

STUDY OF THE EFFECT OF PRE-ANNEALING ON SEDIMENT TL USING A TECHNIQUE OF GLOW CURVE ANALYSIS

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INTRODUCTION

A short annealing at elevated temperatures is now commonly given to sediment samples before measurement of their TL. It has been claimed (Wintle, 1985) that pre-heating at 230°C for 1 minute selectively removes the low temperature TL peak relative to the higher temperature peak. The effect of a given pre-heat treatment may be expressed in the following form:

$$I_B^P(T) / I_A^P(T) = P(T) \cdot I_B^N(T) / I_A^N(T)$$

where I_A^P and I_B^P are the TL intensities after pre-heating of the low and high temperature signals respectively, I_A^N and I_B^N are the natural intensities (ie without pre-heating) of these signals, and $P(T)$ is the enhancement factor: all are functions of glow temperature, T . Then, the condition that selective removal of the low temperature signal (signal A) relative to the high temperature signal (signal B) is achieved at a given glow temperature, T , is $P(T) \gg 1$. This paper describes a measurement of $P(T)$ in the temperature range 270°C to 320°C, which covers that part of the plateau region most useful for dating, and concludes that pre-heating has only very limited application for separating the two signals. This study introduces a technique of glow curve analysis which has wide potential application.

METHOD OF GLOW CURVE ANALYSIS

In practice, measured TL emissions are mixtures of different signals, and, because 100% pure signals cannot be realised, a direct evaluation of $P(T)$ is not possible. The method used here, therefore, is to observe the effects of pre-heating on a series of four natural glow curves which contain differing proportions of the signals A and B, and to extrapolate the measurements to the limiting compositions representing 100% purity in A and B respectively. In this way, the reductions in the intensity of each signal caused by pre-heating can be determined individually. The signal compositions of the four natural glow curves, all measured from the same sediment (lab.ref. PND3), were varied by altering sample preparation and measurement conditions. These glow curves are labelled I to IV and are shown in fig.1. As can be seen, curves I to IV form a progression of glow curve shapes from one which is dominated by signal A, to one which is composed largely of signal B. Sample preparation for curves I and II consisted of fine-grain selection, while the sample for curves III and IV had been additionally stirred in 35% fluorosilicic acid for 3 days. Two different wavelength ranges were selected for the TL observations: curves I and III resulted from use of a Schott UG11 (UV transmitting, blue absorbing) filter, and curves II and IV were viewed with a Corning 7-59 (blue and UV transmitting) and Chance-Pilkington HA3 filter combination. In both cases, the PM tube was a quartz-windowed EMI 9635.

