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A model for sensitivity change of IRSL signals

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Introduction

Sensitivity increases or decreases of infrared stimulated luminescence (IRSL) signals after laboratory bleaching procedures have been reported (Li and Wintle, 1991, 1992a&b, Duller 1991, 1992b). It was found that the sensitivity change is related to the extent of laboratory bleaching, age of the sample and the extent of sunlight exposure which the sample experienced prior to deposition (Li and Wintle, 1991). This change in sensitivity was explained by the competition between hard-to-bleach (H-type) traps and easy-to-bleach traps during irradiation in an empirical model proposed by Li and Wintle (1992b). Here, we explore the competition in further detail.

The model

1. Two types of trapped charges and IRSL sensitivity.

We consider two types of trapped charges: E-type and H-type. It is hypothesised that the E-type are easy to bleach trapped charges, i.e. they are bleached easily by sunlight and correspond to the IRSL signal measured. The IRSL growth curves used in ED determinations represent the dose response of this type of trapped charge.

The other type, the H-type, are hard to bleach trapped charges, i.e. they are hard to bleach, but not non-bleached by sunlight. They can be bleached by prolonged sunlight exposure. The concentration of H-type charges increases with dose, but they do not contribute to the growth curves of the IRSL signal used for dating.

Both types of trapped charges build up with dose toward a maximum level.

In this competition model, it is hypothesised that there is competition between both types of traps during irradiation. The sensitivity of the IRSL signal (X) is related to the concentration of H-type charges (I). The higher the H-type charge concentration, the higher is the sensitivity of the IRSL signal. Hence, the sensitivity, X, can be empirically described as

$$X = \eta I + X_o \quad (1)$$

where η and X_o are constants relating to the competition and the minimum dose response respectively.

2. Sensitivity changes

Sensitivity change was observed experimentally by comparing the additive dose growth curve with the regeneration growth curve. The sensitivities may be expressed by the initial slopes of both curves. X_A and X_R (fig.1), and relate to the responses after deposition (A) and after laboratory bleaching (C) respectively. (B) represents the natural sample.

The sensitivity of the additive dose growth curve X_A relates to the concentration of H-type charges after the sample was last exposed to sunlight, I_a (fig.2). Hence, equation (1) can be written as

$$X_A = \eta I_a + X_o \quad (2)$$

and X_A is thus a function of the degree of sunlight exposure prior to deposition.

Similarly, the sensitivity of the regeneration growth curve X_R is related to the concentration of H-types charges after laboratory bleaching, I_c. This represents a portion of the charges in the natural sample, I_b (fig.2), which increased as a function of dose, F(D), and is added to the concentration I_a. Hence,

$$I_b = F(D) + I_a \quad (3)$$

After laboratory bleaching

$$I_c = f(S)I_b = f(S)F(D) + f(S)I_a \quad (4)$$

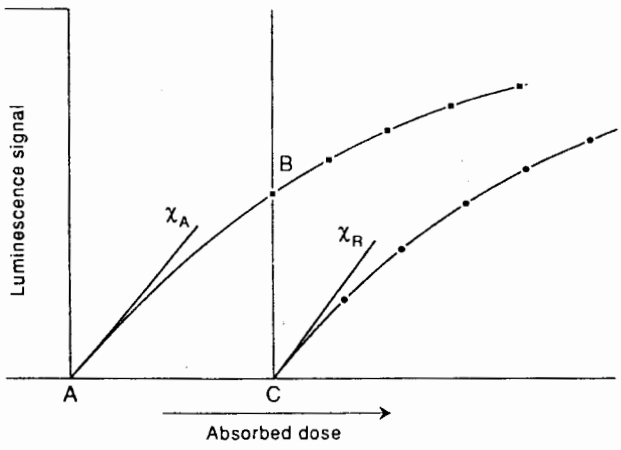

Figure 1.
Schematic diagram of luminescence signal growth curves: the response to added dose (filled squares) and response to dose after laboratory bleaching (filled circles).

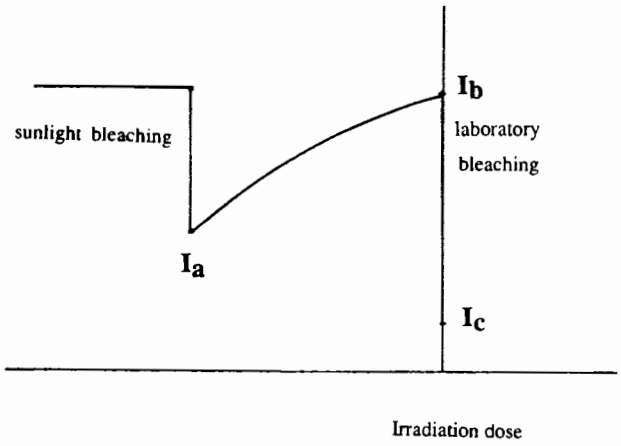

Figure 2.
Schematic diagram showing the change in concentration of H-type (the hard to bleach) trapped charges as a result of natural and laboratory bleaching procedures.

where f(S) is a bleaching factor, which depends on the bleaching time, spectrum and the strength of the bleaching light.

Since the H-type charges can be bleached by prolonged sunlight exposure, their concentration will decrease with bleaching time. Hence, f(S) will be smaller for longer bleaching. The spectrum of light used in laboratory bleaching is likely to be either similar to sunlight, e.g. solar simulator, or a relatively narrow wavelength band. In the latter case, a particular light may or may not reduce the H-type charges when the E-type charges are removed by the light exposure. Since the model assumes that there is no charge migration into the H-types traps during bleaching, f(S) can be defined in the range of $O \leq f(S) \leq 1$.

Hence, substituting (4) into (1), the sensitivity of the regeneration curve is,

$$X_R = \eta I_c + X_O = \eta f(S)F(D) + \eta f(S)I_a + X_O \quad (5)$$

Comparing equations (2) and (5), the sensitivity change after laboratory bleaching is given as,

$$X_R - X_A = \eta f(S)F(D) + \eta f(S)I_a - \eta I_a \quad (6)$$

The sensitivity change is thus related to the age of the sample F(D), laboratory bleaching f(S) and the bleaching prior to deposition as represented by I_a. The sensitivity can increase or decrease depending upon those three conditions.

Predictions of the model

Various predictions can be made from this model for the response of different types of sediments.

1. Shortly bleached sediments (colluvial deposits).
For sediments which have experienced only short sunlight exposure prior to deposition, the H-type charges have not been removed at deposition, because of the short sunlight exposure. The concentration I_a is very high, and may even be close to saturation, $I_a = I_{max}$. For these sediments the H-type charge is likely to be close to its maximum value and hence $F(D) \approx 0$, and $I_b = I_a$. Therefore equation (6) becomes

$$X_R - X_A = \eta I_a[f(S) - 1]$$

As $O \leq f(S) \leq 1$ by definition, a sensitivity decrease will be observed after laboratory bleaching which releases H-type charges. The longer the bleaching, the greater is the sensitivity decrease expected.

One exception of this behaviour will occur when bleaching of the IRSL signal is carried out using the wavelength that is used for stimulation, i.e. IR. The H-type traps will remain full because this wavelength will remove only the E-type charges and will not affect the population of H-type charges, i.e. $f(S) = 1$. This can be seen from the large TL signal which remains after the IRSL is reduced to <1% by 1000 seconds of IR exposure (Li and Aitken, 1989; Duller, 1992a). In this case the E-type traps will fill under the same competing regime as in nature and the sensitivity of the signal response to dose when regenerated will be the same for a sample of any age.

2. Well bleached sediments (aeolian deposits).
Since aeolian sediments were well exposed to sunlight at deposition, H-type traps were empty at deposition, $I_a=O$. Hence, equation (6) becomes

$$X_R - X_A = \eta f(S) F(D)$$

Since F(D) is positive $X_R - X_A \leq 0$, the sensitivity will always increase after laboratory bleaching, unless a prolonged bleaching is applied or a zero age sample is studied (F(D)=O). The degree of increase is dependent upon f(S) and F(D), which relate to the degree of laboratory bleaching and the natural irradiation of the sample respectively. After a short bleach, i.e. fixed f(S), a sensitivity increase would be expected for different age samples; the older the sample, the greater the sensitivity increase expected after the same short bleach. However, for a young sample, F(D) is relatively small and may be negligible; in this case no significant sensitivity change should be observed after any laboratory bleaching. For samples of the same age, i.e. fixed F(D), the sensitivity change will decrease with increased light exposure.

These predictions are in agreement with data reported in the literature (Li and Wintle, 1991; Duller, 1991; 1992b) and in particular with the results of an experimental programme designed to test this simple but predictive model (Li and Wintle, 1992b).

Conclusions

A change in sensitivity of an IRSL signal brought about by laboratory bleaching can be explained in terms of a competition model involving two trapped charge populations. Either increase or decrease in sensitivity can occur after laboratory bleaching and irradiation. Such a model has important implications for laboratory procedures used to determine the equivalent dose. The regeneration method is not suitable without careful consideration of the most appropriate bleaching procedure. When using the regeneration method, it is necessary for the IRSL signal to be bleached to a negligible level and for the H-type trapped charges to be bleached to the level they occupied at deposition.

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High sensitivity TL spectra of quartz and feldspar

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Introduction

During the last three years a new high sensitivity TL spectrometer has been designed and constructed at the University of Sussex (Luff and Townsend, 1993). The basic arrangement of the new instrument is shown in fig.1. The detection system features a pair of spectrometers with gratings blazed for the UV-blue (200-450nm) and blue/green-red (400-800nm) and a pair of IPDs (imaging photon detectors). The performance details are as follows: temperature ranges 20-300K, RT-400°C and wavelength range of 200-800 nm. Heating rates are typically 0.1K s^{-1} in the range 20-300K, and 2.5 °C s^{-1} above RT. The main advantage of the arrangement is that there is simultaneous recording of all wavelengths in the spectral range with photomultiplier photon counting sensitivity. For archaeological or geological TL applications, the effective sensitivity is within a factor of 10 of that of most conventional TL systems using polychromatic light via filters.

Some indication of the sensitivity of the system is that we are easily able to detect spectra from $CaSO_4$:Dy after 1 milligray of x-ray irradiation, and the signal to noise ratio is such that measurements to 10 micrograys are possible. In order to demonstrate the spectral range achieved, a spectrum for CaF_2:Cr_2O_3 is given in figure 2. We are now able to measure the natural TL spectra of geological and archaeological quartz and feldspar. The purpose of this paper is to demonstrate the value of the additional spectral information that we have obtained in a series of preliminary experiments.

