A cautionary note: apparent sensitivity change resulting from curve fitting

Sheng-Hua Li
Institute of Earth Studies, University College of Wales, Aberystwyth SY23 3DB, U.K.

Introduction

Curve fitting procedures are widely used in TL dating studies (Berger and Huntley, 1989; Mejdahl, 1985). In particular a saturating exponential curve has been applied over a wide dose range when the TL signal does not grow linearly. Exponential fitting has been used for samples of either pure or mixed minerals. The form of exponential curve can be written as

$$I = A - B e^{-CD}$$

Where A, B and C are fitting parameters, and D is the laboratory applied dose (Gy). The characteristics of the curve are governed by factors A and C, which relate to the asymptotic TL level and the saturation dose. If we assume no residual TL signal, then A = B.

In this paper I explore the effect of applying simple exponential fitting to the combined computer generated TL signals from two hypothetical minerals, each of which has different values of A and C.

Data used

Let us assume that for one mineral the TL growth curve is given by

$$I_1 = 443 - 443\, e^{-0.00268\, D}$$

where parameters $A_1 = B_1 = 443$ and $C_1 = 0.00268$ were used (curve 1 in figure 1). Now let us suppose that the TL of the other mineral saturates at a lower dose and that the asymptotic TL level is half that of the other mineral, i.e. $A_2 = B_2 = 221.5$; $C_2 = 0.0134$, then the TL growth curve of this mineral is given by

$$I_2 = 221.5 - 221.5\, e^{-0.0134\, D}$$

This is shown as curve 2 in figure 1.

The TL growth curve for a sample made up of equal weights of the two minerals is $I = I_1 + I_2$, curve1+2 in figure 1.

Let us now consider the ED determination for such a sample which has received 300 Gy. Application of an exponential fitting program to the TL for the 300 Gy point and the TL responses for the five higher doses used as an "additive dose set" produced an ED of 428 Gy, 128 Gy higher than expected. Shifting the combined curve1+2 by 300 Gy indicated that a significant sensitivity change would be observed between the regeneration and the additive dose curves (figure 1).

A similar fitting was applied for the TL signal resulting from 20 Gy and using 6 added doses up to 500 Gy. An ED of 23 Gy is given from the exponential fit, which is only 10% larger than that expected. The apparent sensitivity change is negligible (figure 2).

If the two minerals are assumed to have a similar saturation dose, no significant difference is found between the ED given by curve fitting and the expected ED. This suggests that the relative saturation doses of the minerals producing the TL signals is important in the application of exponential curve fitting.

Implications for polymineral samples

For a polymineral sample, such as 4-11 µm grains used in many TL laboratories, the saturation doses may be different for each mineral. Feldspar and quartz have been shown to have significantly different saturation doses (Mejdahl, 1985). Although real polymineral samples may not behave in exactly the same way as the above examples, use of a saturating exponential fit could result in an apparent sensitivity change when the additive dose and regeneration curves are compared.

Acknowledgment

The author would like to thank Dr. A.G. Wintle for correcting his English.

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PI: Reviewer's Comments (S.W.S McKeever)

This note illustrates one of the dangers of curve fitting additive dose growth curves using an assumed single exponential function for the growth. The problem, as described by the author, need not be confined to the situation where one is dealing with a polymineralic sample, however. A TL signal made up of two unresolved, overlapping components, each from the same mineral but displaying different growth curves and saturation levels, may present the same difficulties.

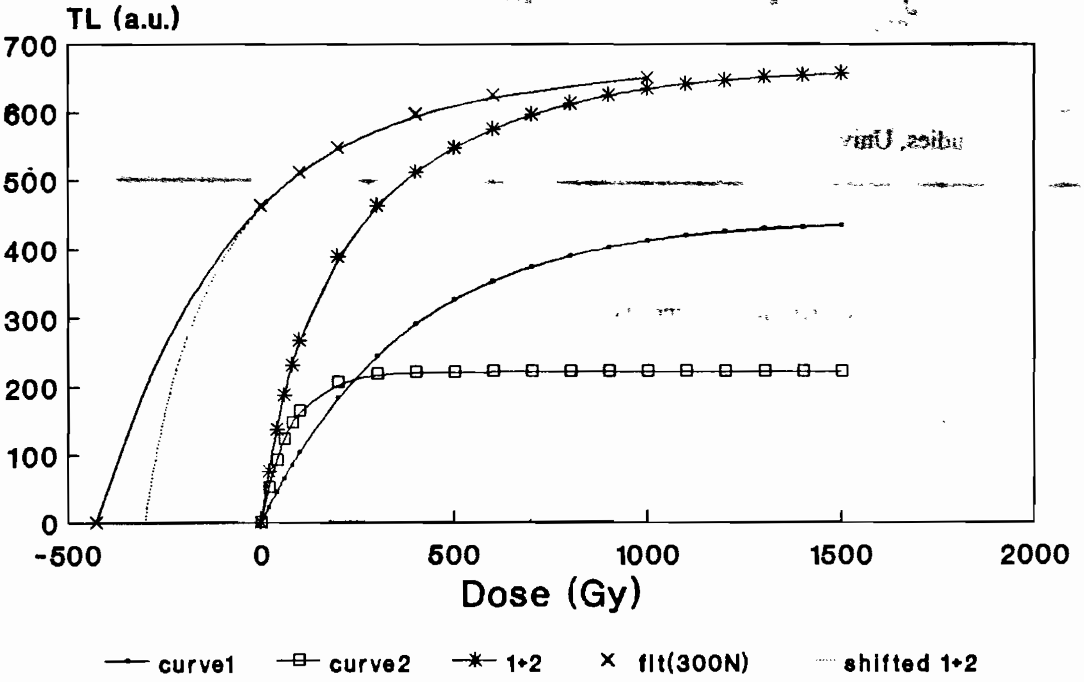

Figure 1.
Calculated TL growth curves.
(•) growth curve 1: equation I_1 (see text), (□) growth curve 2: equation I_2, (*) curve1+2, (x) saturating exponential fitting of curve1+2, 300 Gy as the natural and using added doses up to 1000 Gy, (...) shifted curve1+2.

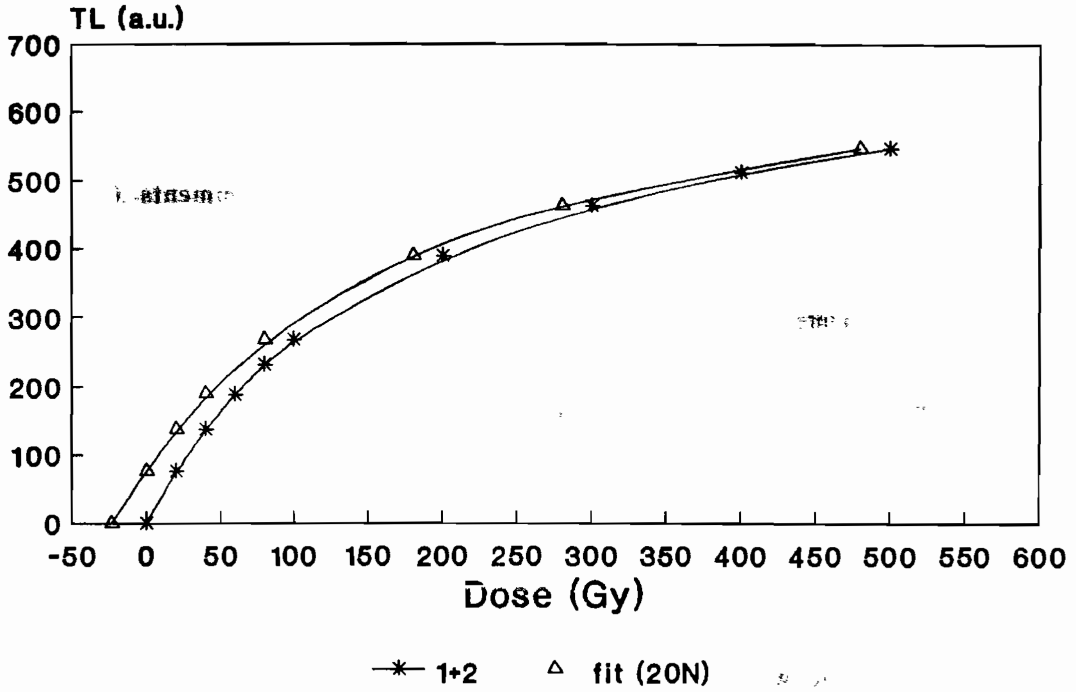

Figure 2.
Calculated TL growth curves. Parameters are the same as in figure 1, but taking 20 Gy as the theoretical natural point and using added doses up to 500 Gy.

ESR behaviour of the paramagnetic centre at g=2.0018 in tooth enamel

Edward J. Rhodes and Rainer Grün
Subdept. of Quaternary Research, Cambridge University, Free School Lane, Cambridge CB2 3RS, U.K.

Introduction

The dating of mammal teeth from archaeological and geological sites has played an important role in both the field of human evolution (Grün and Stringer, 1991) and in assigning oxygen isotope stages to Quaternary terrestrial sequences (eg Zymela et al., 1988). Although many ESR age estimates have been published using the signal at g=2.0018 in tooth enamel, no systematic study has been carried out with respect to optimizing the measurement conditions for AD determination.

One problem of using the signal at g=2.0018 for AD determination is the correct definition of the signal. In many cases this signal, which has a line width of about 0.35 mT, is superimposed by narrower signals which have been attributed to various organic substances (Ikeya, 1982; Grün, 1985; Grün et al., 1987; Bouchez et al., 1988; see figure 1). Based on some coarse plateau-tests, Grün et al., (1987) suggested recording the signal with a modulation amplitude of 0.5 mTpp (see figure 2a).

Hoshi et al. (1985) studied the saturation characteristics of tooth enamel at room temperature, showing that microwave saturation occurs at powers of greater than about 1 mW. However, no study of the influence of measurement power on the size of systematic or random deviations in the AD determination has been undertaken.

In this study measurements have been conducted to test the influence of modulation amplitude, temperature and microwave power on the AD determination. The aims of this study were to (i) confirm the validity of previously measured dates and (ii) determine the optimum conditions for AD measurement.