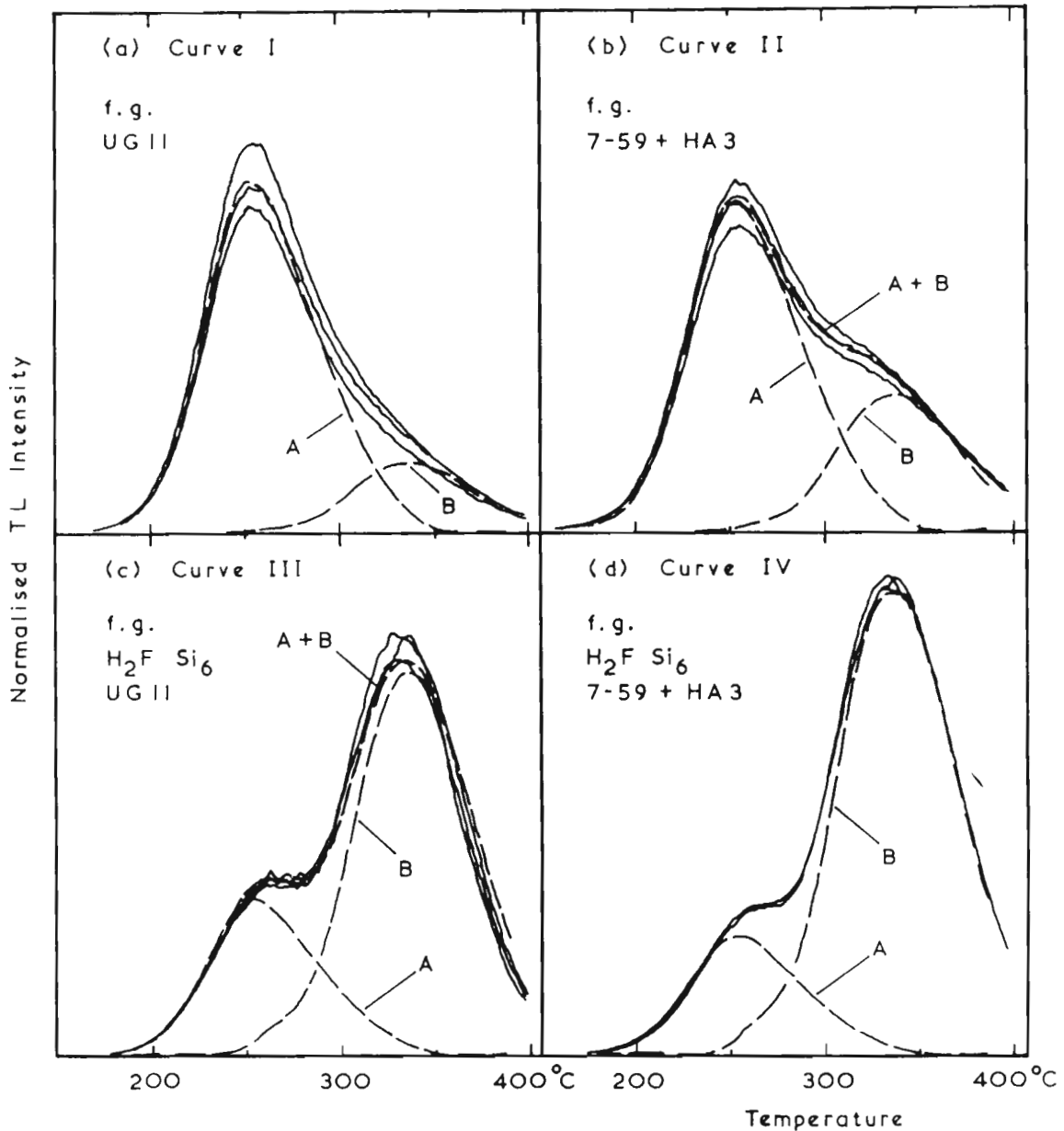


Figure.1. Continuous lines show natural TL glow curves, labelled I to IV, of fine-grains from sediment PND3, normalised by the zero-glow method, and measured at 2.5°C/s heating rate. Broken lines give the shapes of signals A and B as derived from curves I and IV, and their summations, A+B, as fitted to curves II and III. Note that both the shoulder in curve II at about 310°C and the peak in curve IV, 25°C higher in temperature, result from signal B.

The variation of glow curve forms under changing sample pre-treatment and wavelength selections has been described previously (Debenham and Walton, 1983). In that paper, signal B was labelled as a "quartz" signal, and signal A described as "non-quartz". The latter has also been referred to as a "feldspars" or "K-feldspars" signal.

In order to interpret the effects of pre-heating on the natural curves I to IV it is necessary to determine their signal composition at each temperature coordinate. This was done by fitting the curves I to IV with a combination of two curves which represent the shapes of the pure signals A and B. Curve I is composed mainly of signal A and curve IV is dominated by signal B. If two signals only are present, the shape of signal A can be derived by subtracting from curve I a proportion of curve IV:

$$I_A^N(T) = I_I(T) - f \cdot I_{IV}(T)$$

In this equation, f is a free parameter, but has a maximum value, f_{\max} say, defined by the condition that $I_A^N(T) > 0$ for all values of the glow temperature, T . In this work, the shape of signal A has been determined by setting f equal to f_{\max} . Likewise, signal B has been derived by subtracting from curve IV a maximum proportion of curve I:

$$I_B^N(T) = I_{IV}(T) - g \cdot I_I(T)$$

where $g = g_{\max}$. These forms are not unique, since the ranges $0 < f < f_{\max}$ and $0 < g < g_{\max}$ describe other possibilities. It should be noted that the choice of f and g in no way alters the forms of the curves that will be fitted to the measured glow curves II and III by combining $I_A^N(T)$ and $I_B^N(T)$. However, the ratios in which the signal shapes will need to be combined to fit those curves do depend on the values of f and g . With the selection of $f = f_{\max}$ and $g = g_{\max}$, the fitting procedure will, if anything, return overestimates of the intensities of the lesser components in curves I to IV; ie of signal B in curves I and II, and of signal A in curves III to IV. This can only lead to an overestimation of the enhancement factor, $P(T)$, when the data are extrapolated to the limits of signal composition. In other words, the choice of $f = f_{\max}$ and $g = g_{\max}$ ensures that the degree of separation of signal A from signal B by the pre-heat treatment is not underestimated.

The shapes of $I_A^N(T)$ and $I_B^N(T)$, labelled A and B respectively, are shown in fig.1(a) in the correct proportions to form curve I, and in fig.1(d), where they combine to give curve IV. Fits to the measured curves II and III using curves A and B are shown in figs.1(b) and 1(c) respectively. It should be noted that both the shoulder in curve II, which appears at 310°C, and the peak in curve IV, 25°C higher in temperature, are manifestations of the same signal (curve B). The shoulder at 310°C is common in blue wavelength emissions from sediments.

Implicit in the curve fitting procedure described above are the assumptions that (i) there are only two signals present in the glow curves I to IV, and (ii) that the shapes of signals A and B are not wavelength dependent. If a third signal were present, and if its emission spectrum, glow curve shape and behaviour under sample pretreatment differ from those of signals A and B, its intensity relative to the other two would vary between the curves I to IV. It would then be impossible to fit curves II and III with combinations of $I_A^N(T)$ and $I_B^N(T)$ alone, but a third shape would additionally be required. If, on the other hand, the properties of the third signal are identical to those of either A or B, then for all practical purposes it is not a separate signal at all. The second assumption is also tested by the ability of $I_A^N(T)$ and $I_B^N(T)$ to fit curves as the temperature approaches cut-off. This is not significant, since the re-heat curve which is subtracted from the first glow does not represent a true background in this temperature region, but includes a rising component of actual TL. It is also clear that the fit to curve III betrays problems due to small differences in thermal lag. These points aside, the fitted curves give good descriptions of the measured data. This fact justifies the use of a simple two

measured data. This fact justifies the use of a simple two component curve fitting procedure. As described below, these two signals have been found in a wide range of sediments.

Signal compositions can be expressed in terms of the fraction of signal B present at a given temperature:

$$C_B(T) = I_B^N(T) / [I_A^N(T) + I_B^N(T)]$$

The fitting procedure allows the temperature variations of C_B to be calculated for each of the measured curves I to IV. The results are summarised in fig.2.