TL spectra of natural quartz fractions

The basic pre treatment procedures for samples destined for archaeological TL analysis were established in the 1970s and have remained virtually unchallenged since then. One key procedure is the use of strong etching, with hydrofluoric acid to remove the alpha-damaged surface portions of quartz grains, and to remove any contaminant grains from quartz fractions. The efficacy of the etching method is the issue here, and we therefore present some results of a series of etching tests undertaken with the quartz fractions, in this example from dune sand collected from sand ramps in the Mojave Desert. The quartz fractions were separated using heavy liquid (sodium polytungstate) and then etched for either 40 minutes or 80 minutes in cold 40% hydrofluoric acid. Monolayers of the etched grains were prepared on clean 10mm diameter aluminium discs and the natural TL spectra recorded over the temperature range RT to 400°C. Examples of the resultant emission spectra are shown in figures 3 & 4.

Two samples were chosen to emphasise the range of behaviour which has been seen. After the 40 min etches for both of the samples DL11Q and DL12Q, the TL shows two strong emission bands at 320 and 280nm with, in the case of DL12, broader and weaker features near 400nm and 620nm. After the 80 min etch, the spectra are dramatically different, in DL11Q the intensity of the emission band at 320nm is strongly enhanced whereas in DL12Q the peaks at 280 and 320nm have almost disappeared leaving a broader (noisy) emission peaked at about 350nm and broad emission features peaked at 430 and 620nm.

Several questions are raised by this new spectral information. The first is the extent to which the relatively strong natural TL signals detected at 280 nm and 320 nm result from the presence of feldspar grains or feldspar inclusions or indeed, what evidence do we have that the 280 and 320 nm signals do not come from quartz? Our current spectral measurements made

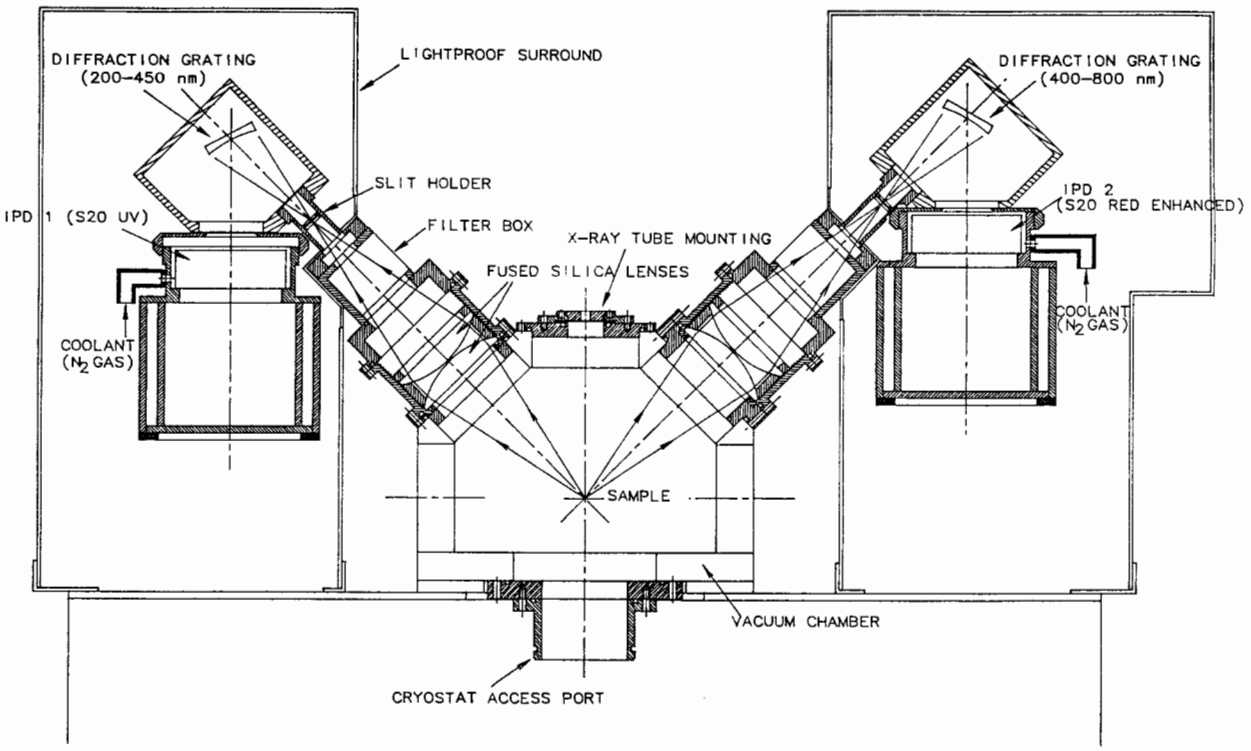

Figure 1
Schematic design of the new TL spectrometer.

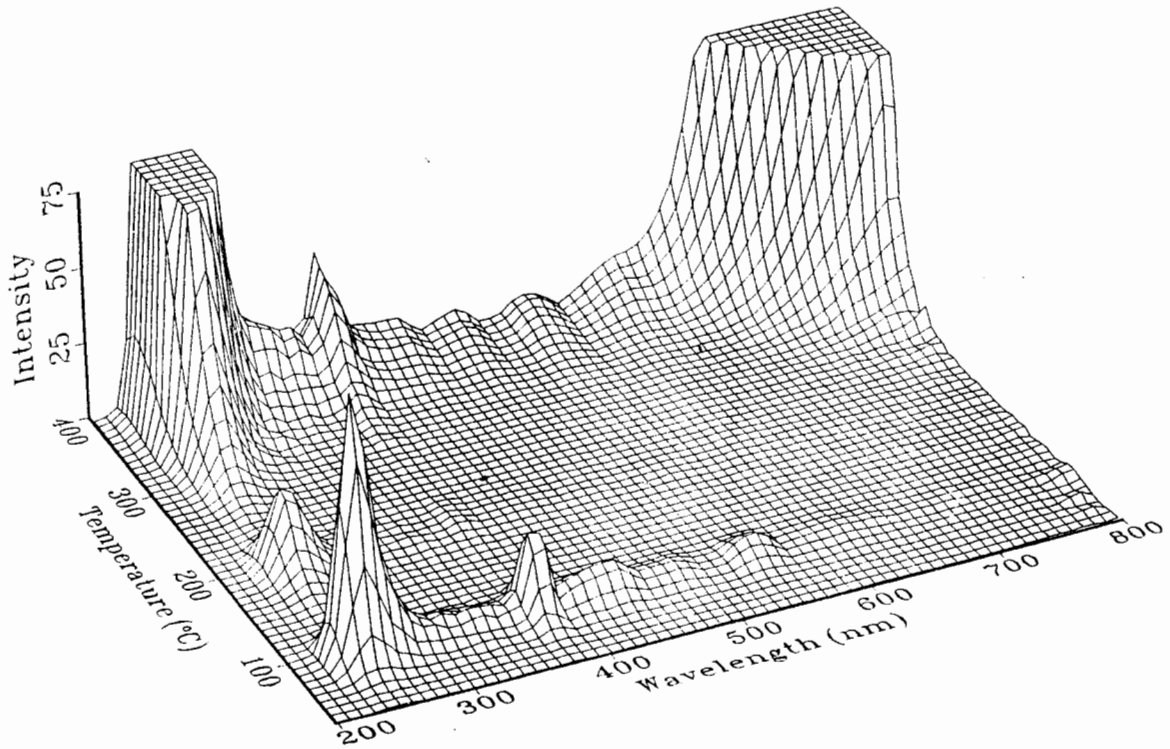

Figure 2.
TL emission spectrum for CaF_2:Cr_2O_3 after x-ray irradiation.

on silicate groups (SiO_4), the recombination luminescence which is strongly localised will appear at nominally the same wavelength, no matter which is the host material.

In the case of quartz and silica emission, peaks reported in this blue/UV region include examples at 260, 290, 380, 390, 400, 450, 460, 470 and 480 nm. The models ascribed to them are variations on the intrinsic $e^- - h^+$ recombination, near 470 nm, or perturbations of the SiO_4 group caused by intrinsic defects (oxygen vacancy, E' or E'_{beta} centres) or impurities such as H, Al or Ge. The variations in sample purity and defect states imply that quartz and feldspar signals may be coincident in wavelength. The chemical etching in HF removes the exposed feldspar and the alpha-damaged quartz more rapidly than quartz which only contains isolated point defects. The crucial observation is that changes in spectra are still continuing after 80 min HF etching which implies that the standard HF etch for 40 min may be only partially successful in separating quartz grains from other material.

The second significant observation is that for DL11Q the luminescence peak at 320 nm increases with etching. From a solid state viewpoint this is surprising but may just indicate the loss of surface defects which were offering non-radiative recombination routes. For dating, the differences between the sensitivity to etching for the component emission bands should be noted and their subsequent influence on dating needs careful consideration.

TL spectra of natural feldspar

The results of TL dating of potassium feldspars have been the subject of considerable debate (Balescu & Lamothe, 1992; Rendell, 1992; Wintle & Duller, 1992). During this debate it has become apparent that in the case of some samples, the values of Equivalent Dose obtained were a function of the choice of emission band detected via a particular broad-band filter combination. The choice of filter has until now been a result of educated guess-work. The potassium feldspar fraction was separated from a small sample of material from the Belcroute site in Jersey (Balescu and Lamothe, 1992). The fraction comprises the 355-250 μm grains

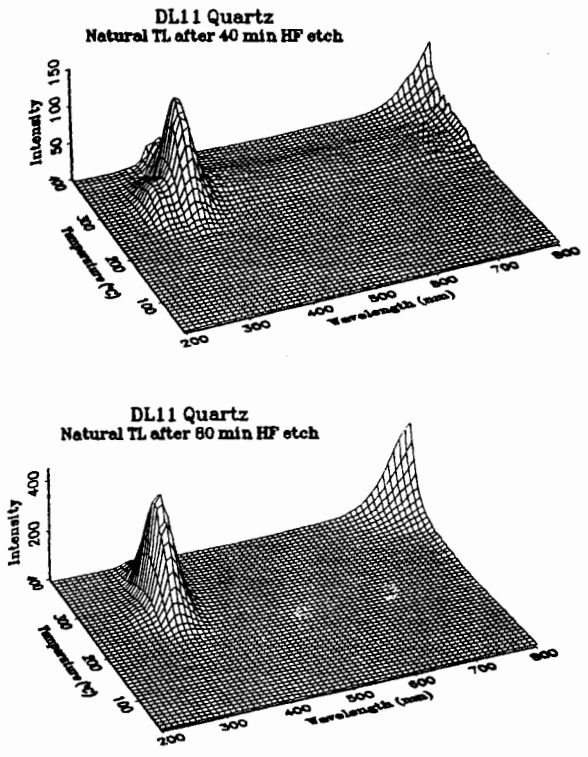

Figure 3.
Emission spectra of natural TL from quartz fractions of sample DL11Q after different etching times.

