material is washed directly into the Lake.
- (iii) shore-line material is eroded directly by wave action and redistributed within the water body.
- (iv) a small component of aeolian dust is contributed especially during times of drought.

The core studied is located in the lake bed approximately 1.2 km from the rising edge of the western escarpment. It can be expected to contain material from all four of the above sources in varying proportions.

It is possible to envisage in particular a substantial amount of sediment coming into the area of the core from source (ii) over a short period of time due, for example, to strong wave action at a time of storm. All this material would have the same near-zero TL stored energy at the time of deposition and would tend to produce a plateau-like feature in the TL vs. depth profile obtained today.

Another mechanism which would lead to a reduction in the stored TL energy in the sediment of the Lake relative to expectation otherwise would be an extended period of strong soil formation. This would be anticipated to be associated with dry periods. However, it is difficult to see how this could influence a depth increment as large as 3m.

The feature at 54 cm could be explained by the presence in the core of a single piece of debris carried from the escarpment out onto the floor of the Lake. This is known to happen when the Lake is dry. This piece of debris could retain some stored TL energy over and above what is fully dispersed sediment would display. Another more extreme explanation of this particular observation is mentioned elsewhere, Mortlock and Price (1984).

It has to be remarked here that the description of a visual inspection of the core along its length given by Singh et al. does not draw attention to any particular anomalies that might be of value in this discussion.

In conclusion it can be stated that the present study has demonstrated that the TL vs. depth profiles for lakes such as Lake George can exhibit irregular features which are unlikely to be found with deep ocean sediments. It may be possible to use these features to throw further light on the geological development of the Lake. Other similar fresh water lakes can also be expected to exhibit irregular TL vs. depth profiles.

The authors are indebted to Dr. J. Bowler, Dr. G. Singh and Professor K. Campbell of this University for useful discussions. Also to Ms. W. Cobcroft of Osaka, Japan, who established contact for us with Dr. Miono of Osaka University.

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Co-operation Sought

If you are seeking co-operation, either as a recipient of certain types of samples for TL research or as a donor seeking TL dates, please send us a brief summary of your requirements and we will include them in this new section of Ancient TL.

A Cautionary Note On The Measurement of Quartz TL Immediately after Irradiation

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Observations have been made of the TL from coarse grain quartz immediately after a laboratory beta-irradiation. It has been found that the scatter in the measurements decreases with time after the irradiation. More important, several observations were made which indicate that in some cases there is an apparent fading immediately after a laboratory irradiation. It is suggested that this may not necessarily be due to the loss of charges from deep traps, but could be due to a transfer of some charges associated with the 100° peak to higher temperature traps during heating.

The effect was originally observed whilst coarse-grain dating an aboriginal ovenstone (EB1/F2-L). The first and second growth curves at 340° are shown in figure 1. It can be seen that the discs which were left for several days showed linear growth with good reproducibility between discs. On the other hand, discs which were glow-out within a few hours of beta-irradiation had a high TL in all cases and greater scatter. This was thought at first to be a change in instrumental efficiency, but further measurements in which all TL outputs were measured on the same day showed the effect to be real.

Not all quartz samples appear to be subject to this phenomenon. Measurements carried out on a sample of baked sand (SC3/6) showed that the TL after six days was within one standard error of the TL after fifteen minutes, for all temperatures between 270° and 460°.

A marine sediment sample (RED42-40) also shows the effect. The TL was measured for a range of time intervals after irradiation of from ten minutes to eight weeks, with all glow-outs made on the same day to minimize errors introduced by equipment sensitivity changes. The sample discs were initially glow-out to give the natural TL, and then given a 37.7 Gy beta dose. The discs were normalized using three

methods: natural TL, weight and dose. The dose normalization involved giving each disc a further 37.7 Gy dose and measuring the TL (3rd glow) after one week. All of the normalization methods gave similar results.

Figure 2 shows the TL response for discs left for more than twenty hours compared with those left for less than six hours. These are typical time periods that would apply if the samples were either glowed on the same day as irradiation or left until the next day. The TL was integrated over sixty degree intervals to minimize errors introduced by small temperature shifts in the glow curve. The boundary between the 250° and 310° intervals was in a region where the TL was increasing at a fast rate, which led to the errors for these intervals being larger than those for the higher temperatures. The data are consistent with an average fading of about 5% and the uncertainties are such that no conclusion can be drawn about whether fading depends on glow temperature.

There are very few references to the fading of quartz. Indeed, most researchers have reported no fading. One exception is a series of measurements by Mejdahl (1983) which showed the same fading of between eight and ten per cent for three, six and nine month periods, when compared with control samples that had just been irradiated. Mejdahl does not mention how much time elapsed between irradiation and measurement of the controls, but it was presumably the same day. His results would then be consistent with our measurements. Further evidence has been obtained by Readhead (1982) who also had difficulty obtaining a flat age plateau for quartz if the low temperature TL was not allowed to decay thermally before glowing.

It is thought that the apparent fading is due to charge transfer from 110° peak to higher temperature (deeper) traps during the glow out. Trap competition during heating has been described by Paige (1957), Aitken et al. (1968) and more recently Levy (1983), who produced expressions which clearly show that the higher temperature peaks depend on the initial trapped charge concentration in the lowest temperature trap. Levy now suspects that some of his previous measurements on quartz (e.g. Fuller and Levy, 1978) have produced physically unrealistic kinetic parameters due to charge transfer between different types of traps.

An interesting conclusion from the present measurements is that ambient temperature decay of the 110° peak does not transfer the same amount of charge as does heating the sample (at say 10°C s⁻¹) during TL measurement.