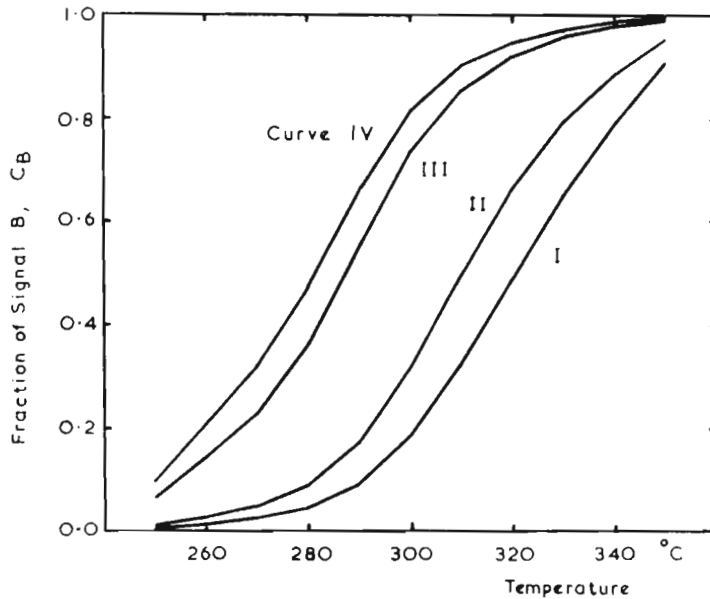


Figure.2. Temperature variations of the fraction of signal B, $C_B = I_B^N / (I_A^N + I_B^N)$, present in each of the measured natural glow curves, I to IV, as derived from analyses of their shapes.

PRE-HEATING MEASUREMENTS

The effects of pre-annealing on the natural glow curves I to IV was measured. Sample discs were held at 230°C for 1 minute on the heater plate of the TL oven. Subsequent glows to 400°C were recorded under the same conditions as the corresponding natural glow curves, and normalised by the same zero-glow method. Figure 3 compares the pre-heated glow curves with the natural curves. It is seen that the forms of the pre-heated curves depend considerably on the natural curve shapes. This fact alone means that they must still be composed of more than one component, and that a single pure signal has not been achieved by the pre-heat treatment.

The fraction of the natural TL intensity remaining after pre-heating,

$$I^P / I^N = (I_A^P + I_B^P) / (I_A^N + I_B^N)$$

was calculated for each curve in 10°C temperature intervals and plotted against signal composition, C_B . As can be seen from fig.2, it is only between 270°C and 320°C that C_B values vary sufficiently among curves I to IV to allow reliable extrapolation to the composition limits. Therefore, data are presented, in fig.4, for just six 10°C temperature intervals in this range. Since only two component signals

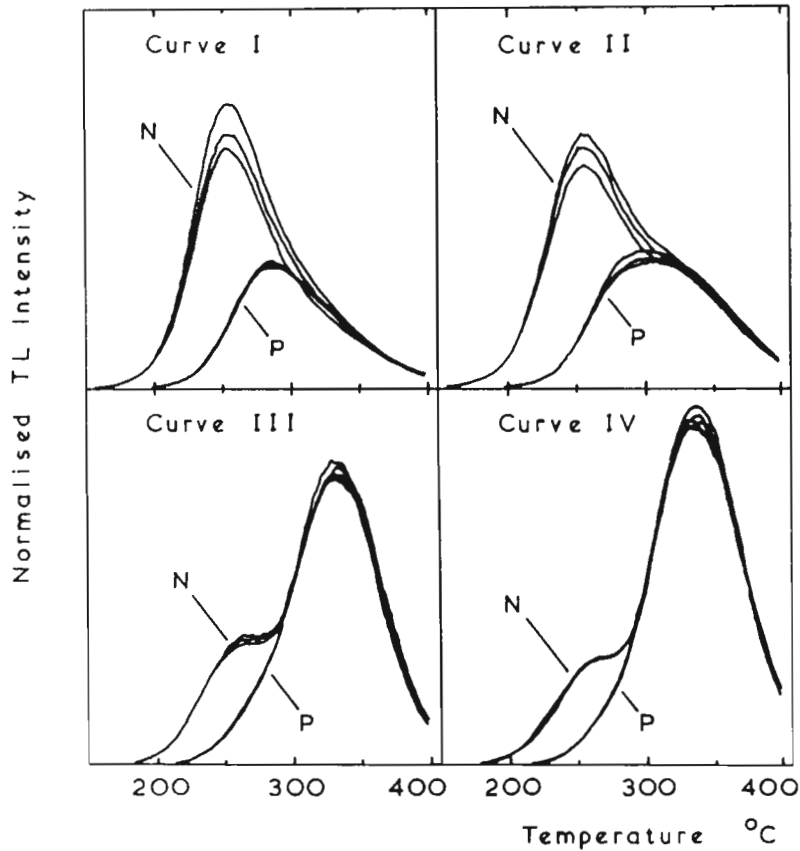


Figure.3. Natural TL glow curves I to IV (N) and corresponding pre-heated glow curves (P) of the sediment PND3. The pre-heat treatment consisted of a temperature hold for 1 minute at 230°C immediately before TL measurement.