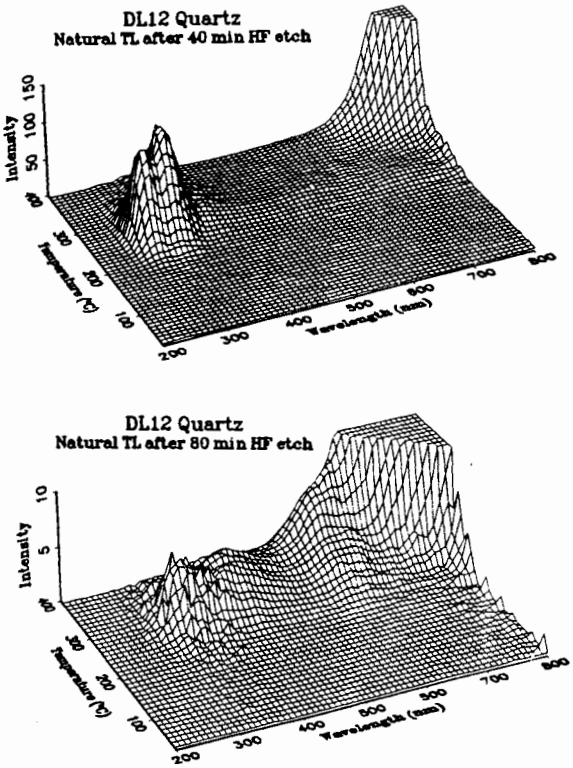

Figure 4.
Emission spectra of natural TL from quartz fractions of sample DL12Q after different etching times.

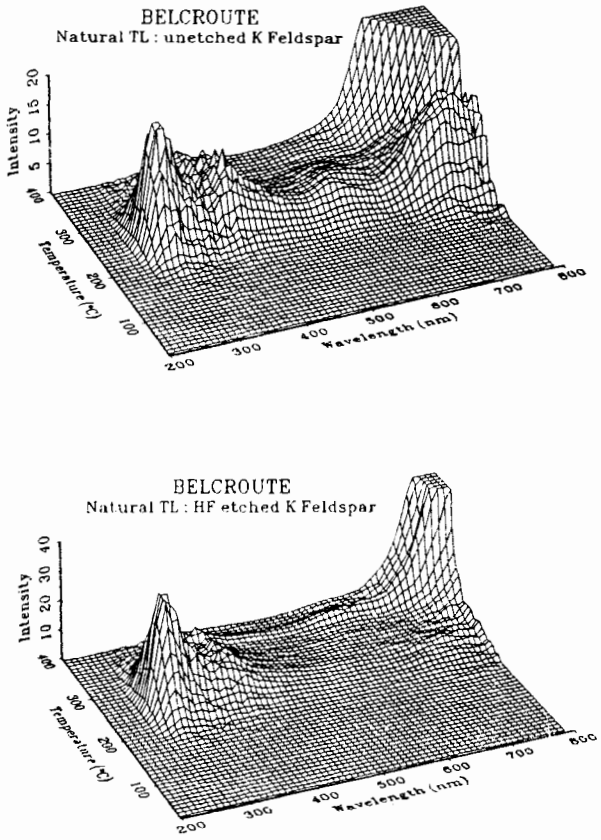

Figure 5.
Emission spectra of natural TL from potassium feldspar fraction of a sand sample from Belcroute before and after etching.

with s.g.<2.58. Spectra of the natural TL are shown before and after etching in 10% hydrofluoric acid (figs.5a and 5b).

The reason for showing both spectra is that, within the luminescence community, some consideration has been given to trying to date separated, but unetched, material. The spectrum of the unetched material (fig.5a) shows emission bands at 280, 340, 380-400, 550 and 730 nm, with glow curve shape varying as a function of wavelength in the blue/UV region. The spectrum of the etched material is dominated by emission in the blue/UV (fig.5b). In particular the intensity of the 280nm emission has increased relative to that at 380-400nm at 260°C, however at temperatures above 320°C, the latter.emission band has a higher intensity.

Any simple filter/PM tube combination will thus offer an integrated TL signal which will certainly be unsuitable for kinetic analysis. More importantly, for dating applications a filter view of such signals will be misleading if the ratio of the component glow peaks and spectral features vary with dose, storage, bleaching or re- irradiation. Unfortunately for this material these variations do indeed exist and so one may derive erroneous ED values. With the hindsight of the spectral information, one should pursue normal TL measurements for such materials using a much more precisely selected spectral region, with perhaps a monochromator, rather than a broad band glass filter.

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PI S.W.S McKeever
This is an interesting series of observations which needs further study. A methodical investigation of the effects of different filter arrangements on calculated EDs is called for. Regarding figure 3, in addition to the increase in TL emission, there is also an evident increase in the black-body emission. It is not obvious why this should be so.

Important date/strange material

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Introduction

A few years ago a beautifully crafted, fossilised bone harpoon point was uncovered at Katanda near the Semliki River in Zaire (Fig.1). Two more whole points and fragments of five others, as well as an equally well crafted dagger like tool were also found in the same stratum. An abundance of large catfish bones was also present at the site. While in workmanship these specimens are not unlike those found at the very end of the Upper Palaeolithic, about 14,000 BP, geological evidence based on the site stratigraphy suggested a far older age, perhaps as old as 100,000 years. Archaeologists Alison Brooks and John Yellen, discoverers of these objects (Shreeve, 1992), aware of the requirements for TL and OSL dating procured samples of the sediments in the immediate vicinity of the specimens, collecting them excluding daylight exposure.

Procedure

Samples were prepared by defloculation followed by sieving, retaining the fraction 95 - 125 μm in diameter. The resulting material was reacted with 2:1 concentrated HC1:H_2O for 15 mins at 70°C to dissolve carbonates and iron oxides that were found to be present. In order to obtain a pure quartz fraction an etch in full 48% concentrated HF was applied for 40 min at 70°C. The resulting material was again sieved, retaining the >90 μm fraction and then density separated first with a ρ = 2.708 g/cm^3 liquid, taking the light fraction and followed by using a ρ = 2.621 g/cm^3 liquid, keeping the heavy fraction.

In spite of these measures, scanning electron microscopy, including x-ray studies, still showed some quartz grains with strongly adhering micro crystals. A wide field scan of about 250 grains confirmed that by weight the material was predominantly quartz. However, x-rays from selected small attached micro crystals indicated the presence of minerals with atomic compositions (ordered by line strength); (Si, Mg, Al), (Si, Mg, Al, Ca), (Si, Ca, Cl), (Si, Mg, Al, Ca, Cl), and even (Si, W). These results are to be contrasted to those for the relatively very pure quartz grains found in the Kalahari sand dunes (Hornyak, et al., 1992). In that case the number of grains with bonded micro crystals was considerably fewer. Their principal atomic composition found in x-ray analysis was (Si, Mg, Al, Fe) and (Si, Al, K, Na). It is not evident what these differences for the two materials would have on the TL and OSL characteristics.

The first TL technique utilised to arrive at a palaeodose for the Katanda material was to use a slowly bleaching signal. This signal was isolated by a light bleach of 10 min at 550 nm (using a 100W halogen lamp with suitable filters yielding an illumination rate of 23 mW/cm^2).

All observation of luminescence was in the UV using a Schott UG11 filter in combination with a Chance HA3 filter and an EMI 9635Q photo tube. Figure 2(a) shows the glow curve for the Katanda natural dose sample, and in Fig.2(b), the glow curve for the Kalahari natural dose sample multiplied by a normalisation factor of 2.5, and Fig.2(c) the difference (a) - (b). All these glow signals are slowly bleaching. A small admixture of peak (c) in the Kalahari data near the region A may be present. In both materials the glow peak component (b) is identified with the well known "375°C" SBP, studied extensively (Hornyak, 1992). At a heating rate of 1°C/s this peak appears at 331°C with green emission and at 312°C with UV emission. It was found that the slight anomaly at A in peak (b) if indeed due to the presence of peak (c) appeared to be a very weak contribution at all doses, in contrast to the Katanda material where peak (c) was a prominent dose dependent signal. This leaves the attribution of peak (c) to quartz uncertain, therefore only

the area in the temperature range R (320-380°C) was used to define a unique quartz signal. In view of the fact that the SBP at "375°C" behaves as if it were approximately obeying second order kinetics, all glow curves were height normalised and temperature shifted to bring the region defined by R into coincidence.

Dose growth curves for TL were obtained for the Katanda material using a convenient (Sr-90,Y-90) ß-source which was calibrated against a standard Co-60 source. After each beta dose a 5 min. preheat at 170°C was done followed by the application of the above 550 nm bleach. This procedure was followed for obtaining both the additive dose growth curve (N+ß) and the regenerative dose curve, this latter obtained as described above after a 16 h bleach with the solar simulator.

After noting that the two data sets appeared to closely define a common saturating exponential behaviour, it was assumed both sets of data represented a constant dosimeter sensitivity. The cautionary note by Smith et al. (1990) that the ED from an N + ß determination did not appear to be operating here. Melding the two curves has the advantage of giving a consistent ED even taking into account the presence of the unbleachable residual signal. A saturating exponential fitting routine was used to simultaneously fit both the regenerative data points as well as the additive dose data points which were shifted in dose by constant trial dose. This was repeated for various shifting trial doses and in each case obtaining the variance generated by the fitting program. The dose shift for the lowest obtained variance was taken to be the palaeodose. Figure 3 shows the results for this optimal fit. The insert in Fig.3 shows the variance of the fit as a function of the trial dose. The resulting value of the palaeodose is 170 Gy.