We selected two enamel samples (529a and 533a, horse) that had been previously used in a dating study of Border Cave (Grün et al. 1990). Sample 529a shows very strong interferences by organic radicals whereas these are almost invisible in the ESR spectrum of sample 533a (see figure 1). The ESR measurements for this study were carried out approximately 2 years after the initial measurements of Grün et al. (1990) who used the measurement parameters as recommended by Grün (1989a,b): 2 mW microwave power, 0.5 mTpp modulation amplitude, room temperature. The AD values were determined by exponential fitting using a jackknifing procedure for error estimation (Grün & Macdonald 1989). The previously published AD values were: 68.3 ± 11.8 Gy (529a) and 74.5 ± 4.9 Gy (533a).

All measurements included in this study were performed on a JEOL JES RE1X ESR spectrometer equipped with a ES DVT2 variable temperature unit.

Results

The organic radicals can be suppressed by using a large modulation amplitude (figure 2a), increasing microwave power (figure 2b), or lower temperatures (figure 2c). Using lowered temperatures (in 10 ± 0.5 °C steps) we observed that the sharp organic interferences disappear at about -80 °C. We therefore decided to study the influence of microwave power on AD determination at room temperature and at -100 °C.

Figure 3 and table 1 summarize the results of this study. The first column of figure 3 shows that the samples show microwave saturation effects above 1 mW under any set of measurement conditions. This agrees with the observation of Hoshi et al. (1985) and seems to be intrinsically characteristic for the paramagnetic centre at g=2.0018. Note, that the background increases significantly above 10 mW.

The second and third columns of figure 3 show that the AD determination does not seem to be systematically influenced by microwave saturation. Only the AD determination for sample 529a, measured with a low modulation amplitude of 0.05 mTpp at room temperature shows a trend towards smaller AD values with increasing microwave power. However, this is more likely due to the poor signal definition under these measurement conditions than the effect of microwave power. Some scattering in the mean AD value can be observed in the results of sample 529a at -100 °C with 0.05 mTpp, although there is no systematic trend. All other determinations of the mean AD value seem to be completely independent of temperature, microwave saturation and modulation amplitude.

Figure 4 shows a plot of the signal-to-noise (s/n) ratio versus the error in AD estimation. The s/n ratio is highest in the region of 10 mW. However, the minimum fractional error does not consistently coincide with the optimum s/n ratio; best results in AD determination seem to be obtainable for microwave powers in the range of 1 to 10 mW.

Although the low temperature measurements suppress the organic signals very effectively, the size of the errors do not systematically decrease.

Discussion

Figure 2 shows that the organic interferences of the signal at g=2.0018 can in principle be suppressed either with large modulation amplitudes, increasing microwave powers or measurement at lower temperatures. Measurements at very high microwave powers have the disadvantage of increasing the error in the AD determination which seems to result from a worse s/n ratio.

The basic improvement for sample 529a, which contains the organic interferences, is the measurement with a 0.5 mTpp modulation amplitude.

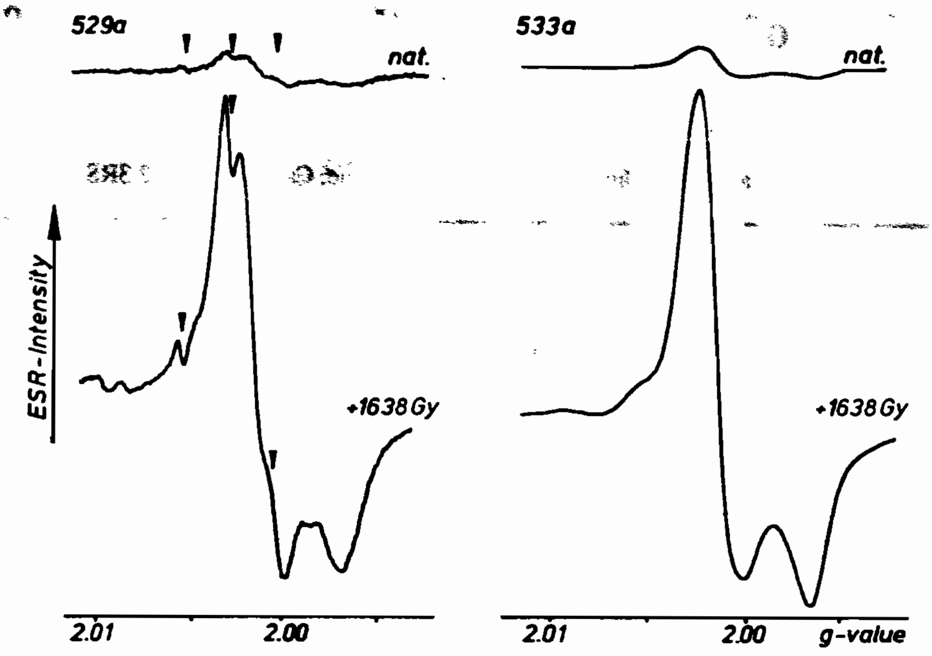

Figure 1.
ESR spectra of samples 529a and 533a (0.05 mTpp, 2 mW, 20 °C). Upper spectrum: natural sample; lower spectrum: +1638 Gy. The arrows indicate the interferences by organic radicals.

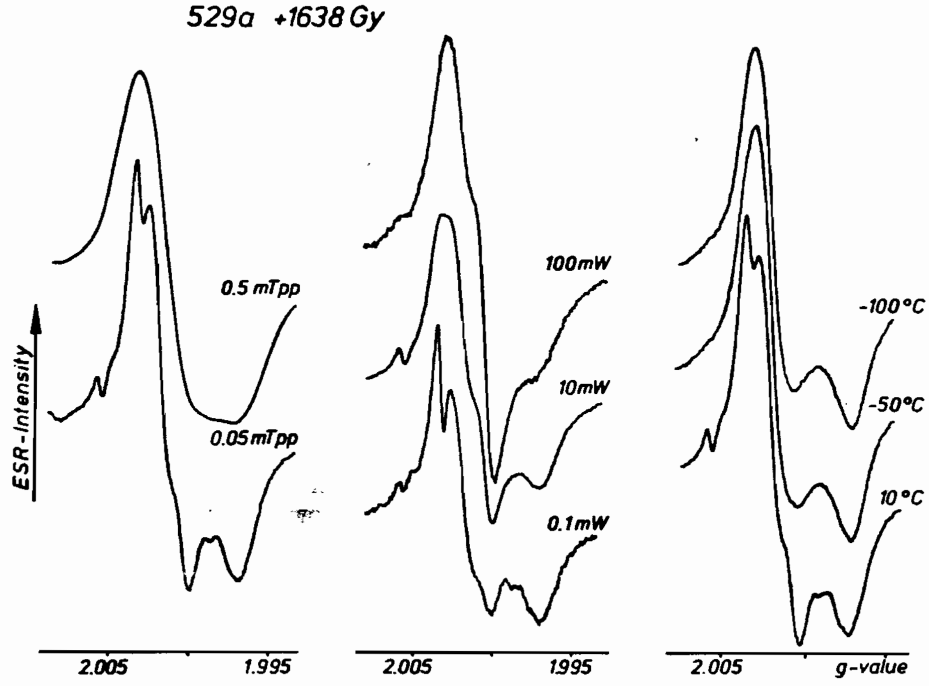

Figure 2.
(a) influence of modulation amplitude (+1638 Gy, 2 mW, 20 °C); (b) influence of microwave power (0.05 mTpp, 20 °C, +1638 Gy); (c) influence of temperature (0.05 mTpp, 2 mW, +1638 Gy).

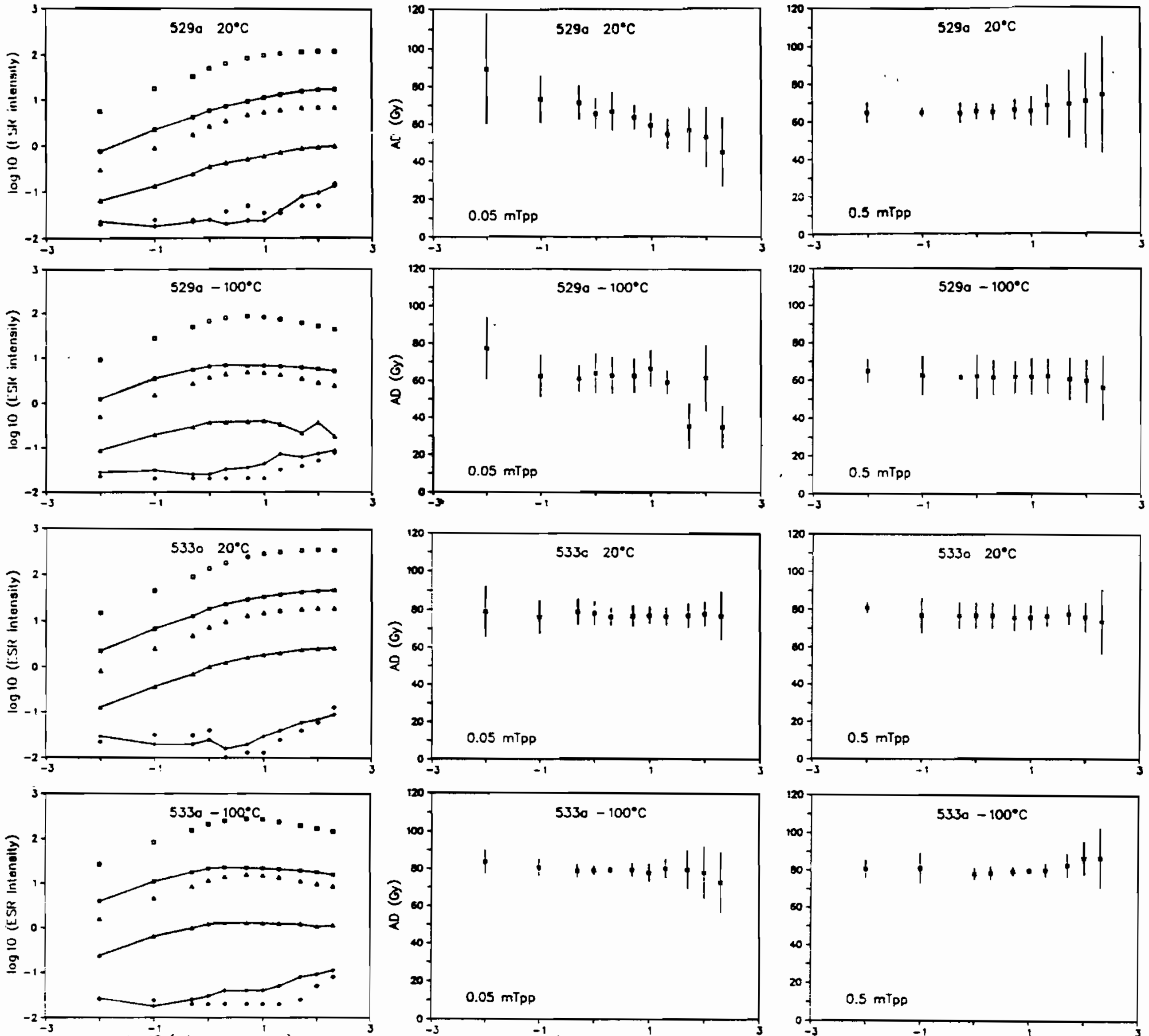

Figure 3.