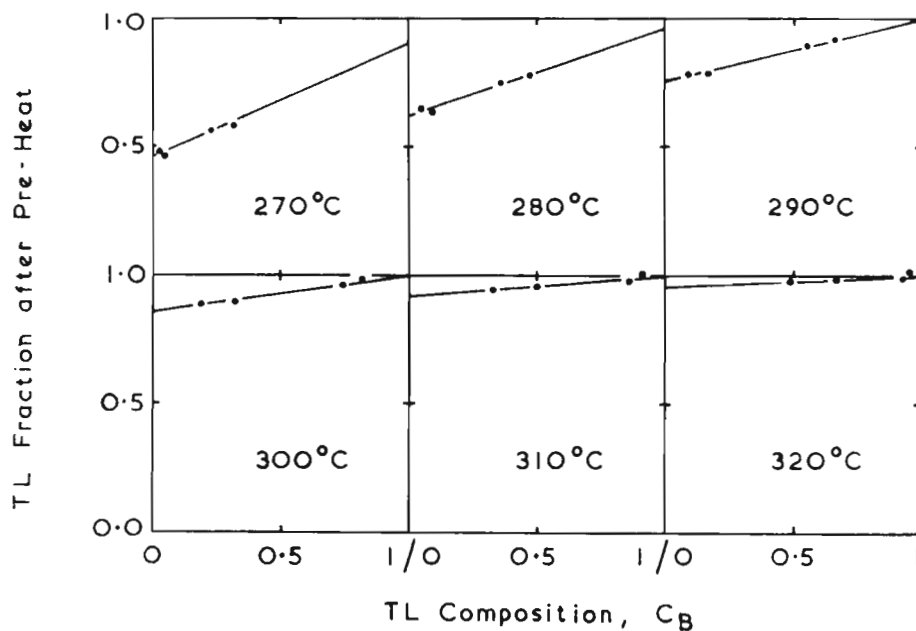


Figure.4. Data, in six temperature intervals of 10°C width between 270°C and 320°C, giving the fractions of TL surviving a 230°C pre-heat treatment plotted against TL signal composition, $C_B = I_B^N / (I_A^N + I_B^N)$, as measured from the glow curves I to IV. Straight line extrapolation of these data to $C_B = 0$ measures the effect on signal A (ie. I_A^P / I_A^N), and similarly that to $C_B = 1$ gives the fraction I_B^P / I_B^N .

are present, I^P / I^N will vary linearly with C_B , and the data have been extrapolated accordingly. The intersections at $C_B = 0$ and $C_B = 1$ then evaluate the ratios I_A^P / I_A^N and I_B^P / I_B^N respectively.

These two fractions are plotted against temperature in fig.5. The factor, $P(T)$, by which signal B is enhanced relative to signal A by the pre-heating, is simply the ratio of these values, and its variation with temperature is also plotted in fig.5.

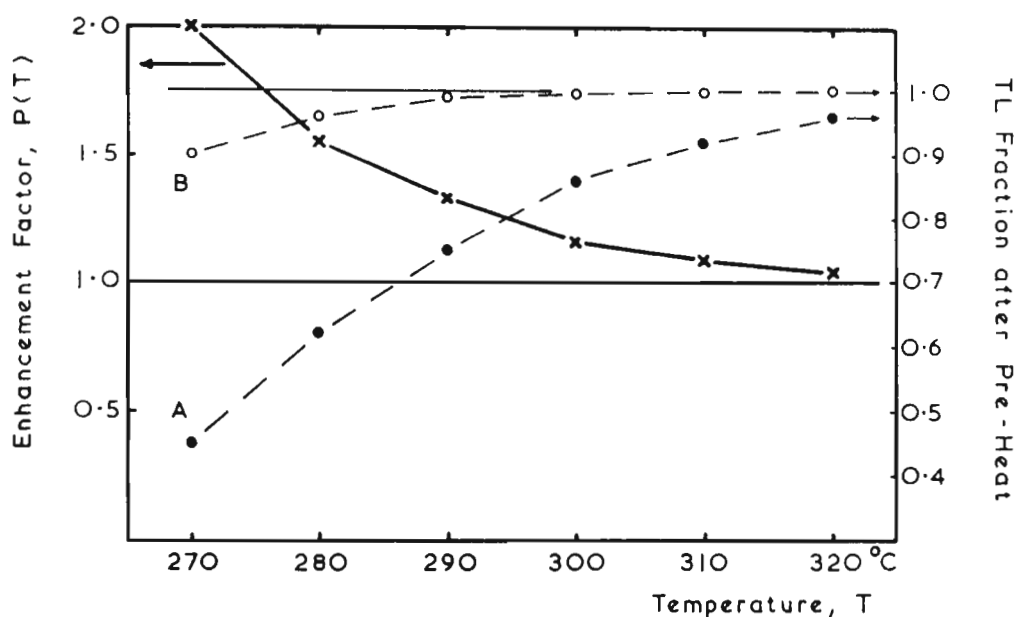


Figure.5. Temperature variations of the fractions of signals A and B surviving a pre-annealing at 230°C for 1 minute, (broken lines), as extrapolated from data given in fig.4. From these is calculated the enhancement factor, $P(T)$, (continuous curve) which gives the increase in the ratio of signal B to signal A resulting from the pre-heat treatment.

DISCUSSION AND CONCLUSIONS

As stated above, the method for deriving the forms of $I_A^N(T)$ and $I_B^N(T)$, whereby f is set equal to f_{\max} and g equal to g_{\max} , will if anything give an overestimation of the enhancement factor, $P(T)$. The values presented in fig.5 should therefore be regarded as upper limits of $P(T)$. These data show that, given a pre-heat treatment at 230°C for 1 minute, useful enhancement of signal B relative to signal A occurs only at glow temperatures below 280°C. However, note from fig.2 that even curve IV, although dominated overall by signal B, derives less than 50% of its TL intensity from that signal at such low temperatures. It is concluded that the claim made for pre-heating (Wintle, 1985), namely, that it totally removes the lower temperature peak, is a false one. This result brings into question the significance of plateau tests performed on pre-annealed samples, since improved plateaux have been taken to indicate a purification of the measured signal.