The same procedure was applied to the Kalahari material, including selecting the same temperature region R for the data sets. Again, a very satisfactory melding together of the additive dose and regenerative dose data sets was possible. To a surprising extent these unified dose growth curves for the Katanda and Kalahari data matched each other as well as the additive dose curve obtained for the Kalahari material observed in the green emission when it is properly normalised. For example, the ratio for the observed TL signal at a dose of 170 Gy (the Katanda palaeodose) to that for a dose of 35 Gy (the Kalahari palaeodose) is just 2.5, the normalisation value needed in Fig. 2 to obtain the subtracted result 2(c). The success for using a single saturating exponential to fit the melded data taken in temperature range R is probably due to the exclusion of peak (c).

The strange luminescence behaviour of the material immediately became evident when TL signals based on the rapidly bleaching peak (RBP) was measured in order to establish an additive dose growth curve (N + ß). The RBP at each dose was generated by subtracting the glow curve obtained for the SBP as described earlier, from glow curves without the 10 min, 550 nm bleach (Franklin and Hornyak, 1990). The scatter in the data even after measuring many aliquots was considerable. Similar poor results were obtained for the TL regenerative dose curve using this subtraction method following a 16 h solar simulator bleach.

Figure 1.
Fossilised bone harpoon found at Katanda, Zaire.

Equivalent RBP additive dosing and regeneration dosing information was sought using the single aliquot OSL technique (Duller, 1991). The results are shown in Fig.4 both for the additive dose curve (a) and the regeneration dose curve (b). The behaviour shown for both curves is identical with the TL subtraction data set, only in this instance with virtually no scatter in the data points. Every ß-dose was followed by a preheat of 5 min., at 170°C. The OSL signal followed was for a 5 min., 550 nm exposure and observation of the luminescence was in the UV. A natural dose aliquot was given a repeated sequence of preheats and OSL reads to obtain a measure of the OSL signal loss during the entire experimental run. Ten repeated sequences gave a loss function

$$1.018 \exp(-1.797 \times 10^{-2} n)$$

where n is sequence number (n = 0 is not defined). The resulting correlation coefficient for this exponential fit is 0.9973. The data shown in Fig.4 have been corrected for this loss.

The anomalous minimum in the OSL additive dose growth curve shown in Fig.4(a), suggesting the presence of some unknown type of dose induced fading is also present in the RBP TL data based on the subtraction technique. Attempts to combine curves (a) and (b) for added doses above ~120 Gy, where a rise towards saturation is evident, in the manner that led to the Fig.2 results were unsuccessful. However, the regeneration dose corresponding to the natural OSL signal (intersection produced by line AA') yields a palaeodose of 192 Gy. It should be noted that the natural OSL signal does not properly belong in the remainder of the additive dose set. If any radiation-induced fading is involved in the procedure used, this datum point is not affected as of course would be the case with the rest of the data set.

A third method to obtain the palaeodose was using single aliquot OSL regeneration of the isothermal luminescence decay under continued illumination with 550 nm light. The procedure was to follow each dose with a 5 min, 170°C preheat, a recorded OSL luminescence decay for 300 s and a continued bleach up to 1200 s. It had been determined using the natural material that a 1200 s bleach left less than 1 percent of the original OSL signal. Decay curves were obtained for a range of regeneration doses spanning the results for the natural material. At 5 s intervals of the decay curves the regenerated dose corresponding to the natural material was determined. The results are plotted in Fig.5. The apparent decrease to an asymptotic limit at long times was simulated by a decreasing exponential function plus a constant using a computer program fitting routine. Various trial constant doses were tried with corresponding best fitting exponentials. The correlation coefficient for the fit as a function of the constant trial dose is shown in the insert of Fig. 5. The highest correlation coefficient occurred with a trial dose assumed to be equal to the palaeodose of 185 Gy.

The reason for employing the asymptotic value to represent the palaeodose is exhibited in Fig. 6. The data set labelled N is for the natural state, the set labelled ß is for a ß-dose of 185 Gy. It was found by superposing all the various ß-dose data that a fairly constant decay function shape resulted such as that shown for the set labelled ß. However, the data for the natural state shows a considerably larger rapidly decaying component to be present. This accounts for the larger palaeodose results for the earlier time slices. Fig. 6 also shows that for longer times into the decay a more all inclusive shape

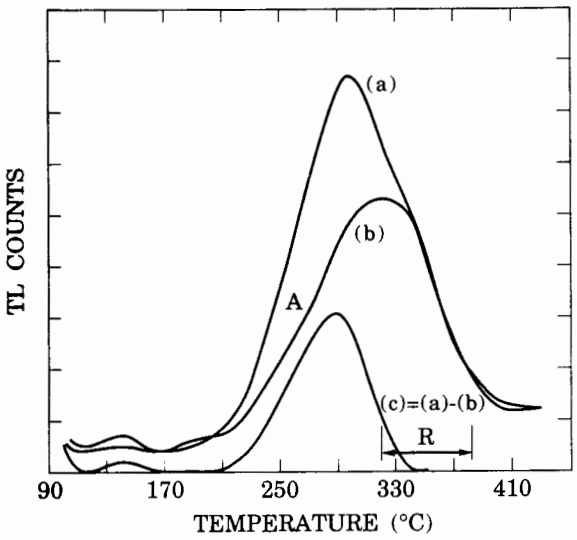

Figure 2.
Glow curves for the slowly bleaching signal observed in the UV at a heating rate of 1°C/s. (a) Katanda natural dose (about 175 Gy); (b) Kalahari natural dose (about 35 Gy) x 2.5; and (c) the result (a) - (b). The region spanned by the temperature range R was used to calculate the dose rate growth curves.

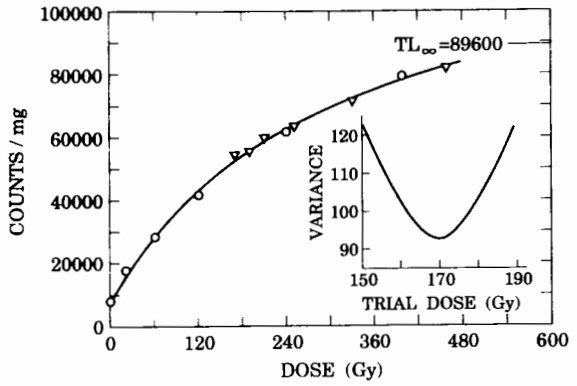

Figure 3.
The result of melding the TL additive dose growth data (triangles) and the regenerative dose growth data (open circles) for the SBP in temperature range R, when the former points are shifted by an optimal trial dose. The insert shows the variance in an overall saturating exponential computer program fit as a function of the trial dose. All data points are for 10 weight normalised aliquots. The resulting palaeodose is 170 Gy.

develops. Unfortunately using this region of time slices substantially increases the statistical uncertainties, and hence the resort to the adopted procedure.

A radioactivity assay of 15 g of sample material was measured using a large volume and a thin intrinsic Ge γ–ray detector. Over 20 γ-ray line strengths obtained spanning the entire radioactive Th and U chain of elements were directly compared with those from National Bureau of Standards certified pitchblende-sand and monazite-sands standards. No decay chain disequilibrium was detected. The assay results were Th-232, 4.11±0.07 ppm; U-238, 0.76±0.02 ppm; and K-40, 1.49±0.02 ppm.

Weight loss measurements were conducted for etching with 48% concentrated HF at 70°C as a function of etch time for pure Kalahari quartz sand (90 - 125 μm diam.) believing that the major luminescence was produced by the quartz component in the Katanda material. This measurement gave an equivalent etch depth of 6 μm for 40 min of etch time. Using the estimations of the α and ß-ray reduction in irradiation effectiveness produced by the etch loss as contrasted to that for γ-rays given by Fleming, 1979, leads to a dose rate of 2.08±0.01 Gy/ka at a confidence level of 95%.

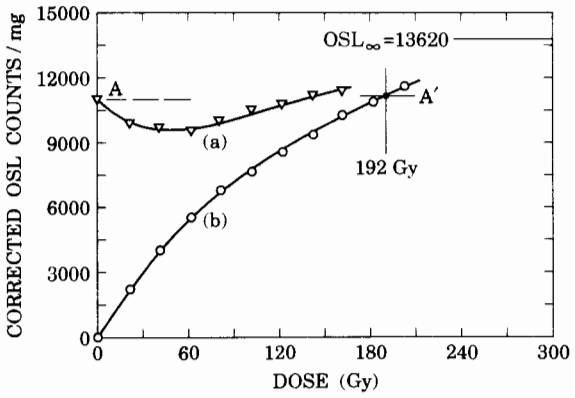

Figure 4.
(a) The single aliquot OSL result for the additive dose growth data and (b) the result for the regenerative dose growth data. The data sets for the two separate aliquots used are weight normalised. The standard procedure for determining the palaeodose is also shown, resulting in a value of 192 Gy.

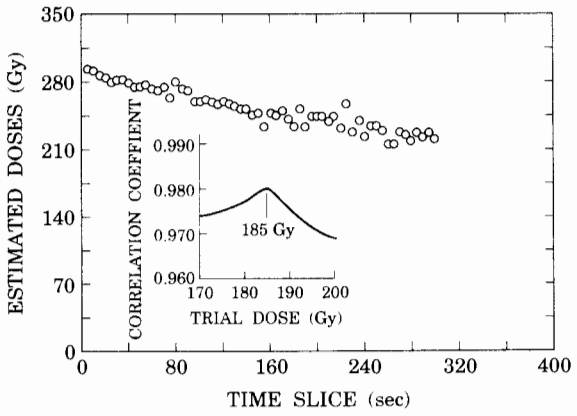

Figure 5.
The result for the time slice determination of the palaeodose from 17°C isothermal decay data obtained under continual illumination with 550 nm light using a single aliquot. A fit to this data was performed using a program with a decaying exponential plus a trial asymptotic constant combination. The insert shows the correlation coefficient to the program fit as a function of the trial constant. The optimum fit occurs for a palaeodose of 185 Gy.