Left column: signal and noise intensities versus microwave power.

Open symbols: 0.5 mTpp; closed symbols (joined by lines): 0.05 mTpp. Diamonds: background; triangles: natural sample; squares: +1638 Gy. Middle column: AD versus microwave power (0.05 mTpp modulation amplitude). Right column: AD versus microwave power (0.5 mTpp modulation amplitude).

Measurements at low temperatures do not seem beneficial for the AD determination of sample 529a, but show some improvement for sample 533a. Although it seems desirable to measure at temperatures around -100 °C, most ESR spectrometers are not equipped with a temperature unit that can keep this temperature very stable and the measurements take a considerably longer time since the equipment has to get into thermal equilibrium for each measurement.

A side-effect of this study is that we cannot observe anomalous fading of the signal at g=2.0018 after two years.

Conclusion

The optimal ESR measurement conditions for AD determination using the paramagnetic centre at g=2.0018 in tooth enamel are at room temperature at around 1 to 10 mW microwave power and a modulation amplitude of 0.5 mTpp. Measurements at -100 °C, which suppresses the organic signals very effectively, do not seem to provide significant advantages. We cannot observe an anomalous fading component after about two years.

Our results confirm the validity of the measurement conditions (and age estimates) that have been suggested by Grün (1989a,b).

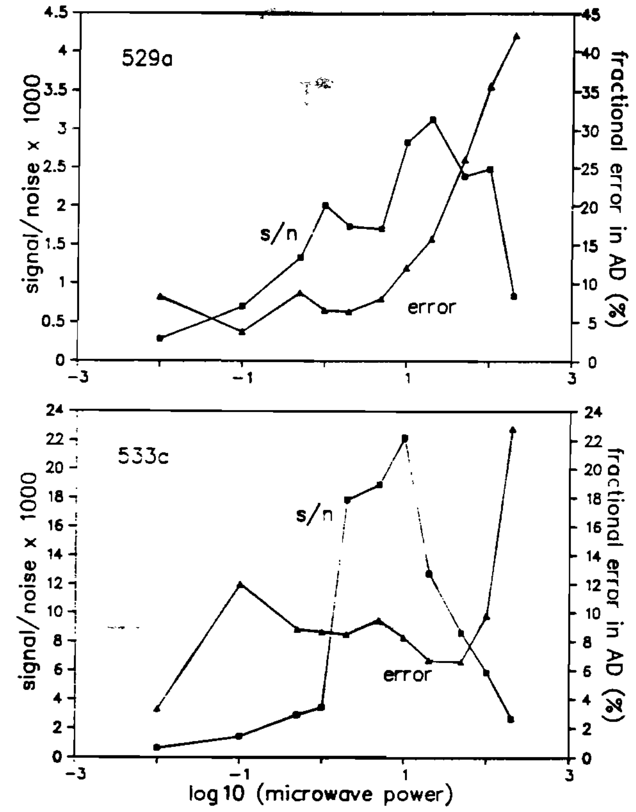

Figure 4.
Signal-to-noise (s/n) ratio and fractional error versus microwave power for 529 and 533 (20 °C, 0.5 mTpp).

Table 1.

i) sample 529a

microwave power(mW)	20 °C 0.05 mTpp	-100 °C 0.05 mTpp	20 °C 0.5 mTpp	-100 °C 0.5 mTpp	Grün et al. (1990)
200	44.9 ± 18.0	34.8 ± 10.8	73.7 ± 31.1	55.9 ± 16.8	
100	52.9 ± 15.7	61.4 ± 17.5	70.4 ± 25.1	59.5 ± 11.3	
50	56.2 ± 11.1	35.2 ± 12.2	68.6 ± 17.9	60.5 ± 11.0	
20	54.4 ± 8.2	59.0 ± 6.3	68.0 ± 10.8	62.1 ± 9.0	
10	59.1 ± 6.3	66.4 ± 9.5	65.0 ± 7.8	61.8 ± 9.3	
5	63.6 ± 6.4	62.6 ± 9.0	65.8 ± 5.3	61.7 ± 8.2	
2	66.8 ± 10.0	62.8 ± 9.6	64.8 ± 4.1	61.6 ± 9.1	68.3 ± 11.8
1	65.7 ± 8.3	64.1 ± 10.5	65.0 ± 4.2	62.0 ± 11.3	
0.5	71.6 ± 9.0	61.2 ± 7.0	64.3 ± 5.6	61.4 ± 1.3	
0.1	73.2 ± 12.2	62.2 ± 11.0	64.9 ± 2.4	62.3 ± 10.0	
0.01	89.3 ± 28.7	77.1 ± 16.5	65.3 ± 5.4	64.8 ± 6.1	
means	63.4	58.8	66.9	61.2	
scatter in means	12.1	12.7	3.0	2.2	

ii) sample 533a

microwave power(mW)	20 °C 0.05 mTpp	-100 °C 0.05 mTpp	20 °C 0.5 mTpp	-100 °C 0.5 mTpp	Grün et al. (1990)
200	76.7 ± 12.5	72.7 ± 16.0	73.5 ± 16.8	86.9 ± 15.7	
100	77.8 ± 6.0	78.1 ± 13.8	75.8 ± 7.4	86.9 ± 8.7	
50	76.9 ± 6.3	79.7 ± 10.2	77.4 ± 5.1	82.9 ± 6.4	
20	76.5 ± 4.5	80.1 ± 4.9	76.4 ± 5.1	80.4 ± 3.3	
10	77.1 ± 4.4	78.1 ± 4.6	75.8 ± 6.3	80.1 ± 1.2	
5	76.7 ± 5.4	79.4 ± 3.6	75.8 ± 7.2	79.6 ± 2.3	
2	76.3 ± 4.6	79.4 ± 2.0	76.8 ± 6.5	78.7 ± 3.2	74.5 ± 4.9
1	78.2 ± 6.4	79.2 ± 2.7	76.8 ± 6.7	78.3 ± 3.2	
0.5	79.0 ± 6.7	79.0 ± 3.5	76.8 ± 6.8	80.3 ± 7.9	
0.1	76.2 ± 8.6	80.5 ± 4.4	76.8 ± 9.2	81.3 ± 8.1	
0.01	78.8 ± 13.2	83.7 ± 6.1	80.9 ± 2.7	80.8 ± 4.6	
means	77.3	79.1	76.6	81.5	
scatter in means	1.0	2.6	1.8	3.0	

Acknowledgement

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PI Reviewers Comments (Anne Skinner)

I am strongly in favour of this type of work. It is a pity that the constant pressure to produce "useful" results (ie dates) limits the opportunities to go back and check (and recheck) fundamental assumptions about the nature of the phenomenon being used. Therefore I would rate this contribution as both interesting and a constructive addition to the literature. There is one minor comment. One would expect the relatively sharp peaks of the radical species to vanish when the modulation amplitude is increased. This is simply an artefact of the measurement process. It is not clear that they are "suppressed", in the sense of no longer contributing to the spectrum. Rather, the intensity of these peaks may be contained within the envelope of the larger peak. In contrast, when the temperature of the sample is lowered, the relaxation time of the radical may be such that it cannot respond to the magnetic field. In that case, the effect from these radicals has truly been removed from the spectrum. In the example shown in this paper it does not seem to matter what technique is used, which suggests that the contribution by the radical peak to the overall intensity is insignificant.

The apparent decrease of AD with increasing power at low modulation amplitude, shown by sample 529, is initially of some concern. However, the use of sample 533 as a "control" suggests that the variability is indeed a function of difficulty in interpreting a complex spectrum, rather than a real problem. The authors were fortunate to have a sample which was free of radicals.

Perhaps the most useful information is that on low temperature behaviour. As the authors point out, low temperature work is not practicable in many laboratories. It is a comfort to see that it is not necessary.

Zero thermoluminescence for zero age

J.R. Prescott and R.A. Purvinskis
Department of Physics and Mathematical Physics, University of Adelaide, Adelaide, Australia 5001.

Introduction

Among the criteria set down by Wintle and Huntley (1982) in the review paper with which they discussed their application of thermoluminescence (TL) to the dating of sediments, was the requirement that very recent deposits should have zero TL age. In practical terms this does not necessarily mean that the sample should have zero TL in an absolute sense but that the procedures used in dating should yield zero age. Nevertheless the requirement of zero age is closely bound to the degree of resetting of the TL clock by exposure to sunlight during the time that the sediment was being laid down. Doubts always exist because the samples being dated may not have been exposed to sunlight for "long enough" to reset the clock.

The literature (see e.g. past TL/ESR Workshop proceedings) has many examples of ambiguities due to uncertainty in the degree of zeroing of the samples and there has been considerable study of the bleaching process itself and of methodology designed to minimise the uncertainty in bleaching. A representative list of references would include: Wintle and Huntley, 1982; Singhvi et al, 1982; Huntley, 1985; Redhead, 1984, 1988, Spooner et al, 1988; Bailiff and Poolton, 1989, Berger, 1990, and Robertson et al, 1991.

