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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:

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

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 $P(T) >> 1$. 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.

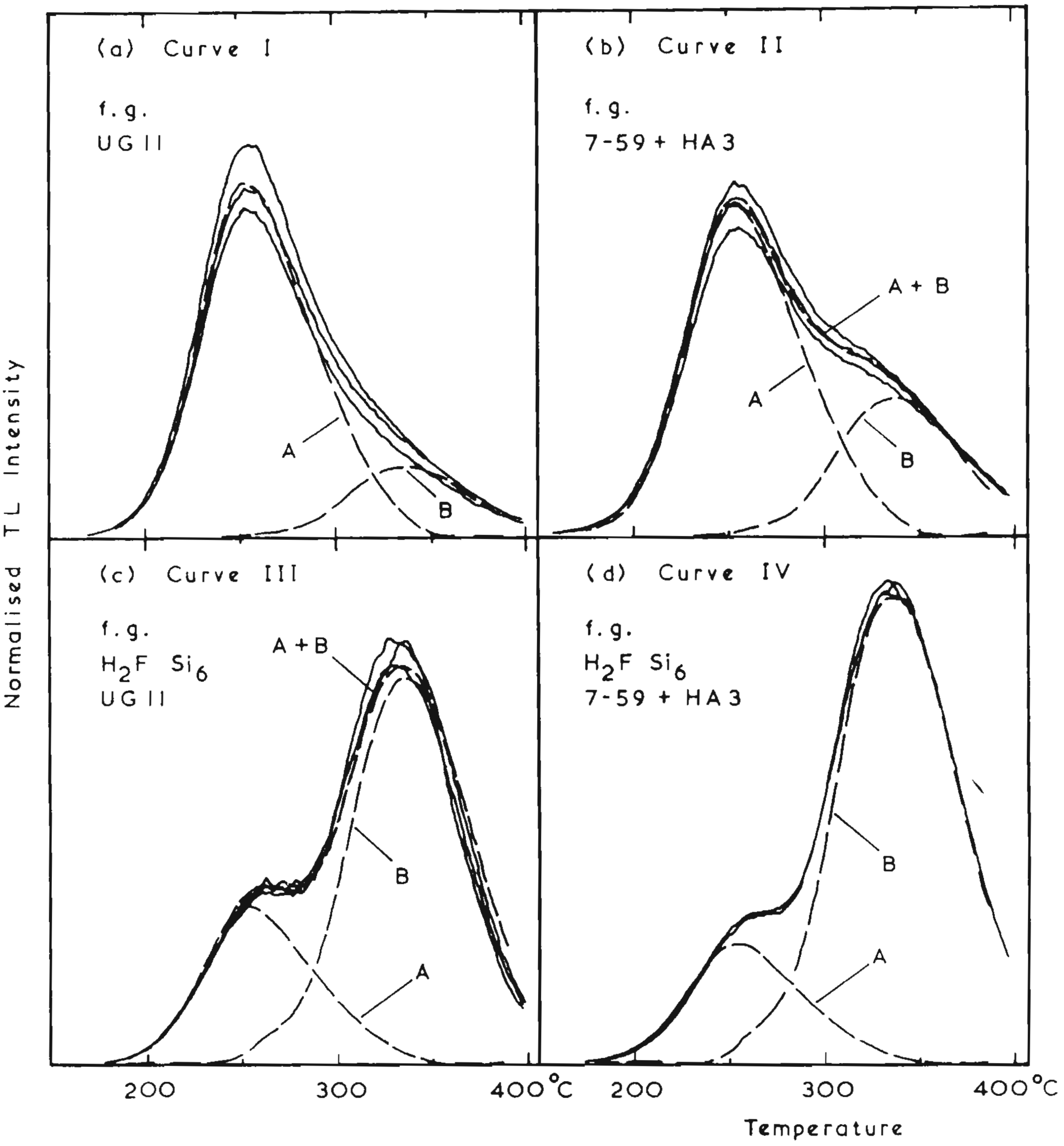

Figure.1. *Continuous lines show natural TL glow curves, labelled I to IV, of fine-grains from sediment PND3, normalised by the zero-glow method, and measured at 2.5°C/s heating rate. Broken lines give the shapes of signals A and B as derived from curves I and IV, and their summations, A+B, as fitted 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:

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

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:

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

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.