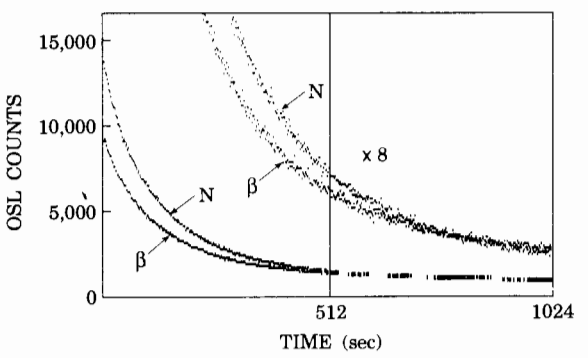

Figure 6.
The family of isothermal decay curves used in Fig.3 all have very similar shapes to that labelled ß, which is reproduced for a dose of 185 Gy. The decay curve for the natural sample labelled N is uniquely different showing an additional rapidly bleaching component. The two sets appear to merge at longer bleach times suggesting a common asymptotic limit. Background counts in these runs are less than 100 per channel.

Conclusions

The palaeodose for the quartz component of the Katanda material is only taken from the SBP results to be 170±10 Gy. In this instance the quoted error is in the nature of a limit of error based solely on the methodology used in the data analysis. Combining this palaeodose with the above value for the dose rate gives an age of 82±8 ka. Due to the uncertainties in the dosemeter material behaviour the quoted error is perhaps no better than a "standard deviation". Particularly uncertain is the role played by the non quartz-like contaminants in the TL and OSL responses. In spite of these problems the two OSL results for signals that may properly be associated with quartz give some supportive evidence for the more unambiguous quoted palaeodose of 170 Gy particularly when noting that bleaching sensitisation effects may be present in the OSL determinations (Li and Wintle, 1991). It is to be noted that the same RBP and OSL experiments conducted with our pure quartz material from the Kalahari did not show any of the anomalies observed with the Katanda material.

Tentative results using ESR dating of a hippopotamus tooth found in the same stratum as the harpoon tips has yielded a possible age ranging between 60 and 110 ka (Henry Schwarcz, private communication).

In any event, the barbed harpoon tips are in fact much older than might have been expected from a comparison with other dated archaeological objects of similar sophistication.

Acknowledgements

The material used in this research was provided by Alison Brooks and John Yellen (see postscript) who also provided much information on the stratigraphy of the archaeological site and the disposition of the objects and the surrounding matrix. We are also indebted to Scott Treude for assisting with some data taking. We wish to acknowledge the NSF support of this project under contract #BNS-9107652.

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The unusual behaviour of the optically sensitive signals from this material (i.e. the RBP TL peak and the OSL signals) are a pertinent reminder of the complexity of TL and OSL processes in natural samples. This is especially so since one might look at the benign behaviour of the SBP TL signal and be seduced into thinking that the processes are straightforward and easily understood. The data and discussion clearly indicate that, for the Katanda material, whenever there is a natural component present in either the RBP TL signal (i.e. the additive dose and the continuous OSL decay curves), unusual behaviour is noted. One is tempted to speculate, after examining the glow curves and reading the discussion regarding the incomplete removal of non-quartz components, that the difficulties encountered may be a result of the unwanted admixture of extraneous minerals. If so, one may perhaps have faith in the date produced since the authors have wisely limited their conclusions to what appears to be the quartz only fraction. Arriving at a physical explanation of the observed properties of the optically-sensitive components, however, will have to wait.

Postscript

John Yellen, the anthropologist who found the artefact, has provided the following comment:

This work is important because it sheds light on the question of when and where behaviorally modern humans first appeared. It suggests, in concert with paleontological information, that this transformation took place in sub-Sahara Africa prior to 50,000 years ago.

Collection of ESR samples from the interior of mammoth teeth causing minimal damage

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Uranium uptake into fossil bones and teeth constitutes a major problem in ESR dating. The actual process of U-uptake is not well known and the uncertainty of ESR age estimates is proportional to the amount of uranium in the samples analysed (see e.g. Grün and Stringer, 1991). Fission track maps of mammoth/elephant molars have shown that most of the accumulated uranium is fixed in the outer layers of the specimen (Grün and Invernati, 1985). This allows us to collect samples from the interior of the teeth which have low U-concentrations and, hence, the uncertainty in the ESR age estimation caused by the unknown U-uptake is rather small. Mammoth molars are one of the few materials that are useful for dating of fluvial sediments, although their frequency in those sediments is variable. The sampling strategy has led to the successful dating of various Quaternary sites, such as the Saskatchewan Sands and Gravels, Canada (Grün et al, 1987), the Balderton Sands and Gravels, England (Grün, 1991) or the interstadial site of Agnadarrah, N. Ireland (Grün et al. in prep.).

For the extraction of the ESR samples in early studies, the molar was cut in half with a diamond saw and a sequence of successive enamel and dentine/cement samples was cut out of one of the halves. During the extraction process the tooth was severely damaged (see e.g. Grün, 1989, Photo 5) and it was usually not possible to reconstruct it afterwards. Although this method was entirely satisfactory from an ESR point of view, only a few zoologists, museum curators or collectors were willing to part with precious specimens for ESR dating and, hence, many Quaternary sites could not be dated.

Our first attempt to limit the damage to the teeth was by the use of diamond corers. The problem with coring is the structure of the mammoth tooth itself: hard layers of enamel alternate with very soft layers of dentine or cement. For the drilling of the enamel the diamond corer has to be water-cooled otherwise the diamonds would become unfastened. It was found impossible to drill through a tooth without breaking the enamel layer off the organic layers and usually only a mix of broken pieces could be extracted. With such samples it was difficult to determine the beta dose rate exactly. Additionally, there was no "plug" of tooth material to seal the drilled hole after extraction.

The second technique tested was the use of a Well 6234 precision diamond wire saw (D.R. Bennett, Ltd). The kerf produced by the wire is very narrow, only about 0·5 mm. About a quarter of the tooth is cut at a very slow rate of about 1 cm/hour. The cut starts from the roots towards the top and then runs to the outside. This leaves the occlusal (chewing) surface intact, which is essential as this is the zoologically diagnostic part of the tooth. ESR samples can now be extracted with a hand held dentists' diamond drill (Figure 1). The two cut segments can be glued together without major visible damage to the exterior of the molar.

We hope that the application of this technique will allow in the future to date many Quaternary sites, which are presently undated because of the reluctance to sacrifice valuable museum pieces for the dating procedure.

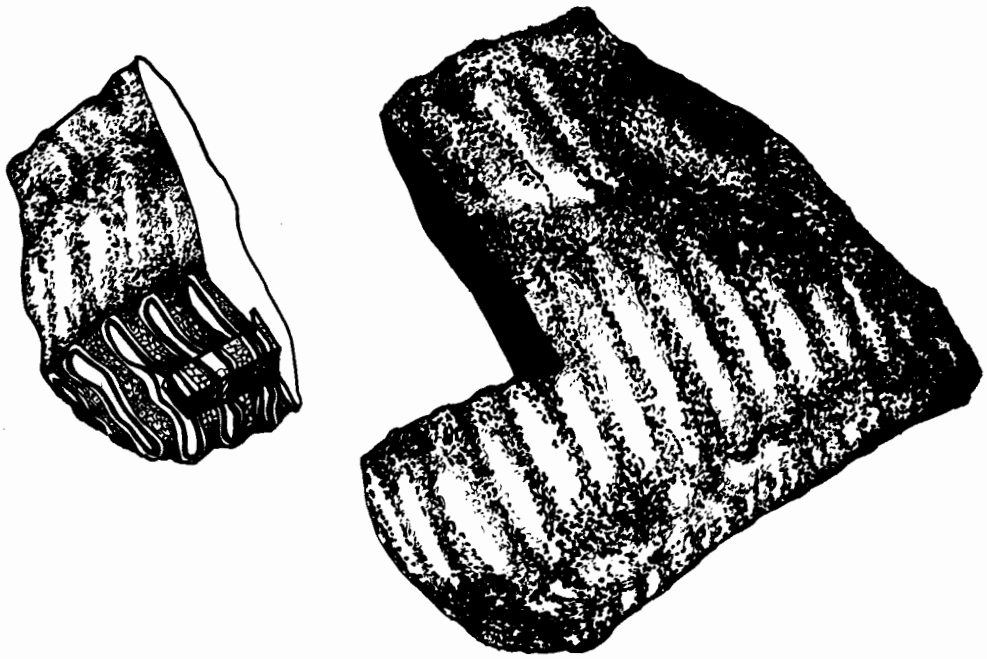

Figure 1.
Cut mammoth tooth. ESR samples were extracted from the top of the quarter.

Acknowledgement
We thank Dr. J. Cook, Schlumberger Research Centre, Cambridge, for his attempts to core some of our specimens and Mrs. F.M. Grün for drawing the figure.

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This is sound advice for the preparation of ESR samples from elephant teeth. The equipment described (Well precision diamond wire saw) is not, however, generally available and is a very expensive piece of equipment. A satisfactory substitute is a diamond-blade band-saw, such as can be found in shops which prepare decorative mirrors and glass table-tops, etc. We have found it possible to make use of such equipment for the same purpose. The kerf of the diamond band-saw is of the order of 1mm or less, and cutting does not cause splitting of the tooth.

Cautions on the use of extended duration preheats in the optical dating of quartz

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In an optical dating study of some Australian quartz extracts, we found that a post-irradiation preheat of 220°C for 5 minutes yielded the correct palaeodose (P), whereas longer, lower temperature preheats resulted in P underestimates. Young ages are obtained for near-modern samples preheated at 220°C and older samples yield optical dates in accordance with thermoluminescence (TL) dates and ^{14}C age determinations on associated charcoal. Thermal transfer of charge, from optically-insensitive traps to optically-sensitive traps, actuated by the 220°C preheat appears to have an insignificant effect on P. Measurements of optically-stimulated luminescence (OSL) and related 325°C and 110°C TL emissions following various preheats show that prolonged low-temperature preheats result in a dose-dependent increase in sample sensitivity, with a consequent decrease in P. The sensitivity change may be related to activation of the "pre-dose" mechanism (Zimmerman, 1971). In this paper, we present evidence pertaining to one such sample ($Ox_{OD}K166$), collected from an archaeological site in northern Australia.

Introduction

In the first demonstration of optical dating by Huntley *et al.* (1985), two irradiated quartz samples were given a "cut" heat to 250°C prior to laser stimulation in order to evict electrons from traps that were thermally unstable on the relevant geological timescale. A thermal treatment (termed the "preheat") of 220°C for 5 minutes was employed subsequently for quartz by Smith *et al.* (1986) and Rhodes (1988), and this preheat appears to be supported by good agreement between optical ages and independent age estimates (Rhodes, 1990; Smith *et al.*, 1990a, 1990b). As an alternative to high-temperature preheats, longer preheats at lower temperatures have been suggested (Godfrey-Smith *et al.*, 1988; Smith *et al.*, 1990b), such as a preheat of 160°C for 16 hours (Stokes, 1992).