In recent articles in this journal, Franklin and Hornyak (1990) and Prescott and Fox (1990) have suggested the use of new procedures to overcome some of these uncertainties when quartz is concerned. These authors suggest the use of the 325 °C glow-curve peak which emits at 380 nm and is very rapidly bleached. The bleaching time of this peak to effective zero intensity depends to some extent on the sample but is of the order of minutes of full sunlight. Other quartz glow-curve peaks may require times of the order of days to be fully zeroed (Spooner et al 1988, Prescott and Fox 1990). The use of optical filters can distinguish between the "rapidly bleaching peak" (using the nomenclature of Franklin and Hornyak) and the slowly bleaching peaks in the glow curve. The former is selected by the use of a UG2 or UG11 filter and the latter by first removing the rapidly bleaching component with a short exposure to sunlight through a filter with a short wavelength cut-off at, say, 475 nm. This then gives the baseline from which 325 °C peak rises. We refer to this as "*selective bleach*". In the course of TL dating of a range of Australian sediments we have encountered some examples for which the conventional *total bleach* method of Singhvi et al (1982) has given apparently anomalous results: in particular, in giving non-zero ages for zero age deposits (Prescott 1983, Tejan-Kella et al 1990). In these cases, the discrepancy can now be shown be due to insufficient bleaching for the *total bleach* methods to work. i.e., to the sample's not having been set sufficiently to zero when it was originally deposited.

Results

We report here tests of the "selective bleach" technique, designed to show whether or not it avoids the discrepancies for zero age samples that were noted at the time of the original published measurements (Prescott 1983, Tejan-Kella et al 1990). The emphasis is on identifying zero age. Revised dates for other samples will be reported elsewhere.

For these "modern" samples the method used was to compare the glow curve for the "natural" sample with the glow curve for the same material after bleaching by 30 minutes of natural sunlight filtered by a 475 nm filter (cutoff to 1% at 475 nm). If these glow curves are the same, it indicates that there was no 325°C component in the original sample and that, regardless of the apparent age found by conventional total bleach methods, the TL age is zero within the sampling errors.

Figure 1 shows this for sample CA20S/1, modern dune sand from the Carlo Blow near Cooloola in Queensland, Australia (Tejan-Kella et al 1990). This sample comes from a depth of 1 m in the toe of an actively mobile dune. The time since the sample was last exposed to light can scarcely exceed a decade because the dune is currently encroaching on living vegetation. This sample gave an age of several thousand years when dated by conventional *total bleach* methods. From figure 1 it is clear that there is little or no 325 °C component *per se* in the glow curve although there is a substantial amount of light in the glow curve in the corresponding temperature region. It was found that 20 hr of full sunlight reduced the TL of this sample by about half. The conclusion drawn is that the revised procedures now give a "modern" age to this sample, which is consistent with its geomorphology.

TL dating of sand dunes from the Roonka prehistoric archaeological site has been described in Prescott (1983). In that reference it was shown that surface samples, which would therefore be zero TL age, could be bleached to about 50% of their "natural" level in the temperature region 300-400°C and had apparent ages significantly different from zero. We have remeasured one of these surface samples, EB1S/02, and the natural glow curve is compared with the selective bleach curve in figure 2. Since these curves are the same within the reproducibility errors, the new technique has recovered modern age for a sample which previously gave an age of some 400 years.

In both cases, limited tests show that a true ED of about 0.1 Gy would be detectable. It may be noted that the glow curve for EB1S/02 differs from that for CA20S/1 because of the presence of a large 280 °C component in the latter.

It is concluded that the proposed ***selective bleach*** technique gives zero age for modern samples for which *total bleach* methods give non-zero ages and satisfies at least this criterion of Wintle and Huntley. An expanded

Figure 1.
Comparison of natural (crosses) and selective bleach (circles) glow curves from Carlo Blow (sample CA20S/1). The place where the 325 ℃ peak would be found is indicated by the arrow. The absence of such a peak demonstrates the modern age of the sample. In this figure and in figure 2 the curves have been glow-normalised and the error due to counting statistics lies within the symbols.

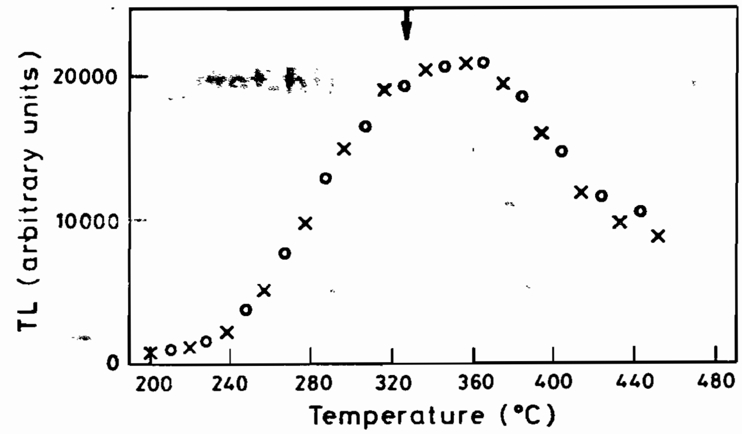
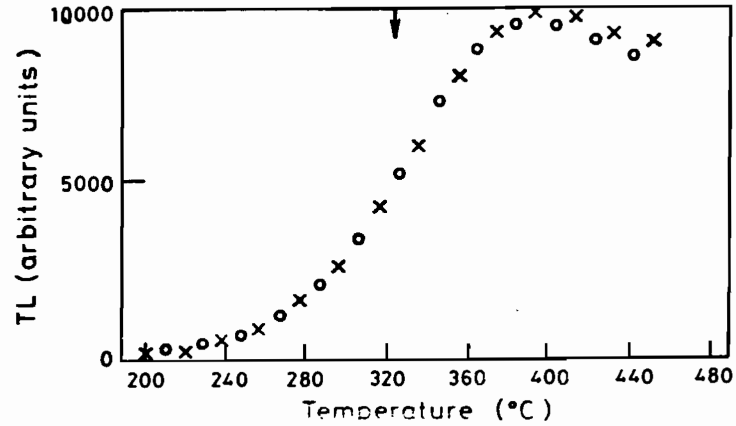


Figure 2.
Comparison of natural (crosses) and selective bleach (circles) glow curves for surface material from Roonka (sample EB1S/02). The place where the 325 ℃ peak would be found is indicated by the arrow. The lack of any difference between the two curves shows that this currently mobile dune surface is modern.



version of this note will appear in *Proceeding of the Fourth Australasian Archaeometry Conference.*

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PR Reviewed by D. J. Huntley

The hypothesis of mid-term fading and its trial on Chinese loess

J. Xie† and M. J. Aitken
Research Laboratory for Archaeology, 6 Keble Road, Oxford OX1 3QJ, U.K.

†Permanent address: Xian Laboratory for Loess and Quaternary Geology, Academia Sinica, Xian, China.

The model

To correct for fading of the TL signal in alkali feldspars Mejdahl (1988)uses a model based on a single trap-type with a single decay lifetime - determined as being between 800 ka and 5400 ka in the samples studied. Hütt and Jaek (1989) use a similar approach in discussing laboratory reconstruction of paleodose. While Mejdahl's model appears to fit the facts for a single trap-type in alkali feldspars we present here a modified model which seems to be appropriate for the optical dating signal obtained from polymineral samples of a long loess section at Duanjapo, near Xian, China. Application of the model to make correction for fading requires, as does the Mejdahl model, the availability either of a sample of known age - in the present case this is from the layer containing the Brunhes-Matuyama boundary (730 ka) - or of a sample having a minimum age sufficient for its expected paleodose to correspond to laboratory saturation.

The essentials of the model are:

(i) that the dating signal consists of an unstable component and a stable component,
(ii) that the lifetime of the latter is long compared to the age of the oldest sample, and
(iii) that the lifetime of the unstable component is 'mid-term', i.e. long compared to laboratory time but short compared to the ages being determined.

In the present instance a lifetime of several hundred years can be hypothesized - though its actual value does not enter into the correction calculations. It is further assumed that the luminescence characteristics of the loess are the same throughout the section.

In the postulation of two components the model is similar to that developed for zircon TL by Templer (1985) and successfully used for the TL dating of polymineral grains from pottery by Clark and Templer (1988). An earlier suggestion that a mid-term fading process might be operative was by Grün et al (1989).This was in respect of 30 - 40% underestimation of TL age, using K-feldspar (and a UG-11 filter in the detection system).

Procedure

As found by many workers on Chinese loess both TL and optical ages are very substantial underestimates for samples having expected ages beyond one or two hundred thousand years; typically an age of the order of 150 ka is obtained for a sample dated to 730 ka by its occurrence close to the Brunhes-Matuyama (B-M) boundary. Figure 1 illustrates the situation in principle - the actual experimental plot is less straightforward. As will be seen the observed signal from the B-M sample is some 37% lower than that expected on the basis of the laboratory growth curve obtained by dosing bleached aliquots of the sample. Then according to the model the unstable component constitutes 37% of the artificially-induced signal, and hence the growth of the stable component during antiquity can be represented by a curve derived by multiplying the laboratory curve by the factor 0.63.

Figure 2 illustrates the dating of a sample higher up in the section. The growth of the stable component during antiquity is obtained as just indicated and the intercept of the natural signal on the curve so obtained allows evaluation of the paleodose and hence the age.The essential difference from the Mejdahl approach is that because of assumptions (ii) and (iii) the fractional loss of signal by fading is assumed to be independent of the age of the sample (as long as that age is large compared to the hypothesized fading lifetime of the order of several hundred years); in the Mejdahl approach the fractional loss is assumed to increase with age.

Experimental

Signals were obtained using stimulation by 514 nm (green) light from an argon ion laser and detected using an EMI photomultiplier type 9635Q, preceded by four Corning filters type 7-59 and one Schott filter type BG39. Aliquots were presented for measurement as 'middle-grains' (20-50 µm) on aluminium discs, being pre-heated at 160 °C for 2 hours after irradiation. Before irradiation aliquots were bleached to a negligible level by means of a Hönle (SOL 2) solar simulator.