It has been shown that fine-grain natural glow curves of the sediment PND3 are composed of two signals only. Previous work has shown that, while additional signals are present in a small proportion of samples, only signals A and B are

invariably present in all sediments as major components of their fine-grain emissions. Signal A is best observed in the UV region, and its properties have been described in a previous paper (Debenham, 1985). As observed in that paper, the position of the rising edge of its glow curve depends on the age of the sediment and the past ambient temperature of the site. Signal B is enhanced relative to signal A by treatment of fine-grains with fluorosilicic acid and by observing at blue wavelengths, but its potential for dating sediments is limited because of its resistance to bleaching and its early saturation. A signal at 290°C, introduced without explanation by Wintle (1985), has not been observed. The properties of composite TL emissions are complex, and careful investigation should be made before citing them as evidence for new signals. It is suggested that the technique of curve fitting described above provides a practical method for seeking additional components within the TL emissions from sediments.

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- Wintle, A.G. (1985) Stability of TL signal in fine grains from loess. *Nucl. Tracks*, **10**, 725-730.

PR Reviewed by Dave Huntley

THE EFFECT OF PRE-ANNEALING ON SEDIMENT TL

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Post-irradiation pre-readout annealing is used extensively to remove low-temperature peaks in dosimeters such as LiF:Mg,Ti and CaF₂:Dy. These peaks are not required because they are thermally unstable at ambient temperature. The extension of such a pre-heat procedure to minerals being used for dating was first reported by Mejdahl and Winther-Nielsen (1982). They applied it to 100-300 μ m alkali feldspars extracted from pottery and it has more recently been used for 100-300 μ m potassium feldspar and quartz grains separated from sediments (Mejdahl, 1985). Valladas and Valladas (1982) used a preheat procedure in their study of quartz.

The application of a preheat procedure (230°C for 1 minute on the TL oven plate or more recently 150°C for 16 hours on a separate hot plate) to fine grain polymineral sediments (Wintle, 1985) was introduced to reduce the response of the TL between 150 and 280°C (heating rate = 5°C/s). When a Schott UG-11 filter is used, the TL in this part of the glow curve is enhanced relative to that above 280°C. The latter is the temperature region where stability of the trapped electron signal might be expected, and indeed onset of the plateau region before 300°C was reported by Debenham (1985) for a large number of sediments of different ages. I therefore wished to eliminate the high response of the TL in the 150-280°C region since this, combined with small amounts of thermal lag, gave rise to considerable scatter in the growth curves obtained for 10°C temperature intervals. This was reduced by the preheating.

The preheat procedure (230°C for 1 minute) was chosen such that after its application both the natural TL and a matching TL signal regenerated by irradiation after optical bleaching have the same glow peak temperature. This is at about 290°C. As shown in Fig. 4(a) of Wintle (1985) this is not achieved by preheating at lower temperatures. When such a match is achieved an ED plateau is often obtained throughout the region from 220-400°C. The presence of a well-defined peak has the added advantage that any curves which have been apparently shifted to a higher temperature due to thermal lag (eg caused by grains beneath the disc) can be shifted down by an amount corresponding to the difference in peak position.

It is clear from the shape of the glow curves that one is not dealing with a single glow peak obeying either first or second order kinetics. However the glow curves may be interpreted as being due to distributions of electron traps, with increasing trap depths found for increasing glow curve temperatures (as found by Strickertsson (1985) for separated potassium feldspars). If these curves are thought of as being composed of such distributions, then holding at 230°C for 1 minute (by analogy with the decay of the pure first order 110°C peak in quartz) will remove about half of the TL which would have a peak at 280°C and all of that with a peak at 230°C.

The more general approach taken by Debenham (1987) is an interesting one in which only two component signals (designated A and B) are fitted to the natural glow curves of a sample; they are applied to curves obtained after two different sample preparation procedures and when two different parts of the emission spectra are observed. This empirical approach is totally separate from any kinetic behaviour. It implies that the higher peak (330°C for 5°C/s) from a sample treated with H₂SiF₆ for 3 days at ambient temperature is the same as that of the dominant TL signal above 300°C obtained for a polymineral signal. It should be pointed out that although this treatment does not attack the quartz (Chapman et al (1969) obtained 97% recovery of 2-20 µm quartz) it may not totally eliminate all the feldspars. Chapman et al (1969) found a 48% recovery for albite (high Na feldspar), 34% for microcline (a K feldspar) but less than 0.1% for anorthite (a high Ca feldspar) in the same grain size. This treatment was first used by Berger et al (1980) and subsequently by Berger and Huntley (1982) and Wintle (1982). The drastic change in shape of the glow curves that results from this treatment indicates a change in the mineralogical composition.

Wintle (1982) thought that the peak given by the H₂SiF₆ treated sample was not totally responsible for the upper peak of the natural TL of the untreated sample when observed with the same filter (a Corning 5-58 blue glass). It appeared to occur some 30°C lower. A similar temperature difference has been reported for the glow peaks of separated 100-300 µm quartz and potassium feldspar (Mejdahl, 1986). In this context it is interesting to note that these two peaks have been shown to have different behaviour eg the bleaching of the TL by visible light is more rapid for potassium feldspars than for quartz and the TL signal of quartz saturates at a lower radiation dose level than does the feldspar. Since these differences in response are also observed for the two extreme sets of glow curves (I and IV of Debenham (1986)), it may cast doubt on the more widespread application of the two signal approach.