Huntley *et al.* (1985) also recognised that preheating may induce the thermal transfer of charge from light-insensitive traps to light-sensitive traps. Rhodes (1988, 1990) has argued that thermal transfer occurs during sample burial and that the proportion of naturally transferred charge should be replicated by the laboratory preheat. Such equivalence cannot be demonstrated directly because charge transfer at ambient temperatures may take several millennia, so criteria such as zero ages for modern samples and agreement between optical ages and independent age estimates remain a necessity in optical dating.

Luminescence dating procedures

The luminescence sample discussed here ($Ox_{OD}K166$) was collected in 1989 by Roberts and Rhys Jones from an exposed section at the Nauwalabila I archaeological site in the seasonally wet tropics of northern Australia (Jones, 1985).

Quartz grains of 90-125 μm diameter were isolated in the usual manner (Aitken, 1985) and etched in 40% HF acid for 45 min. Aliquots of 5-6 mg quartz were spread over an area of ~0.4 cm^2 on stainless steel discs and the purity tested using infrared excitation (Spooner and Questiaux, 1989). Infrared-stimulated luminescence (IRSL) was detected but at an insignificant intensity (OSL:IRSL ratio $>10^5$). Aliquots were normalised on the basis of a 0.1 s exposure to an argon-ion laser (~12.5 mW cm^{-2} of 514.5 nm light). This "short-shine" normalisation depleted the optical dating signal negligibly (~0.5%).

The additive-dose method was adopted for TL and OSL palaeodose determinations. β irradiations were administered using an Elsec 9022 automated irradiator with a $^{90}Sr/^{90}Y$ source that delivered 3.32 ± 0.11 Gy

Table 1. OSL light sums for $Ox_{OD}K166$ naturals

Group	Preheat	Normalised light sum (mean ± std error)	Number of aliquots
1	none	1.000 ± 0.010	6
2	160°C, 16 h	1.086 ± 0.016	5
3	220°C, 5 min	0.935 ± 0.011	5

min^{-1} (at the 2σ level). This source was calibrated against the AEA Winfrith ^{60}Co source using an annealed portion of $Ox_{OD}K166$ (Roberts, Questiaux, Stoneham and Spooner, in prep.). A 20 h bleach under a UV-deficient sunlamp was used to define the residual level for the TL palaeodose determinations. TL aliquots were not preheated prior to readout whereas OSL aliquots were preheated, prior to final "shine-down", at 160°C for 16 h, 190°C for 1 h, 220°C for 5 min, 250°C for 35 s or 280°C for 5 s.

To determine the OSL palaeodose, preheated aliquots were exposed to the laser for 150 s and the UV-violet luminescence was detected by an EMI 9635Q PMT through Corning 7-51 and Schott BG-39 filters. OSL measurements were made using a modified Elsec 9010 unit and software (Spooner, 1993). Growth curves of OSL intensity (minus the background count rate) versus added dose were constructed for selected time intervals following commencement of laser exposure. The background count rate was calculated as the mean counts s^{-1} for the final 50 s and the P was determined from the OSL intensity integrated over the first 100 s of shine-down. Growth curves were fitted by a single saturating exponential, with each aliquot weighted by the inverse square of its luminescence intensity (Brumby, 1992).

TL measurements were performed on an automated Risø reader fitted with an EMI 9635Q PMT and Corning 7-59 and Chance-Pilkington HA-3 filters. Discs were heated at 5 K s^{-1} and saturating exponential growth curves were constructed (Brumby, 1992) for temperatures between 200°C and 500°C. The P is derived from the TL intensity integrated over the glow curve region that yielded a P plateau (270-430°C).

The environmental dose rate was deduced from high-resolution γ-ray spectrometry measurements of the activities of nuclides in the ^{238}U and ^{232}Th decay chains, and ^{40}K, in sediment samples collected from within a 30 cm radius of $Ox_{OD}K166$. The ^{238}U and ^{232}Th chains are consistent with a condition of secular equilibrium, as noted for similar deposits in the region (Roberts *et al.*, 1990; Roberts, 1991). Water contents were estimated from "as collected" moisture determinations and observations made while augering similar deposits during the tropical Wet and Dry seasons (Roberts, 1991). The internal α activity of the etched quartz extracts (determined by thick-source alpha counting) and the cosmic-ray dose rate (Prescott and Hutton, 1988) constitute ~5% and ~20% respectively of the total dose rate.

Preheat experiments on $Ox_{OD}K166$

We conducted four groups of experiments to investigate the effects of preheating. Aspects considered were the form of the OSL shine-down curve, thermal erosion of the TL glow curve, sensitisation of the 110°C TL peak, and thermal transfer of charge to the OSL source traps.

1. OSL shine-down curves

The "naturals" were split into three groups of 5-6 aliquots and each aliquot was short-shine normalised. One group was not preheated, a second group was preheated at 160°C for 16 h and the third group was preheated at 220°C for 5 min. The three groups were stored at room temperature for one day and then given a 150 s laser exposure. Table 1 contains the measured light sums (integrated over the first 100 s after background subtraction), normalised by the mean light sum of Group 1.

These results show that the OSL signal is eroded slightly (6.5%) by the 220°C preheat whereas the 160°C preheat induces an 8.6% increase. While the 220°C preheat result is consistent with expectations from thermal erosion of the OSL signal (Spooner, unpub. data), the 160°C preheat result suggests that sensitisation has occurred. This sensitisation may be as

much as 15% if both preheats initially eroded the OSL signal (i.e. the 325°C TL peak) to the same extent.

2. TL glow curves

We assessed the extent of thermal erosion of the TL, using three additional preheats to elucidate the relation between sensitisation and preheat temperature/duration. These preheats were derived from the assumption that preheats of 160°C for 16 h and 220°C for 5 min are "kinetically equivalent" (i.e. remove the same trapped charge population). E and S values for a hypothetical peak having a lifetime of 16 h at 160°C and 5 min at 220°C were calculated to be 1.61 eV and 1.00 x 10^{14} s^{-1} respectively ($\tau_{20°C}$ ~1.6 Ma). From these parameters, the three additional kinetically equivalent preheats were calculated to be 190°C for 1 h, 250°C for 35 s and 280°C for 5 s.

TL glow curves were obtained for six groups of four natural aliquots. Each aliquot was short-shine normalised: this method of normalisation differs from those usually employed for TL dating but is considered appropriate for quartz extracts whose glow curves are dominated by the easy-to-bleach 325°C peak (e.g. Ox_{OD}K166). Five groups of aliquots were preheated then stored at room temperature for one day, then all six groups were glowed out. To further remove disc-to-disc scatter, the mean glow curves for the preheated groups were scaled (by TL intensity) so that each overlay the mean glow curve for the unpreheated naturals over the temperature range unaffected by the preheats (350-400°C). Representative mean glow curves are shown in Fig. 1. At temperatures <300°C, the glow curve for the 190°C group overlies that of the 160°C group, while that of the 220°C group is indistinguishable from the glow curves for the 250°C and 280°C groups. Fig. 2 gives the light sums integrated over the TL peak temperature region (315-335°C) for all six groups, normalised by the mean light sum of the unpreheated naturals.

Two main conclusions stem from these results. First, all five preheats are equally effective at emptying the low-temperature (<300°C) TL traps (Fig. 1). The 160°C and 190°C preheats may be marginally more effective than the >200°C preheats but the difference is not statistically significant. We certainly do not observe the

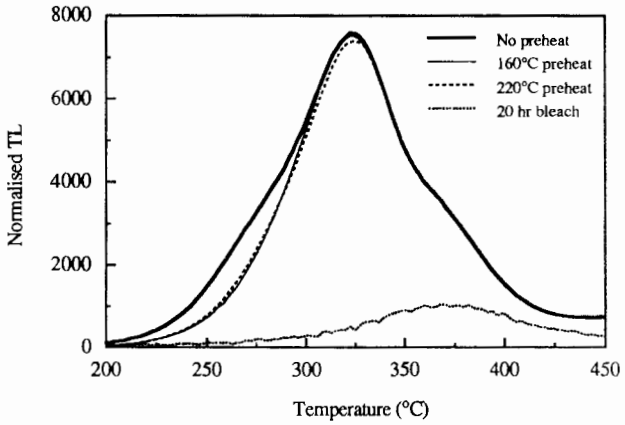

Figure 1.
Glow curves for natural (unpreheated), preheated and bleached aliquots of Ox_{OD}K166. Glow curves for aliquots preheated at 160°C for 16 h or at 220°C for 5 min are shown. At glow curve temperatures <300°C, the 160°C curve overlies the glow curve obtained for aliquots preheated at 190°C for 1 h, and the 220°C curve is superimposed by the glow curves obtained for aliquots preheated at either 250°C for 35 s or 280°C for 5 s. Natural aliquots were exposed to a UV-deficient sunlamp for 20 h to derive the bleached curve. Note the significant bleaching of the 280°C TL peak.

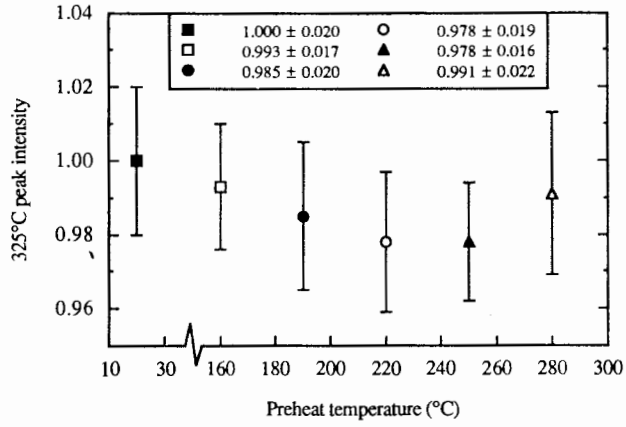

Figure 2.
TL intensity of the 325°C peak (315-335°C peak area integration) for natural and preheated aliquots of Ox_{OD}K166. The intensities are normalised by the mean intensity of the naturals, which were stored at room temperature (~20°C). The legend lists the mean normalised intensity ± standard error for each treatment. Note the ordinate scale.