A ^{90}Sr/^{90}Y source was used for beta irradiation and the beta dose to middle grains was taken to be 1.08 times that to fine grains (4 - 11 µm). The a-value for the latter was found to be 0.07±0.01 using an Am-241 source in vacuum for alpha irradiation. It was assumed that uranium and thorium were distributed uniformly throughout all grains; since only about 20% of the signal is due to alpha particles the validity of this assumption it not too critical. Radioactivity measurement techniques included thick source alpha counting, potassium analysis and high resolution gamma spectrometry.

An important aspect of the measurement procedure was that all samples were subject to identical preparation and pre-heating conditions. To this end two or more samples were prepared, preheated, and measured in parallel in such a way as to 'normalize' to the B-M sample. Further details of the procedures used are given elsewhere (Xie and Aitken, 1991).

Figure 1.
Illustration of how the size of the mid-term fading component is evaluated using the known age of the sample from the B-M boundary. The regenerated laboratory growth curve is of the form

$$L = L_{sat} (1 - exp (-D/D_s)$$

where D is the laboratory dose and D_s = *600 Gy. The natural signal is* $L_{B\text{-}M}$, *and* $L_{B\text{-}M}$ *is experimentally found to be 0.63 times the value of L at D = 3.4 kGy; the latter is the predicted paleodose corresponding to an age of 730 ka, the annual dose being 4.7 Gy/ka. The hypothesized curve for the growth of the stable component is drawn so that at each dose value it is 0.63 times the co-ordinate of the laboratory curve at that dose value. The age obtained for this sample if it is assumed that there has been no fading is 127 ka.*

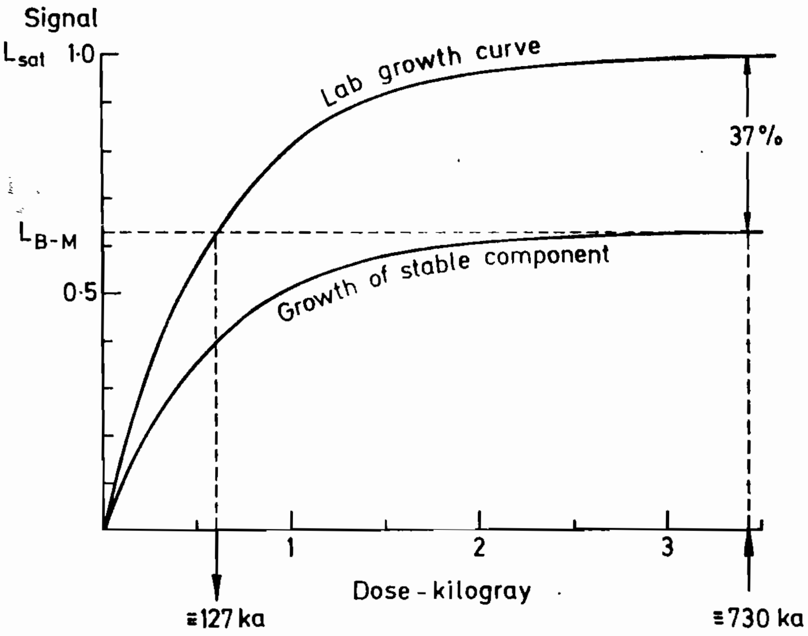


Figure 2.
Illustration of dating procedure for sample from 12 m. As in figure 1, the growth curve of the stable component is taken to be 0.63 L_{sat} *(1 - exp(-D/*D_s*). On this basis the age of the 12 m sample is 230 ka whereas if no correction is made for fading the age is 95 ka.*

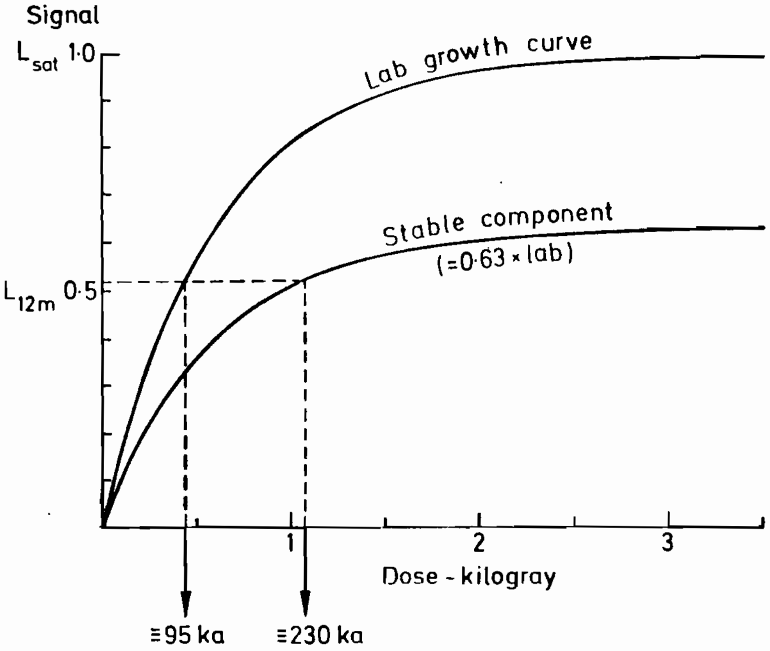
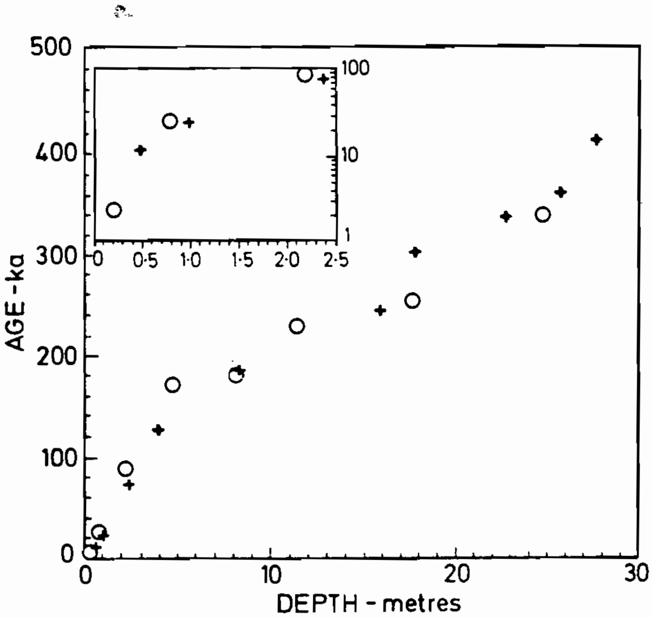


Figure 3.
Ages obtained for samples from the upper 25 m of the section - indicated by circles. Crosses show the ages according to the astronomical chronology for the oxygen-isotope stages assumed to correspond to the various palaeosol-loess boundaries of the section. The inset is a semi-log plot of the younger dates.



Results of application

Figure 3 shows ages derived on the above basis for 8 samples from the upper 25 m of the section. Also shown are the ages obtained by matching the successive palaeosols to oxygen-18 climatic stages and hence to the astronomical timescale. It will be seen that the agreement with the latter is encouraging. On the other hand the corresponding ages obtained in an attempt to use the Mejdahl method of correction were substantially too low - as is inherent in any model in which the fractional loss becomes small for young ages; of course the Mejdahl method is strictly applicable only where a single trap-type and lifetime is involved.

Discussion

The purpose of this note is primarily to call attention to a possible model for fading that has not so far been considered (except for the mention by Grün et al noted above). Secondly it is to report that in application to one section of Chinese loess at any rate the fading correction based on the model yields ages consistent with the section's interpretation in terms of oxygen-18 isotope stages. Clearly confirmatory measurements and estimation of error limits need to be done before consistency can be claimed definitively. It would also be interesting to apply the method to sections having more direct age constraints and to try it using TL.

In conclusion it must be stressed that it is necessary to have a layer of known age, or an undated layer having a minimum age limit sufficient for saturation to have been reached; also that the luminescence characteristics have to be the same throughout the section. It should also be borne in mind that the upper layers of the section may be too young for the validity of the assumption that the lifetime of the fading component is substantially shorter than sample age.

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PI Reviewers comments (Ann Wintle)

The model of Xie and Aitken assumes that:

(a) The unstable component has the same growth characteristic D_S as the stable component. If it is a totally separate luminescence signal, the values of D_S are likely to be different, which would invalidate the simple approach of multiplying the laboratory growth curve by 0.63.

(b) The filling of the electron traps giving rise to the stable component is unaffected by the fact that the electron traps giving rise to the unstable component remain empty, i.e. there is no competition for electrons during natural irradiation of the sample.

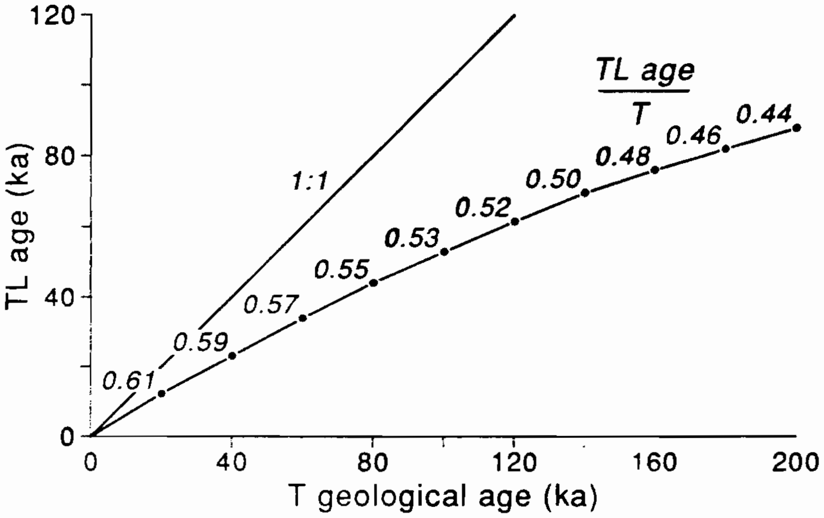
(c) The fraction of the signal which is unstable is constant for all samples. If this were the case, then the correction proposed for the measured ED would be irrespective of the measured dose rate for the sample.

The model suggests that substantial underestimation will be encountered irrespective of which method of ED determination is used.