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PI Reviewer's Comments (Martin Aitken)

It is useful to have this paper following soon after Debenham's on the same topic since the author makes clear that the genesis of her pre-heat procedure was the practical desirability of obtaining TL glow curves for natural and artificial that had the same peak temperature. It is reassuring to note that Debenham (fig 3a) finds the same peak temperature (290°C) for the same pre-heat procedure. He interprets this glow curve as a composite of the two basic shapes, A and B, and he concludes that A contributes despite the pre-heat. While this calls into question the statement in Wintle (1985), that the pre-heat totally removes the TL from the lower peak it does not detract from the practical advantages of the pre-heat indicated in the present paper.

In all this discussion we are hampered by inadequacy of nomenclature. Does 'a peak at 290°' mean a maximum in a composite glow curve or a signal from a single trap type? In the latter case how do we indicate trap type having a distribution of depths? We need a TL lexicographer - if that's the right word!

KINETIC STUDIES OF QUARTZ THERMOLUMINESCENCE AS APPLIED TO SEDIMENT DATING

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INTRODUCTION

The calibration of the radiation sensitivity of material with the help of powerful laboratory radiation sources is a fundamental component of the thermoluminescence (TL) dating method. However, the accuracy of using such sources is poorly substantiated, even though it is of special importance since there is a change in dose rate of 6-8 orders of magnitude when transferring from natural to laboratory conditions. Studies of this problem have been one of the principal objectives of our research work. In addition, we have investigated the use of ultra-violet light to bleach the TL stored in minerals since bleaching is the dominant process for zeroing the TL chronometer in sediment dating. To study palaeodosimetric problems more precisely, we focussed our attention on quartz, taking the 300°C TL peak lightsum (integrated emission) as a basic unit of measurement. We also studied the behaviour of the 180°C TL peak which is absent in the natural glow curve, but present with high sensitivity after artificial radiation .

EXPERIMENTS AND DISCUSSION

Our studies have confirmed that the initial section of the dose response curve is complicated by a form of supralinearity (fig. 1.). Unlike data published elsewhere, which states that this phenomenon is true for a small proportion of samples, we have found supralinearity in all the samples we have studied. In a number of cases it even transforms into a section with negative sensitivity and subsequent normal growth after passing through a minimum value (fig. 1, curve 2). Such a phenomenon is called by us a "dose pit" and is typical for one third of the samples under study.

Good correlation between the level of dose at which the 300°C peak supralinearity finishes and that at which onset of saturation of the 180°C peak occurs can be seen in figure 1. Such behaviour can be explained by shallow traps acting as a competitive mechanism. The dose-pit phenomenon can also be explained within the framework of this theory if a radiation-induced decay component is taken into account as well as a thermal decay component, which is small for deep traps. This process can be described by the following formula:

$$\eta = 1 - (1 - \eta_0) \exp(-\kappa D) - p[1 - \exp(-q\kappa D)]$$

which is presented graphically in figure 2, where

η = normalised trap population relative to the saturation level,

κ = radiation sensitivity,

D = absorbed dose, and

p and q = parameters characterizing the competing decay processes.

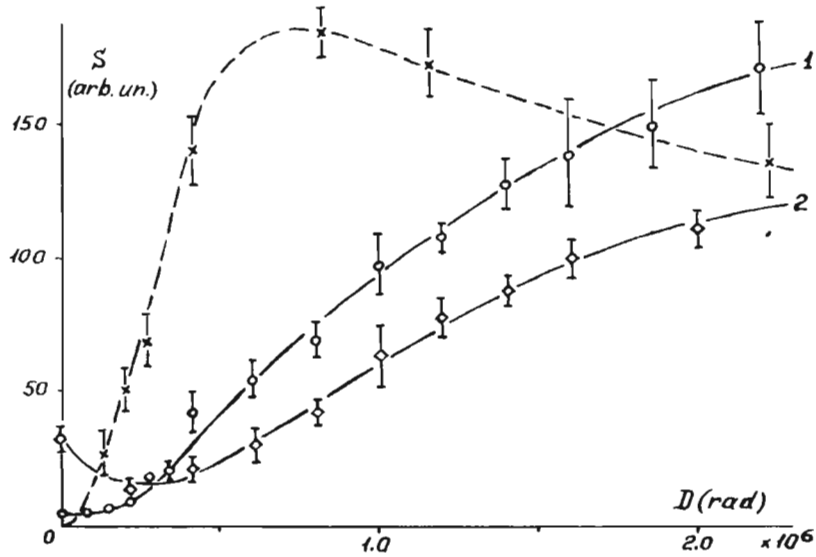


Figure 1. Dose dependencies of a lightsum of the 300°C (solid line) and 180°C (broken line) peaks of the quartz TL showing; 1) simple supralinearity and 2) the "dose-pit".