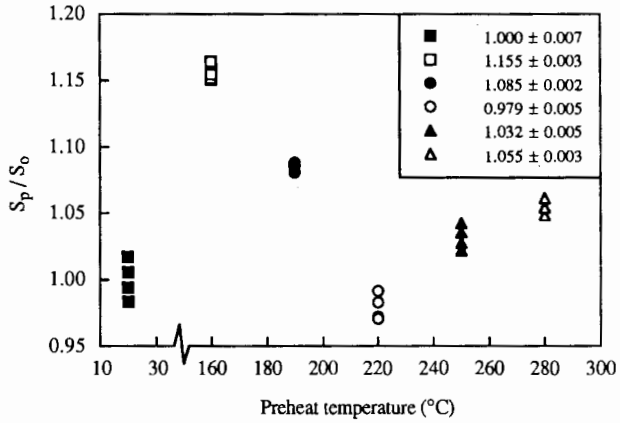

Figure 3.
Sensitivity of the 110°C TL peak (105-115°C peak area integration) for natural and preheated aliquots of Ox_{OD}K166. The sensitivities are expressed as a ratio of S_p (peak intensity after preheat) to S_o (peak intensity before preheat), normalised by the mean ratio of the naturals, which were stored at room temperature (~20°C). The legend lists the mean normalised S_p/S_o ratio ± standard error for each treatment.

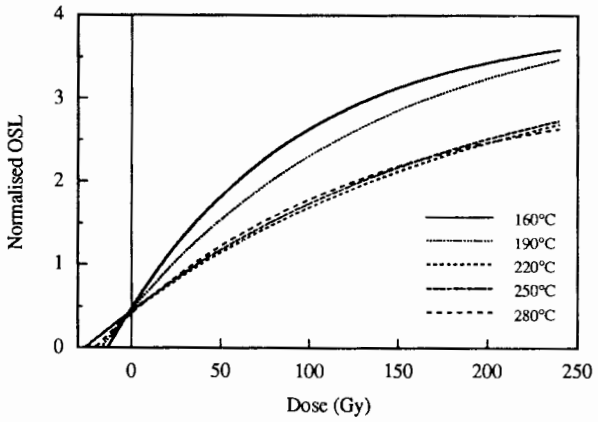

Figure 5.
OSL growth curves for preheated aliquots of Ox_{OD}K166. Each growth curve is fitted to eight naturals and four aliquots at each of six dosage levels (5, 10, 20, 60, 120 and 240 Gy) using a weighted saturating exponential function (Brumby, 1992). The OSL intensity for each aliquot is calculated from the light sum integrated over the first 100 s of laser exposure, minus the background count rate.

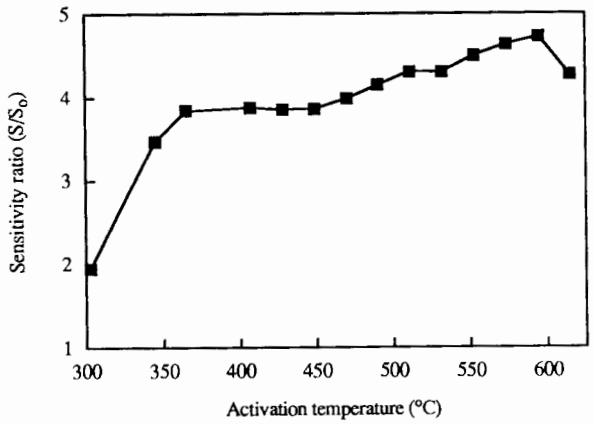

Figure 4.
Thermal activation characteristic (TAC) for Ox_{OD}K166, derived using a single natural aliquot and the multiple activation procedure (Aitken, 1985).

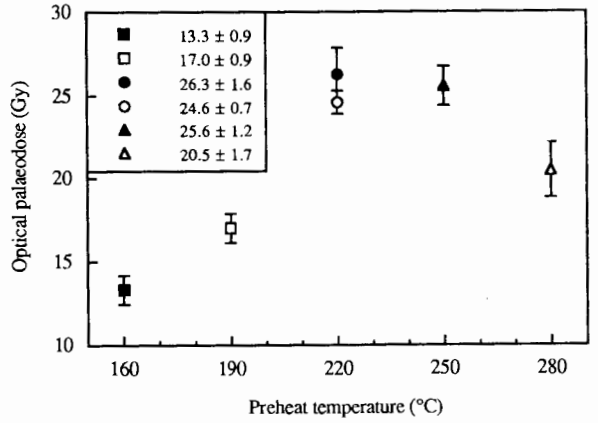

Figure 6.
Optical palaeodoses for preheated aliquots of Ox_{OD}K166 derived from the growth curve fits in Fig. 5. The legend lists the mean palaeodose ± standard error for each treatment, along with the palaeodose (24.6 ± 0.7 Gy) from a subsequent trial using 52 aliquots and the 220°C preheat. The TL palaeodose for this sample is 23.1 ± 1.0 Gy.

major differences reported by Stokes (1992) and our glow curves are sufficiently reproducible (± 2% standard error after short-shine normalisation) that any such discrepancies would be apparent. In contrast, we note that the 280°C peak is erased by all five preheats, in accord with the findings of Smith *et al.* (1986) and Rhodes (1990). Note that the 280°C peak should be removed prior to laser stimulation because it is light-sensitive (see the bleached glow curve in Fig. 1) and thermally unstable (with a lifetime of ~300 ka at 20°C; Spooner, unpub. data).

Second, the mean values shown in Fig. 2 suggest that the 325°C TL peak is sensitised by the 160°C preheat, and to a lesser extent by the 190°C and 280°C preheats, with respect to the 220°C preheat. In itself, this information is not conclusive because the mean values are indistinguishable at the 1σ level. However, we note that the mean trend for the preheated aliquots mimics the sensitisations reported below for the 110°C TL peak (Fig. 3) and the OSL signal (Fig. 6). The TL sensitisation induced by the 160°C preheat is less marked than that suggested by OSL (Table 1), presumably because OSL is a "pure" measure of the trapped charge population associated with the 325°C TL peak whereas the 325°C TL in Fig. 1 contains a major proportion of TL from the overlapping 375°C peak. This will reduce the apparent TL sensitisation of the 325°C peak unless the individual peaks are isolated

3. 110°C TL peak sensitivity

In an attempt to circumvent the problem of the overlapping 325°C and 375°C TL peaks, we examined the behaviour of the 110°C TL peak. A relationship between the 110°C and 325°C TL peaks has been postulated by Wintle (1974). The 110°C peak and OSL emission are also believed to be related, sharing the same recombination centre(s) because both signals emit strongly at 360-380 nm (Zimmerman, 1971; Bailiff, 1979; Akber *et al.*, 1988; Huntley *et al.*, 1991) and showing similar dose-dependent sensitivity changes (Aitken and Smith, 1988; Stoneham and Stokes, 1991).

In this experiment, 24 natural aliquots of $Ox_{OD}K166$ were divided into six groups of four aliquots. Each aliquot was heated to 180°C at 5 K s^{-1} (to erase any low-temperature TL), allowed to cool to room temperature, given a test dose of 0.1 Gy, then immediately reheated to 180°C at 5 K s^{-1}. The TL intensity of the 110°C peak (105-115°C peak area integration) was determined from this measurement and represents the initial sensitivity (S_o) of the sample. Five groups were then preheated at 160°C, 190°C, 220°C, 250°C and 280°C (durations as previously); the sixth group remained unpreheated as a control. Following the preheats, the 110°C peak intensities (S_p) of all groups were measured. The S_p/S_o ratios, normalised by the mean ratio of the unpreheated aliquots, are shown in Fig. 3.

The 110°C peak data demonstrate that all preheats, except the 220°C preheat, induce a sensitivity increase. While the sensitivity increase obtained using the 250°C and 280°C preheats may be related simply to activation of the pre-dose mechanism, as inferred from the thermal activation characteristic (TAC) of this sample (Fig. 4), the sensitivity increases induced by the 160°C and 190°C preheats are postulated as resulting from a time-dependent activation of the pre-dose mechanism (Questiaux, in prep.).

For the preheated aliquots, the sensitivity changes observed for the 110°C peak (Fig. 3) show a similar trend to those attained using the 325°C TL peak (Fig. 2). The magnitude of the 325°C peak changes are less than those associated with the 110°C peak because the former are reduced by the TL contribution from the overlapping 375°C peak. In contrast, the 110°C peak and OSL signal exhibit preheat-related sensitivity changes that are, within experimental error, identical in magnitude. The ratio of 160°C to 220°C normalised OSL light sums (Table 1) is 1.161 ± 0.022, compared to 1.180 ± 0.007 for the corresponding normalised 110°C peak intensities (Fig. 3).

4. Thermal transfer of charge

We also investigated the possibility that the 220°C preheat induced a substantial thermal transfer of charge from light-insensitive to light-sensitive traps. Such a mechanism has been invoked to explain the substantial post-preheat luminescence signal generated in modern Canadian quartz (Godfrey-Smith *et al.*, 1988). However, modern samples from Europe and Mali yielded low count rates (and small P) following a preheat of 220°C for 5 min (Stokes and Rhodes, 1989; Rhodes, 1990). Three lines of evidence suggest that thermal transfer is insignificant in the samples collected from this site.

First, as noted above, preheating the $Ox_{OD}K166$ naturals at 220°C for 5 min caused a decrease in OSL intensity (consistent with thermal erosion of part of the dating signal) and not an increase, as might be expected if thermal transfer was significant.

Second, a sample collected from a depth of 1-6 cm at this site ($Ox_{OD}K171$) yielded an optical palaeodose (0.16 ± 0.03 Gy) and age (290 ± 60 a) in accord with the rate of sediment accumulation (deduced from the late Holocene ^{14}C chronology, ethnographic testimony and archaeological evidence such as the appearance of glass fragments; Jones, 1985, p. 182). Note that $Ox_{OD}K171$ is not a surface sample, so the P includes not only any thermal transfer and "recuperation" (Aitken and Smith, 1988) components but also a small dose from the surrounding environmental radiation field and cosmic rays.