In an attempt to assess the potential of this hypothesis as the explanation of apparent age underestimations encountered whilst dating loess sections, I have plotted the luminescence age that would be obtained if no correction were made against the true age. In this way it is analogous to that produced by Debenham (1985) for TL age estimates for European loess from different sections and indeed bears a general resemblance to it. The curve was calculated for a dose rate of 4.7 Gy/ka, with D_S = 600 Gy and a fractional stable component - 0.63, as specified by Xie and Aitken.

The underestimation of the luminescence age will initially be 40%, but because the growth curve is a saturating exponential, this underestimation will be larger for older samples, reaching 53% by 100 ka and 56% by 200 ka. Within the range of radiocarbon dating (<40 ka) the systematic underestimation would be 39±2%. An underestimation of this order of magnitude should have been reported from TL studies; however, apart from a number of radiocarbon dates on secondary carbonates nodules within the uppermost loess unit (the Malan loess), there are no independent dates with which to compare the results of the model. Very few useful radiocarbon dates on suitable material have been reported from loess sections which have been sampled for luminescence dating. (It should be noted that Debenham's youngest age estimates were for samples with indirect and incorrect age control (Debenham, 1985).) In general no such systematic underestimation has been obtained for loess in the range 13 - 30 ka (Wintle, 1990). Hence a fading component with a lifetime of only a few hundred years is unlikely.

Although the correlation of the Brunches/Matuyama boundary is well dated (by potassium-argon dating of lavas flows) at 730 ka, the correlation of the boundaries of the palaeosols with particular oxygen isotope stage boundaries is open to debate. Hence care should be taken when such correlations are used as a test for another dating method. This criticism is particularly true for the first major palaeosol, S_1. In this paper it is taken to date 74 ka to 130 ka (from figure 3) with loess deposition commencing at the 5/4 Oxygen Isotope



Stage boundary, rather than at the 5e/5d Substage boundary at 108 ka (see Wintle, 1990 for discussion), a possible alternative. Correlations for all soils are based on a "count down from the top" approach. No other studies have been published for the 30 m loess section at Dunajapo. It is therefore difficult to confirm the interpretation of the loess/palaeosol sequence at this site.

If loess deposition commenced at 74 ka and if the hypothesis of Xie and Aitken was correct, then it would imply that no TL age estimate over 40 ka would be obtained for the Malan loess by the straightforward interpretation of the TL data (and no age over 85 ka would be obtained for loess within Oxygen Isotope Stage 6). However, published TL ages for the base of the Malan loess range from 65 - 94 ka (Zhou LiPing, pers. comm.). This would suggest that the lifetime for a single fading component comprising 37% of the luminescence signal could not be on the timescale of a few thousands of years.

Although TL age estimates for 100-300 µm feldspar grains from cover sands in Northwest Europe (Dijkmans and Wintle, 1991; Grün, Packman and Pye, 1989) have been systematically about 40% low when compared with reliable radiocarbon dates from the same sections, the results cannot be taken to support the hypothesis of Xie and Aitken. In those cases, and also for older samples measured in the same way (Balescu et al, 1991), the underestimation only occured when ultraviolet pass filters were used for the TL measurements. The effect of the choice of filter was demonstrated by Balescu and Lamothe (1991) and is clearly caused by a different phenomenon.

Though the laser stimulates OSL from both feldspar and quartz, the TL also comes from both mineral components, but the relative intensities may be different and may not allow direct comparison of the dating information. Having criticized the hypothesis on the basis of a world-wide data base of TL results, I will conclude with a evaluation of the OSL results presented by Xie and Aitken (figure 3). For the three samples, from 0.7 to 5 m, the corrected ages appear to be overestimated compared with the palaeosol/∂^{18}O correlation, implying that the correction over-compensates. For older samples it is difficult to judge the agreement since the age uncertainty becomes very large and asymmetric.

In conclusion it should be noted that the approach suggested by Xie and Aitken is highly sensitive to the numerical value for the fractional stable component, i.e. the ratio of the natural luminescence signal for a very old sample (e.g. from the B/M boundary) to its asymptotic signal reached after prolonged laboratory irradiation. An error of only a few % would have a large affect on the calculated age. The error in the age obtained in this way will be even larger after taking account of the scatter in the natural luminescence signal for the sample being dated.

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Reply by the authors

First we would like to reiterate that the primary purpose of our note is to call attention to mid-term fading as a phenomenon that may need to be considered. Currently, with polymineral grains at any rate, we have the impasse that samples which are given a clean bill of health on the basis of laboratory fading tests nevertheless give gross underestimate of age. A mid-term fading component could be the explanation (or at any rate part of the explanation) and the fact that there are difficulties in making correction by means of the relevant model, such as the rather wide error limits that result (as the reviewer points out), is hardly an argument that the phenomenon does not exist. The reviewer's conclusion that the lifetime of the fading component would have to be in excess, not only of a few hundred years, but of a few thousand years, is in the same category of being a difficulty in making correction. As we say in the paper '... the upper layers....may be too young for the validity of the assumption that the lifetime .... is substantially shorter than sample age.' Our suggested lifetime of a few hundred years may indeed be too short - but this does not invalidate the basic hypothesis that a mid-term fading component is present. We accept the possibility of alternative interpretations of the section but those suggested by the reviewer do not destroy the broad agreement between corrected age and the oxygen-18 timescale. We take this broad agreement as encouragment for the basic hypothesis to be given further consideration, henceforth bearing in mind the various caveats that the reviewer has put forward - and we are appreciative of her critical assessment. In conclusion we would emphasize that in the first place we see the hypothesis as applicable to optical dating of polymineral grains from the loess section concerned; whether or not it is applicable to the TL signal from those grains will be investigated by one of us (JX). From the reviewer's comments in respect of TL dating of K-feldspar it is good news that age underestimation can be avoided by wavelength selection. Unfortunately there is no encouagement so far to think that this is also the case for optical dating of polymineral grains (e.g. Spooner and Questiaux, 1989).

Reference

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Removal of the thermally unstable signal in optical dating of K-feldspar

Sheng-Hua Li, Institute of Earth Studies, University College of Wales, Aberystwyth SY23 3DB, U.K.

Introduction

Optical dating has been developed over the past five years since it was first described by Huntley et al. (1985). However, one important aspect of optical dating has not received much discussion - the removal of the thermally unstable component of the signal induced by laboratory irradiation. This component is not stable at ambient temperature over the relevant geological time period. If this component is not removed by appropriate thermal pre-treatment, the equivalent dose, and hence the age of the sample, will be underestimated.

Preheating, or thermal washing, is often used to remove an unstable component in thermoluminescence (TL) dating (Mejdahl and Winter-Nielsen, 1982) and has been used in the optical dating of quartz (Huntley et al., 1985; Rhodes, 1988). Because the majority of the optically stimulated luminescence (OSL) signal from quartz is correlated to the 325 °C TL peak (Smith et al., 1986), a preheating procedure was developed which was based on the TL behaviour of this peak (Rhodes, 1988). However, this procedure would not be expected to be suitable for other minerals. For K-feldspar, the OSL is from at least two kind of traps, as demonstrated by the loss of signal from two TL peaks (Li and Aitken, 1989).

Experimental design

The design of a preheat procedure involves the choice of temperature and time. A very high preheating temperature can result in an extremely short preheating time with risk of failure to reach complete thermal equilibrium and of effects due to sample oxidization. On the other hand, if the preheating temperature is very low (say 50 °C), it may take months or years, making such a procedure impossible in practice.

For a particular preheating treatment to be appropriate, the thermally unstable signal should have been removed and enough stable signal left for measurement. Three possible tests are suggested.

a. Plateau method.

This is similar to the plateau method in TL dating (Aitken, 1985). Once the stable signal has been removed, a plateau of the OSL ratio for natural and irradiated samples is to be expected as the preheating time is increased.

b. Thermal decay rate.

The same thermal decay rate for the OSL signal is to be expected once sufficient preheating is achieved, because the remaining natural and irradiated signals will have the same thermal stability.

c. Similar shape of the TL glow curves.

After a suitable preheating, the TL glow curves should be similar for the natural and irradiated samples. Furthermore, the low temperature peak, which is thermally unstable, should be erased and only the high temperature peaks should be left.

Experimental details

Single wavelength (514.5 nm) light from an argon laser was used for stimulation (Smith et al. 1986). The power of the expanded laser beam incident on the sample was about 5 mW/cm^2. Detection of the stimulated luminescence was by means of a photomultiplier (EMI 9635QA) with four Corning 7-59 filters and one Schott BG39 filter in front. During measurement, the sample disc was placed on a heating plate which could be held at a precise temperature during illumination and all measurements are made at a sample temperature of 17 °C.

TL measurement was performed on a manual TL set with heating rate 5 °C/s. TL was detected with one Corning 7-59 filter and one HA3 filter in front of an EMI 9635QA photomultiplier.

K-feldspar was mounted on 10 mm diameter discs with a monolayer of 100-300 μm grains affixed by means of a silicone oil aerosol spray. Three K-feldspar samples separated from sediments from Scandinavia were used in this study. The Oxford Laboratory numbers are Z10, Z29 and Z30, and the TL equivalent doses are 70, 480 and 1120 Gy respectively (Li and Aitken, 1989). The Nordic TL Laboratory (Risø) reference numbers are 862403, 859008 and 869004.

A short shine (0.1 second) of laser light was used for the OSL measurements. This caused less than 0.4 % loss of the OSL signal and negligible loss of the TL signal (Li and Aitken, 1989). Hence, the discs could be used for more than one measurement.

A purpose-built thermostat-controlled oven was used in the 160 °C preheating studies.

Results and discussion

In practice, two different kinds of preheating are required. One involves a long time (a few hours) at a lower temperature, the other a short time (a few minutes) at a higher temperature. The former can be performed in an oven. The latter can be performed on a TL heating plate and is particularly useful if the same equipment is to be used for both TL and OSL measurements since measurements can be made without moving the discs.

1. Prolonged preheating

From other studies, it is known that there is a TL peak around 160 °C in the TL glow curve after laboratory irradiation. This TL maximum is not found in natural samples and is therefore considered to be an unstable signal.