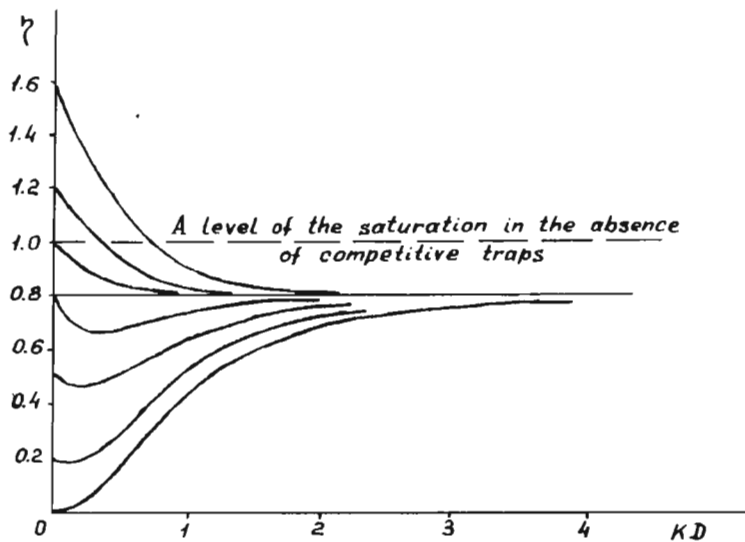


Figure 2. Theoretical dose dependencies in the presence of competing thermal and radiation fading.

Since the 180°C peak is absent from the natural glow curve, we have every reason to assume absence of supralinearity under natural irradiation conditions (i.e. during antiquity). Under laboratory conditions, in addition to the 180°C peak, there are other peaks present at lower temperatures which may be capable of yielding an analogous effect.

When observing changes in the lightsum at 300°C using doses of up to 5 krad, we have also observed complex fine structure which is difficult to characterize. We presume this arises from competition processes within the trap spectrum which introduce additional distortions into a laboratory dose curve.

At higher doses (above 100 krad) we have also observed an "abnormal" signal in the region of the 300°C peak, which is capable of being several times greater than the "normal" component. The main distinguishing feature of this abnormal TL signal is its insensitivity to UV radiation and its generation only under artificial ionizing radiation.

On the basis of these studies, we came to the conclusion that there were fundamental differences in the TL response of samples under natural and laboratory irradiation and that this brings into doubt the validity of established methods of using quartz as a palaeodosimeter in which the natural TL is compared with the response to laboratory irradiation.

The Effect of UV Radiation

In addition to de-activation of the 300°C peak, we have found that UV irradiation can also cause activation. The latter has been observed in the form of a regenerated 300°C peak after complete thermal annealing of the sample (450°C, 10 min.). Lower temperature peaks, similar to those produced by gamma irradiation, are also produced. The growth of the 300°C peak lightsum with UV dose is not of a simple exponential form and the equilibrium level is the result of competing UV-stimulated activating and deactivating processes. Under conditions of solar exposure at middle latitudes, the UV equilibrium lightsum is achieved after one month of exposure. This value, which may serve as a "theoretical zero" of the TL chronometer for sediments, is easily reproduced under laboratory conditions using unfiltered light from a 120W mercury lamp (@ 25cm for 1h).

RESULTS

Our research work has resulted in the development of a new approach to TL dating, the main feature of which is the exclusion of individual radiation calibration of samples. In the absence of competition by shallow traps and "abnormal" TL, the dose dependence of the 300°C peak lightsum follows a simple law of saturation. At the present time, we do not possess sufficient data to establish the order of the kinetics. However, use of two extreme possibilities, with either first or second order kinetics, yields no more than a 20% difference (fig. 3) in the age calculation. For first order kinetics the following exponential growth curve is obtained:

$$S = S_{\infty} - (S_{\infty} - S_0) \exp(-\kappa D)$$

where: S = a lightsum, and "0" & "∞" subscripts indicate the initial state and level of saturation respectively.

When determining D, the absorbed dose, for a given value of S₀ it is necessary to know the two parameters κ and S_∞. κ is a physical constant universal for a given crystalline lattice with a particular trapping centre, and may be determined in advance and used for all the samples. Individual sample properties determined by particular trap concentrations are reflected in the S_∞ parameter.

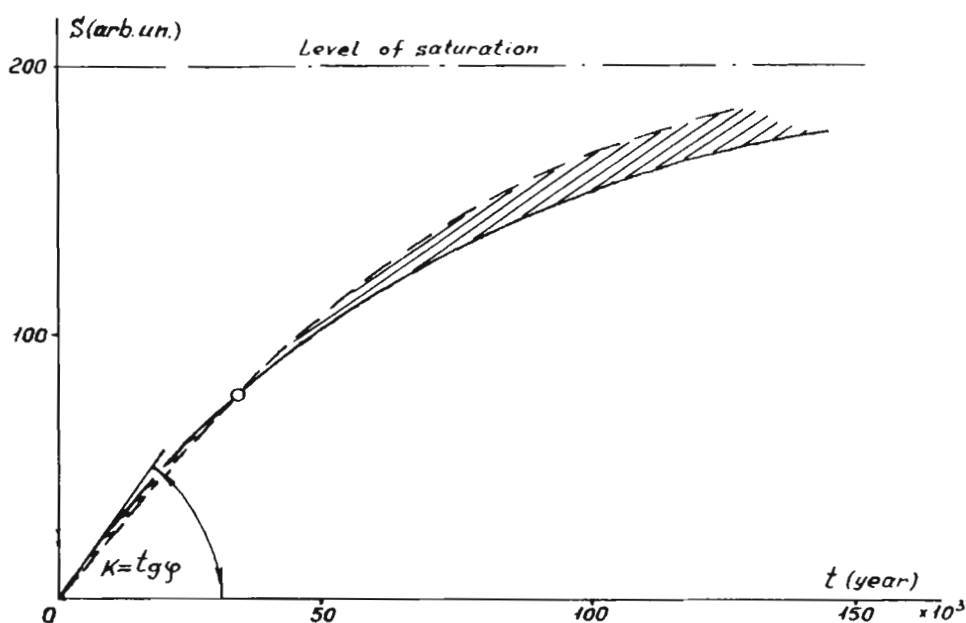


Figure 3. Dependence of the lightsum arising from natural irradiation;