The dependence of TL output on heating rate was checked for sample EB1/F2-L. All discs were glowed out (with a 10°C s⁻¹ heating rate) fifteen minutes after irradiation, but one set was given a preliminary heating to 150°C at 2.5°C s⁻¹. The latter set gave a 350°C peak area 7 (±6)% less than the other. This is consistent with a reduction in charge transfer but unfortunately, as is common with quartz, the uncertainties are large. It does, however, suggest that the phenomenon is not anomalous fading - rather the contrary: anomalous early excess.

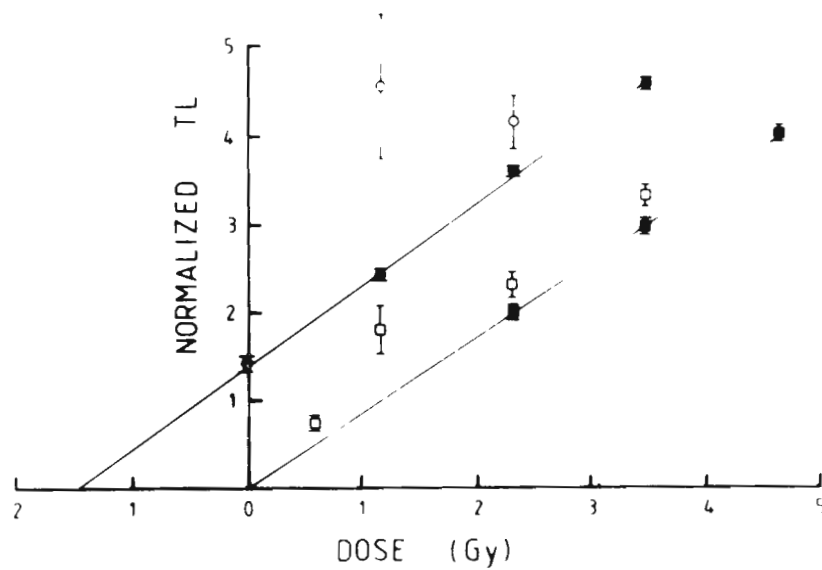


Figure 1. First and second glow TL growth curves at 340°C for sample EB1/F2-L
Error bars show sample standard deviations.
Open circles: 1st glow same day as irradiation.
Filled circles: 1st glow two days after irradiation.
Open squares: 2nd glow same day as irradiation.
Filled squares: 2nd glow five days after irradiation.

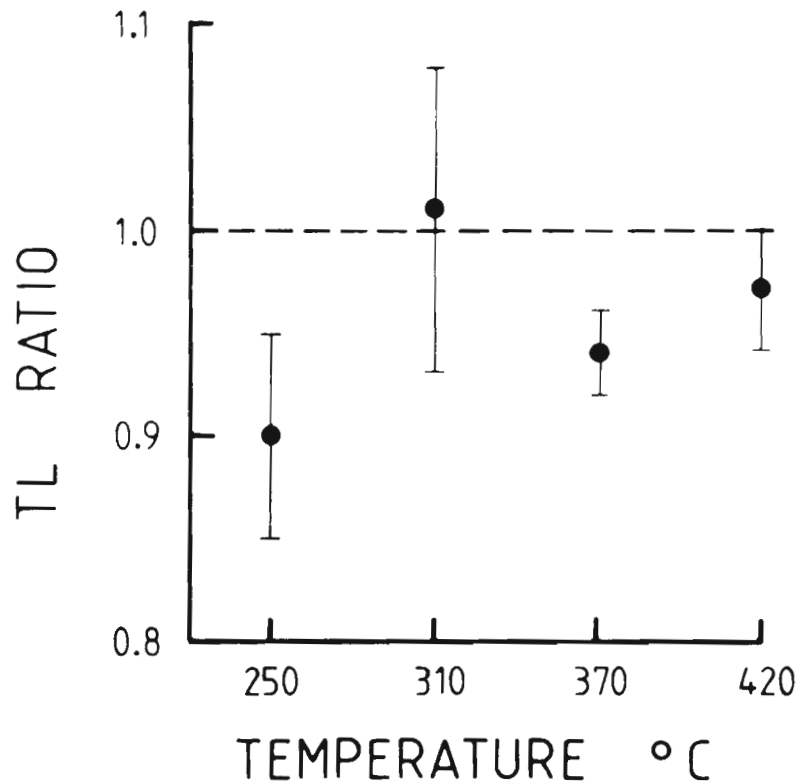


Figure 2. Sample RED 42-40. The ratio of TL for discs left at least twenty hours after irradiation to that for discs glowd after intervals of less than six hours. Sample standard deviations are shown.

It is stressed that anomalous excess may not occur for all quartz samples. We have found it most prominent in quartz which has been recently prepared from the field sample. Repeat measurements on sample EB1/F2-L after it had been sitting in a sample bottle for two years showed that rapid fading after irradiation still occurred, but to a lesser extent than is shown in figure 1. The reason why this made any difference cannot be explained.

Even when anomalous excess was not apparent, a better disc-to-disc reproducibility was obtained if the discs were not glowed for at least a day after irradiation. This is shown in the following table which gives the standard deviations for measurements on identical discs at 350°C (1) immediately after irradiation, (2) at least one day after irradiation, and (3) after the 110°C peak has been removed by an extremely slow heating rate.

SAMPLE	STANDARD DEVIATION (%)		
	(1)	(2)	(3)
EB1/F2-L	10.1	6.8	8.4
RED42-40	11.5	5.8	
SC3/6	23	22	
BPG	6.5	4.7	

The large scatter in measurements of SC3/6 has been discussed in detail elsewhere (Smith, 1983).

If only for the improved precision, we recommend that quartz is left for at least one day after irradiation. If this is not practical, the 110°C peak should first be removed using a very slow heating rate.

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