For sample Z29, two discs, one containing the natural signal and one with an additional irradiation (200 Gy) were placed in a 160 °C oven for a total period of more

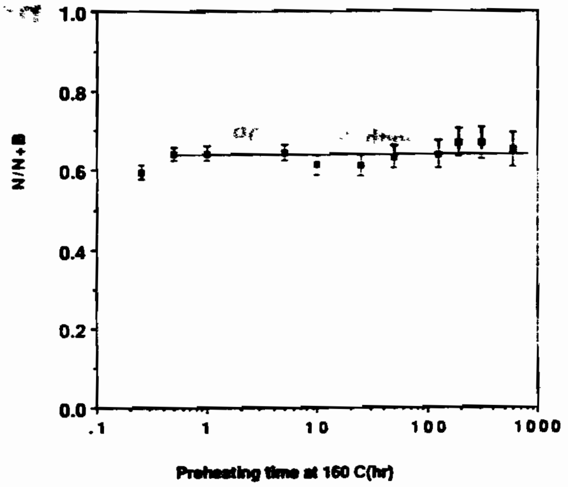

Figure 1.
Sample Z29 - OSL ratio versus preheating time at 160 °C. The ratio is between natural and natural + β dose (200 Gy) discs. (natural TL ≡ 480Gy)

than 200 hours. The discs were periodically taken out for OSL measurement. Figure 1 is the plot of the OSL ratio for these two discs and it shows that a plateau was reached after a half hour at 160 °C. Use of a 0.1 second laser exposure allows the discs to be used many times; hence no normalization is required to improve the precision. This is analogous to obtaining a plot of ED versus preheating time for a group of discs but this requires normalization and curve fitting procedures which reduce the precision.

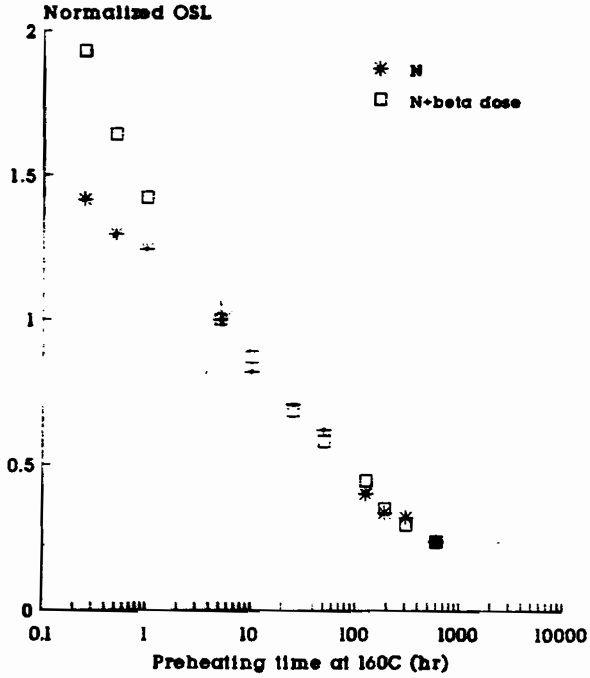

Figure 2.
Sample Z10-OSL signal isothermal decay curves at 160 °C; the added dose is 70 Gy. OSL was normalized to the OSL counts of after 5 hours preheating. (Natural TL ≡ 70 Gy)

Using a similar set of measurement, the thermal decay rates of the natural and irradiated signals can be compared.

Figure 2 shows the thermal decay of the OSL that occurs during the preheating for sample Z10. This shows that the thermal decay rates are the same once the two aliquots have been heated five hours or more and there is a thermally unstable signal in the dosed samples which can be removed after 5 hours preheating at 160 °C.

To demonstrate further the similar thermal response of natural and irradiated samples after preheating, a "quick heating" experiment was performed (Li and Wintle, 1991). The disc is heated to a given temperature, cooled to 17 °C and the OSL measured at 17 °C with the 0.1 second laser stimulation light. This measurement is repeated after heating to successively higher temperatures (figure 3). The responses were similar after preheating for half an hour at 160 °C. This implies that the OSL signal is from a similar temperature range for both discs after this preheating treatment. This was not the case for the OSL from discs which were not preheated.

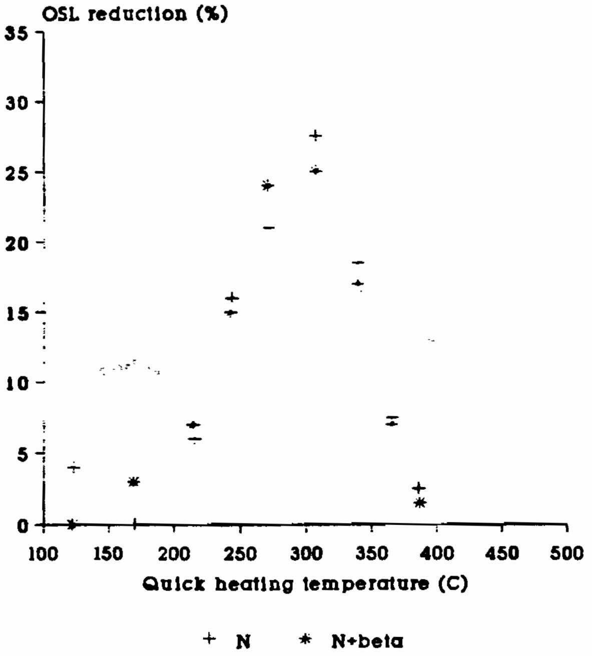

Figure 3.
Sample Z29-OSL reduction (% of whole OSL signal) resulting from each quick heating step , after preheating for half of an hour at 160 °C for natural and N+β samples. (100% is the whole OSL signal before quick heating)

2. Short preheating.

Other dating procedures require the removal of the thermally unstable signal in a short time. Ten minutes was chosen as a convenient preheat time, and the effects of different preheating temperatures were studied.

As already stated the OSL signal results from at least two populations of charge and it is impossible to distinguish their thermal stabilities using the OSL itself (Li and Wintle, 1991). To isolate the OSL signal with

the highest thermal stability, a preheat procedure was developed to leave only a high temperature TL peak.
Preheating experiments were carried out on the TL heating plate immediately before TL measurement. In an attempt to isolate a peak in the TL curve after preheating, the Width at Half the Height (WHH) of the maximum of the TL was measured for each preheating temperature. Because the WHH is independent of the mass of grains on the disc, normalization is not necessary. Figure 4 shows the values of the full WHH for the natural TL and for the second glow response for the same disc sample. The dose used was less than the ED and this caused the deviation of the two data sets. The left half of the WHH (LWHH) gave more consistent results. After ten minutes at 210 °C, the lower temperature peaks were removed, including the 280 °C peak which was found in the natural sample (figure 5). This experiment was repeated for all three samples and similar results were obtained.

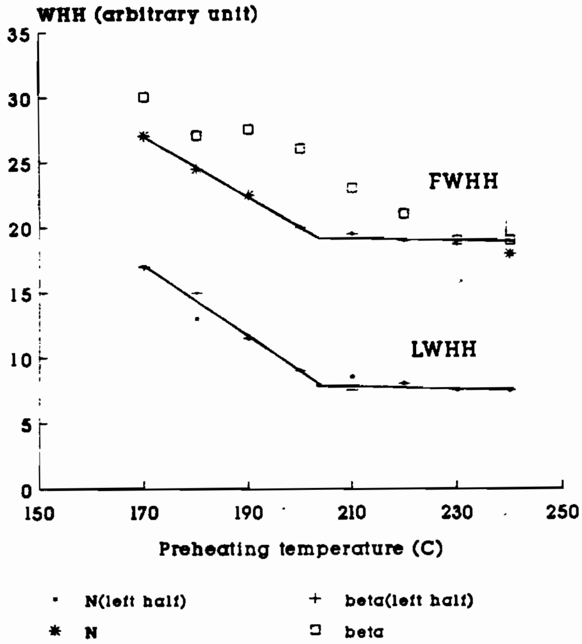

Figure 4.
Sample Z29, plot of WHH (see text) of TL glow curve versus preheating temperature for ten minutes. (*) (•) natural; (□) (+) dosed. The first symbol of each pair refers to FWHH and the second one to LWHH.

Figure 4 indicates that a stable TL signal can be isolated after ten minutes at 210 °C. This was further demonstrated by "quick heat" experiments (figure 6); those gave almost identical results.
In a final experiment, the equivalent dose was obtained for sample Z29 using both preheat procedures, 5 hours at 160 °C and 10 minutes at 220 °C. The values were 272 $^{+22}_{-32}$ Gy and 232 $^{+21}_{-17}$ Gy respectively. While these two values are mutually consistent, they are substantially less than the value of 480 Gy obtained by TL. This suggests either that a substantial relict TL signal remained at deposition in antiquity, or that there was substantial bleaching of the OSL signal during sample preparation.

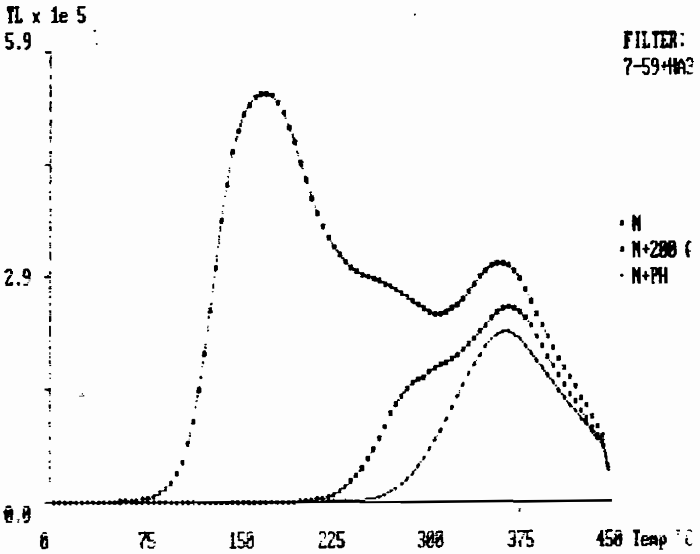

Figure 5.
Typical TL glow curves of samples Z29. (a) Natural+200 Gy dose; (b) Natural and (c) natural + preheating ten minutes at 220 °C.