- 1st order
- - - 2nd order
- //// limits of uncertainty
- o calibration point

The analysis of samples from a collection covering a vast area of the European part of the USSR has revealed that for samples with ages ranging from the beginning of the Middle Pleistocene to the Upper Cretaceous, the natural glow curves are identical within experimental error limits. We thus infer that they are all in saturation, and can be used to obtain S_{∞} for this area. In this way, one of the unknown parameters has been provided by nature itself. A contradiction between a measured saturation of 300-400 ka and a theoretical prediction of 2-3 Ma, based on the thermal stability of the trapped electrons, should be noted. We can explain this contradiction by the exclusion of a radiation-induced decay component in the theoretical model; however, its existence has been recorded by us experimentally. Hence early saturation testifies to the predominance of a radiation-induced decay component over a thermal one.

Having established S_{∞} , the radiation sensitivity value κ can now be obtained for a sample of known age (dated by ^{14}C). We estimate κ to be $4 \cdot 10^{-4} \text{ rad}^{-1}$ when the age, t , and the in-situ dose-rate, E , are inserted in the equation above with $D = t \cdot E$.

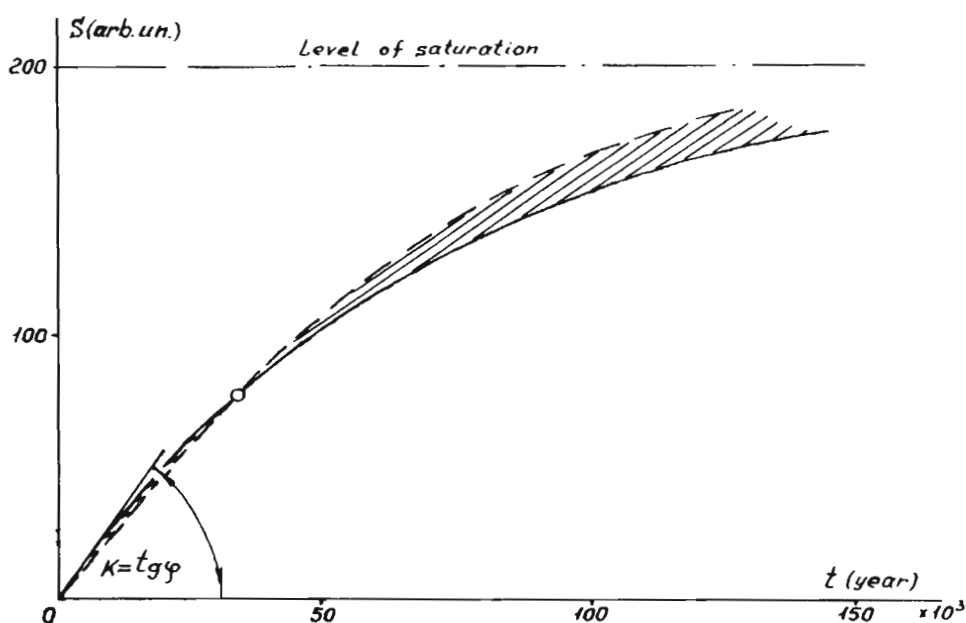


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The analysis of samples from a collection covering a vast area of the European part of the USSR has revealed that for samples with ages ranging from the beginning of the Middle Pleistocene to the Upper Cretaceous, the natural glow curves are identical within experimental error limits. We thus infer that they are all in saturation, and can be used to obtain S_{∞} for this area. In this way, one of the unknown parameters has been provided by nature itself. A contradiction between a measured saturation of 300-400 ka and a theoretical prediction of 2-3 Ma, based on the thermal stability of the trapped electrons, should be noted. We can explain this contradiction by the exclusion of a radiation-induced decay component in the theoretical model; however, its existence has been recorded by us experimentally. Hence early saturation testifies to the predominance of a radiation-induced decay component over a thermal one.

Having established S_{∞} , the radiation sensitivity value κ can now be obtained for a sample of known age (dated by ^{14}C). We estimate κ to be $4 \cdot 10^{-4} \text{ rad}^{-1}$ when the age, t , and the in-situ dose-rate, E , are inserted in the equation above with $D = t \cdot E$.

The age of an unknown sample may now be calculated with the following formula,

$$t = (\kappa E)^{-1} \ln \{ (S_{\infty} - S_0) / (S_{\infty} - S) \}$$

where, E , the in-situ dose-rate, was determined using an industrial scintillating (NaI) prospecting radiometer. The S_0 value (a theoretical zero) was taken to be the lightsum remaining after a three hour exposure performed with the mercury lamp. We assume that the error arising from incomplete zeroing under natural conditions is small compared with the age being measured.

The method was tested using material from Middle-Late Pleistocene deposits from the Central Russian Plain and the lower Volga. With a few exceptions, the results obtained show good internal consistency and compare well with geological estimates of the age.

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ESR Dates

ESR dates relating to the age of the Holsteinian Interglaciation have been reported in two recent papers

- Linke, G., Katzenberger, O. and Grun, R. (1986) Description and ESR Dating of the Holsteinian Interglaciation. *Quaternary Science Reviews*, **4**, 319-331.
- Sarnthein, M., Stremme, H.E. and Mangini, A. (1986) The Holsteinian Interglaciation: Time-stratigraphic position and correlation to stable-isotope stratigraphy of deep-sea sediments. *Quaternary Research*, **26**, 283-298.

Both studies were carried out on molluscs, but the resulting ages were different by about 100 ka.

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