The third, and most direct, indication of negligible thermal transfer was obtained from a portion of $Ox_{OD}K166$ subjected to the regenerative method of palaeodose determination. Natural aliquots of $Ox_{OD}K166$ were short-shine normalised and then bleached for 20 hours. Six bleached aliquots were kept aside while others were irradiated; all aliquots were then preheated at 220°C for 5 min, stored at room temperature for one day and then shone-down. To determine the extent of thermal transfer in each aliquot, the OSL intensity integrated over the first second of shine-down (minus the background count rate) is compared with the corresponding 0.1 s short-shine normalisation (multiplied by ten, to approximate the unpreheated OSL integral for the first second of laser exposure). Table 2 lists the ratios (mean ± standard error) for the aliquots that were bleached then preheated, and the aliquots that were bleached, irradiated (11.2 Gy) then preheated. The amount of charge transfer is equivalent to a dose of 0.014 Gy (i.e. (0.112/91.8) x 11.2 Gy), which is trivial compared to the sample P (~24 Gy).

Palaeodose determinations on $Ox_{OD}K166$

The experiments discussed above indicate that different preheats sensitise to varying degrees the OSL dating signal in natural aliquots of $Ox_{OD}K166$. We now describe the results of P determinations for $Ox_{OD}K166$ which show that:

1) the sensitisation is dose-dependent,
2) only the 220°C and 250°C preheats yield optical P in agreement with the TL palaeodose,
3) only the 220°C and 250°C preheats yield optical ages that concur with ^{14}C ages.

The values of P were determined using the procedures described above. Fig. 5 shows the growth curves for the variously preheated aliquots, where each growth curve is fitted to eight naturals and four aliquots at each dosage level. Fig. 6 gives the corresponding P determinations.

We note that the growth curves for the 220°C, 250°C and 280°C preheated aliquots are similar in shape (Fig. 5). The most likely explanation for the non-superposition of the growth curves for the 160°C and 190°C preheats is that the sensitisation (that has only a small effect on the natural OSL signal) is enhanced by the addition of larger doses, up to ten times the value of P. However, the effect is also significant at lower added doses. In any case, the sensitivity changes caused by the 160°C and 190°C preheats are dose-dependent. Although the 280°C preheat appears to cause a slight dose-dependent increase in sensitivity, the effect is much smaller than observed for the 160°C and 190°C preheats. All these sensitivity increases may result from thermal activation of the pre-dose mechanism, as discussed above.

Table 2. Thermal transfer in $Ox_{OD}K166$ aliquots

Treatment	1 s shine-down/ 0.1 s short-shine x 10 (counts s^{-1} g^{-1})	Number of aliquots
Bleach & preheat	0.112 ± 0.006	6
Bleach, irradiate & preheat	91.8 ± 1.9	6

The TL palaeodose for this sample (23.1 ± 1.0 Gy) compares favourably with the optical P yielded by the 220°C and 250°C preheats (Fig. 6). It corresponds especially closely with the P obtained subsequently using the 220°C preheat and 52 aliquots (24.6 ± 0.7 Gy); this P is used to calculate the optical age. The latter P and the TL paleodose also agree well with the optical P obtained using the regenerative procedure and the 220°C preheat (23.5 ± 0.2 Gy, where the error term reflects only the reproducibility of the naturals). The 220°C preheat optical age (30.0 ± 2.4 ka) is therefore similar to the TL age (28.1 ± 2.4 ka) because the same dose rate is used in both age determinations. In contrast, the optical P obtained using the 160°C and 190°C preheats are smaller than the TL palaeodose by ~10 Gy and ~6 Gy respectively (Fig. 6); the corresponding ages are 16.2 ± 1.6 ka and 20.7 ± 1.9 ka respectively.

Independent support for the validity of the luminescence dates obtained using the 220°C and 250°C preheats is provided by their concordance with the ^{14}C chronology at this site. A suite of 16 charcoal samples have been dated by ^{14}C and these show a coherent pattern of increasing age and decreasing charcoal mass with depth (Jones, 1985, figs. 9.12 and 9.13; Roberts, unpub. data), consistent with stratigraphic integrity and chemical weathering of the charcoal in a tropical climate. Charcoal was collected from the same pit and level as $Ox_{OD}K166$ during an excavation by Rhys Jones in 1981. This sample (ANU-3182) was dated by ^{14}C to 22,840 ± 520 BP, which corresponds to a calendar year age of ~27 ka (calibrated according to Bard *et al.*, 1993). This compares favourably with the TL age and the 220°C and 250°C preheat optical ages. In contrast, the 160°C and 190°C preheat optical ages are erroneously young with respect to both the TL and ^{14}C ages. Contamination of the ^{14}C samples by "old carbon" is unlikely in this sandstone region and no charcoal was recovered from the underlying (i.e. older) levels. Nor is there any evidence for stratigraphic disturbance or artefact displacement.

Conclusions

Examination of quartz extracts from northern Australia has revealed a dose-dependent sensitisation of the OSL signal following extended duration preheats. Here we have reported evidence for one such sample. In the worst instance, a preheat of 160°C for 16 h yielded an optical age only ~60% of the age obtained by TL dating of the same sample and by ^{14}C dating of associated charcoal. In contrast, a preheat of 220°C for 5 min yielded the correct optical palaeodose and age. The 220°C preheat also induced negligible transfer of charge from light-insensitive to light-sensitive traps. OSL sensitivity changes are accordant with those of the 110°C TL peak and imply the involvement of the pre-dose effect.

At present, the charge-transfer processes and luminescence mechanisms affected by preheating are not fully understood. We caution readers that, in this study, a preheat of 160°C for 16 h caused a severe underestimation of the palaeodose, and advise that prolonged low-temperature (<200°C) preheats should be avoided. However, the correct P was obtained using instead a preheat of 220°C for 5 min; others have reported similar success (Rhodes, 1990; Smith *et al.*, 1990a, 1990b). We consequently recommend use of a preheat of 220°C for 5 minutes, the comparison of optical and TL palaeodoses, and the corroboration of optical ages with independent age estimates.

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Compiled by Ann Wntle

Letters

° *From G. Hutt and I. Jaek , Institute of Geology, Estonian Academy of Science, EE0105 Tallinn, Estonia.*
The effect of shallow traps: a possible source of error in TL dating of sediments. *Ancient TL*, 10(2) 1992.

Comments to dating technique, proposed by Shlukov and Shakovets (1987).

1. The technique is based on a number of assumptions which may not be well founded:

1(a). Sediments of different genesis, laid with different rates of sedimentation, where the residual may be far from being in minimum.

1(b). Palaeodose reconstruction on the basis of minerals which have undergone strong light bleaching, especially in the case of quartz, is not valid because of sensitivity changes.

1(c). Correlation with ^{14}C dates is only seldom possible. So this technique may give only stratigrafic information of homogeneous profiles, but not absolute dates.

2. Concerning laboratory calibration procedures we would like to make the following remarks:

2(a). Trap competition is well-known. Deeper traps commonly provide greater competition and the effective cross section of traps is the result of the charge and quantity of defects. Moreover deep traps are usually the first to saturate.

If shallow traps can provide greater competition, which we doubt, deep traps will reach their saturation level at a considerably slower rate (see fig.1, of paper).

2(b). The decrease of the equilibrium level of charge carriers concentration (fig. 2 in paper) is plausible only if special factors apply, including the following:

- fading at elevated temperature of irradiation.
- radiation fading or bleaching as a result of complicated interaction of radiative factors with mineral defects: formation of different channels of recombination, causing loss of information. At elevated temperature, thermally dependent processes may exist. For example, some centres that were are an excited state after stimulation may need

additional thermal activation energy to be included in the recombination processes (like "infrared" mechanism of optical response).

- quenching, caused by hole recombination processes: at elevated temperature holes may be released from valence band or from shallow hole traps with the following recombination at electron deep traps. All these mechanisms (and others) may be responsible for the experimental results obtained in the paper. A more specific model needs additional experimentals results.

The conclusion concerning the simulation of natural processes at elevated temperature is quite questionable, because a lot of possible thermally dependent effects may also be activated.

Reply by Vagn Mejdahl

I am grateful to Ian Bailiff for giving me the opportunity of an immediate reply to the comments by G. Hütt and I. Jaek. I shall reply to the various points in turn.

1(a). Incomplete zeroing. This objection can be raised against all luminescence dating methods except those designed especially to cope with partially bleached sediments. The situation is no different from that applying to the added dose technique which I believe GH and IJ are using. The technique of Shlukov et al. can be used for partially bleached sediments provided that an estimate of the residual dose at the time of deposition can be obtained.

1(b). I am not sure what is meant by "strong light bleaching", but it can hardly be relevant for Shlukov's technique. Unlike the commonly used regeneration technique, Shlukov's method uses no laboratory bleaching; therefore, the only bleaching involved is that occurring in nature and this is common for all luminescence dating techniques.

1(c). Provided that the TL growth curve for a particular mineral from a certain region can be approximated by a single exponential function with its two parameters then only one ^{14}C date is needed for each region, the other parameter being the saturation level.

I think it might be well worth while to look into Shlukov's method, perhaps using feldspars, because it avoids a number of problems, including those of long-term thermal fading and the possible effect of dose rate differences in nature and in laboratory irradiation.

2(a). I am not sure which dosimetry practice GH and IJ are referring to and I find it difficult to see the relevance for the minerals used in dating. My opinion is that if one wants to know the properties of say K-feldspars, one has to study K-feldspars. Doing this it is easy to verify that the signal relating to the 150°C peak reaches saturation at a lower dose than those relating to the peaks at 220 and 320°C (heating rate 8°C/s).

It may be true that trap competition is a well-known fact, but I know of only two earlier studies that have addressed the particular problem associated with the presence of a shallow trap: Aitken et al. (1974) and Wintle and Packman (1988) and these seem to have gone largely unnoticed. I think, therefore, that Shlukov et al. (1992) are to be congratulated for having pointed out this potential problem so clearly.

2(b). I agree that a number of thermally conditioned processes might occur as a result of the procedure described by VM et al., but the main point is that the response of the 320°C peak in K-feldspar is not affected by merely heating the sample to 130°C; this was verified by heating natural and laboratory irradiated samples at 130°C in an oven for the length of time required for irradiation (a few minutes).

I might add that preheating, which is commonly used in TL and OSL dating, would not be possible if the heating caused adverse effects. We use preheating at 290°C for 10 s for TL dating of archaeological samples and 260°C for 100 s for IRSL. Duller (1991) used a preheat of 260°C for 10 min for OSL dating.

I agree with GH and IJ that more studies might be required, but from a practical point of view the main thing is that the procedure (heating to 130°C during laboratory irradiation) seems to work and removes the underestimates seen in earlier studies, e.g. Kronborg and Mejdahl (1989).