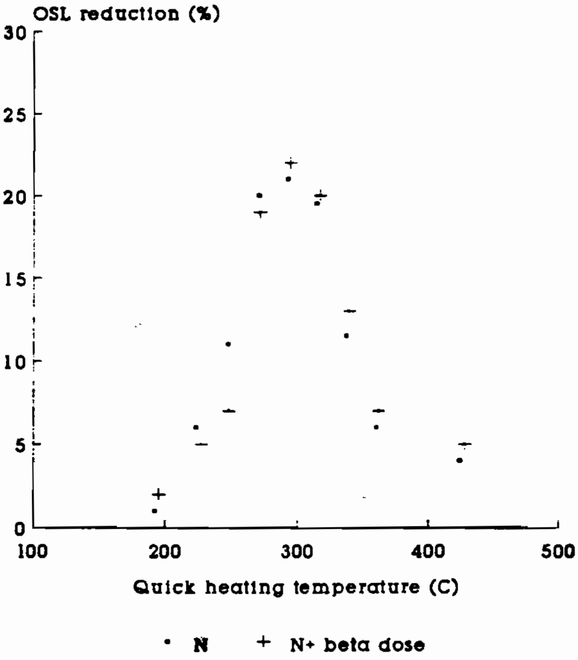

Figure 6.
Sample Z29, OSL reduction (% of whole OSL signal) by each quick heating step, after preheating 10 minutes at 210 °C for natural and N+β dose samples. (100% is the whole OSL signal before quick heating). Beta dose was 200 Gy.

Conclusion

Preheating is needed to remove the thermally unstable signal in optical dating. Combined OSL and TL studies provide an experimental approach to the choice of suitable preheating conditions. Based on results for three K-feldspar separate, preheating for ten minutes at 220 °C or five hours at 160 °C was recommended.

Acknowledgements

I wish to thank Professor M.J.Aitken for his encouragement and supervision and Dr. V. Mejdahl for provision of the samples and EDs measured by TL. Also, thanks to Dr. E. J. Rhodes for useful discussions during this study and Dr.A.G. Wintle for correcting the English and useful comments. Experiments were done at the Research Laboratory for Archaeology, Oxford University while the author was supported for part of the time by a Royal Society Fellowship.

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PI Reviewers comments (Martin Aitken)

Removal of the unstable component is essential if reliable results are to be obtained by optical dating - as indeed pointed out by Huntley et al (1985). Two points additional to those mentioned by the author are in respect of (i) use of ED in fig. 1, and (ii) the possiblity of non-plateau in fig. 1 because of dose-dependent sensitivity change being progressively stimulated by the preheating. As the author points out in respect of (i) use of (N/N+ß) avoids the need for normalization; but on the other hand if there is progressive change in the degree of non-linearity (of growth of OSL with dose) there may be a good plateau if ED is used, but not with (N/N+ß) - the latter giving an erroneously pessimistic assessment of the efficacy of the pre-heating. Evidently neither of these interfering affects are present with the K-feldspar samples under study.

Notices

International Symposium on Evolution of Deserts

February 11-19 1992

The Physical Research Laboratory, Ahmedabad, the Arid Zone Research Association of India, Jodhpur and the Deccan College, Pune are pleased to announce an International Symposium on the Evolution of Deserts to be held at the Physical Research Laboratory, Ahmedabad during February 11-19, 1992.

The symposium will also include the final meeting of the IGCP-252 working-group on the Past and Future Evolution of Deserts. It is proposed that the symposium at Ahmesdabad will be followed by a four day, 1200 km field-trip to the Indian Thar Desert. The symposium will have a theme session on the Climate Changes in Deserts over different time scales. The focus of the theme session will be to compare climatic sequences from various deserts (particularly with that of the Thar Desert) and the marine paleoclimatic records. A series of invited review talks on climate modelling, paleoclimatic studies, remote sensing studies and paleoanthropology are also planned.
Schedule

Registration forms should be requested from the organizer without delay

Timetable:

Second circular:	July 1, 1991
Abstract and registration: fee deadline (receipt in Ahmedabad):	September 15, 1991
Acceptance of abstract (to be mailed by):	October 15, 1991
Scientific programme (to be mailed by):	December 15, 1991

Invitation letters

Scientists needing invitation letters earlier than Sept. 15, 1991 are requested to send an indicative one page abstract(s) on standard A-4 size paper complete with title(s). A revised abstract, if desired, can be submitted later (e.e by Sept. 15, 1991).

Address for Correspondence

Dr. A.K. Singhvi, Convenor, Symposium on Deserts, Earth Science Division, Physical Research Laboratory,
Navrangpura, Ahmedabad 380 009, India

Phone: 0091-272-462129(O), 490116(R)
Fax: 0091-272-445292
Telegram: Research, Ahmedabad Telex: 0121-6397
E.Mail: root @ prl. ernet.in [via Internet to A.K.Singhvi]

Proceedings of a Quaternary Dating Workshop
ANU, Canberra, 1990

The proceedings of the workshop, edited by Richard Gillespie, are now available. 22 papers are included on: radioacrbon dating, amino acid racemisation, TL and disequilibrium dating methods. Copies may be purchased for $15 including postage, prepaid orders in Australian Dollars only, from:

Secretary, Dept. of Biogeography and Geomorphology, RSPacS, Australian National University, PO Box 4, Canberra ACT 2601.

Postdoctoral position
in thermoluminescence and optical dating at Simon Fraser University

Applications are invited for a postdoctoral position in TL and optical dating. Research involves the physics of the luminescence of minerals and development of dating methods. Candidates should have a Ph.D. in physics or geophysics. Salary range is C$23,000-$28,000.

To apply send c.v. and names of 2-3 referees to D. J. Huntley, Physics Dept., SFU, Burnaby, B.C. V5A 1S6, Canada. (bitnet: USERDRHU@SFU).

Interlaboratory Comparisons

1. TL intercomparison on loess - towards a conclusion

It is one year by now that those laboratories involved in the intercomparison on TL dating of four loess samples discussed the preliminary results at the TL/ESR Specialist Seminar in Clermont. The time has come to collect all data for a report, which will be sent out to all participating laboratories for further discussion and comments. I support the suggestion made by some colleagues to request *Ancient TL* to publish the final report as a special issue, but other ideas for publication are also welcome. If necessary, the opportunity to elaborate on any persistent disagreements should be made available as individual comments in the final report under the name(s) of the co-author(s).

In order to advance the completion of the intercomparison final report, all participiants are requested to transmit their results to Heidelberg by email, fax or letter within one month after this issue of AnTL appears. Please include the following points: grain size fraction, radiation sources, light source used for bleaching and duration, delay between bleaching and regeneration, delay between irradiation and and TL readout, pre-heating techniques and efforts to remove anomalous fading, optical filters and photomultiplier specifications, heating rate, method used for equivalent dose evaluation, a-value, radioactivity data (including the method), estimated moisture content, effective dose-rate and TL-age. †

After the compilation of all information received I shall send the draft to a reviewer. After the reviewer`s comments the draft report will circulate among all participiants for further comments before the final report is submitted for publication. It is important to find an early agreement on the form of publication (e.g. as a report by the whole group or as individual short papers) and the periodical (AnTL or other). Let me know your suggestions, please.

Ludwig Zoeller
Forschungsstelle Archaeometrie der Heidelberger Akademie der Wissenschaften, c/o MPI für Kernphysik, Postfach 103980
D-6900 Heidelberg, Germany.

fax: ++49-6221-516540 email: ZOELLER@DHDMPI5V.

† *Ed Note:* A specification for sediment ages for a forthcoming Sediment Date List is in preparation.

2. "Old" (>200 ka) loess sample available for interlaboratory comparison of TL sediment-dating procedures.

Prepared portions (≈4-11 µm size fraction) of an independently dated loess sample will be available to interested laboratories. The age is known from a zircon fission-track date for an associated tephra bed. The expected age of this loess has been obtained by the partial-bleach TL method. The objective of this interlaboratory test is to assess the accuracy of different TL procedures for the dating of old loess, and to compare results (to be made public) from different laboratories. This test may help to resolve the continuing ambiguity, both within the TL dating community and the end-user community of earth scientists, about the accuracy of TL sediment dates older than ≈100 ka.

To simplify this proposed interlaboratory comparison, participants will be asked to measure only equivalent-dose values, by each of two approaches. They should use both their own usual TL procedures and some of those employed by the Heidelberg laboratory. Specific procedures from that laboratory will be recommended to interested participants. This will be a "blind" test, with the independent age and expected equivalent dose being revealed after completion of the test.

Interested persons should write to: Dr. Glenn Berger, Department of Geology, Western Washington University, Bellingham WA 98225-9080, U.S.A.

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Compiled by Ann Wintle

Errata

Cosmic ray dose-rate determination using a portable gamma-ray spectrometer
Ancient TL 9(1), by Andeas Bürgi and Markus Flisch

On page 4, 5th line, the equation $\dot{D}[\mu Gy/a] = 8.5 \times (cpm_{discr.-ch.}) - 54.3$ should have read:

$$\dot{D}[\mu Gy/a] = 8.5 \times (cr_{discr.-ch.}) - 54.3$$

The Editor.

Pairs precision required in alpha counting: further errata

Referring to p.12 of *Ancient TL* **8**(2), in the line immediately following equation (4) the term (α/t) should raised to the power 0.5, i.e. $(\alpha/t)^{0.5}$; also in equation (5) the first term on the right hand side should be $(86.6)^2\,(\alpha/t)$.

Furthermore the denominator in equation (8) should be

$$(\alpha t)\,\{67+796(B-C)\}^2$$

Referring to p.10 of *Ancient TL* 9(1), the correct version of equation (7) is

$$\begin{aligned} D_\gamma &= 67.0\,\alpha_u + 104.7\alpha_h \\ &= 67.0\,\alpha + 37.7\,\alpha_h \end{aligned}$$

and the correct version of equation (9) is

$$\begin{aligned} D_\beta + D_\gamma &= 153.6\,\alpha_u + 162.0\,\alpha_h \\ &= 153.6\,\alpha + 8.4\,\alpha_h \end{aligned}$$

I apologise to readers who have been mystified and I am grateful to D Questiaux for drawing my attention to the first three errors.
M.J. Aitken.