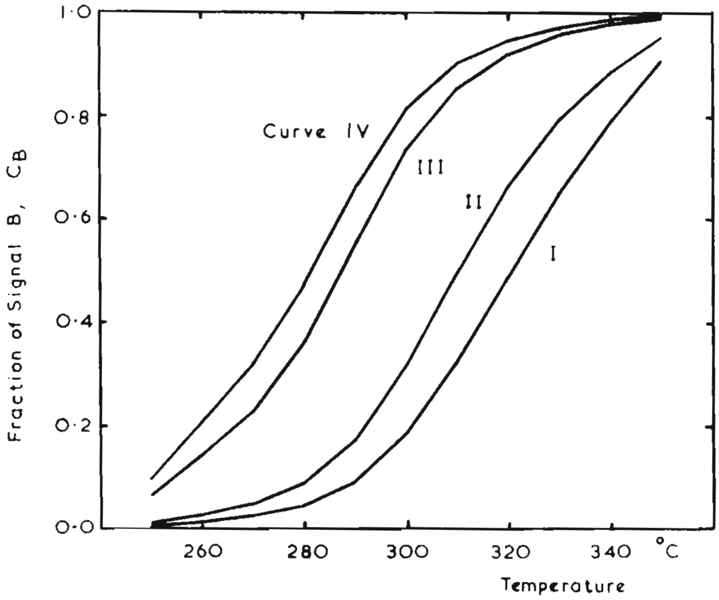

Figure.2. Temperature variations of the fraction of signal B, $C_B = I_B^N / (I_A^N + I_B^N)$, present in each of the measured natural glow curves, I to IV, as derived from analyses of their shapes.

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

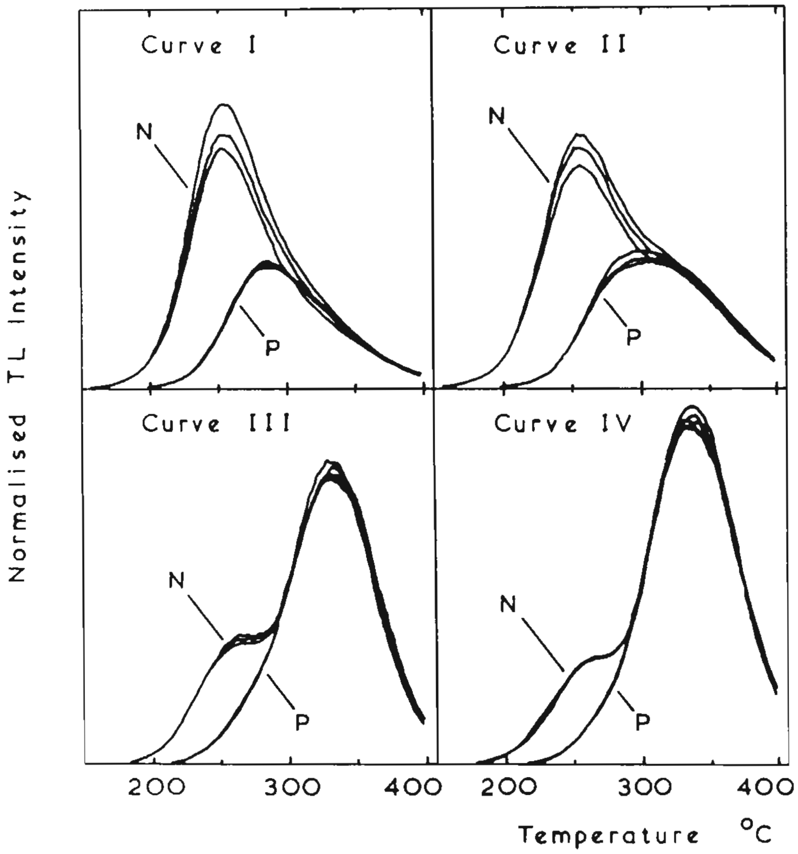

Figure.3. Natural TL glow curves I to IV (N) and corresponding pre-heated glow curves (P) of the sediment PND3. The pre-heat treatment consisted of a temperature hold for 1 minute at 230°C immediately before TL measurement.

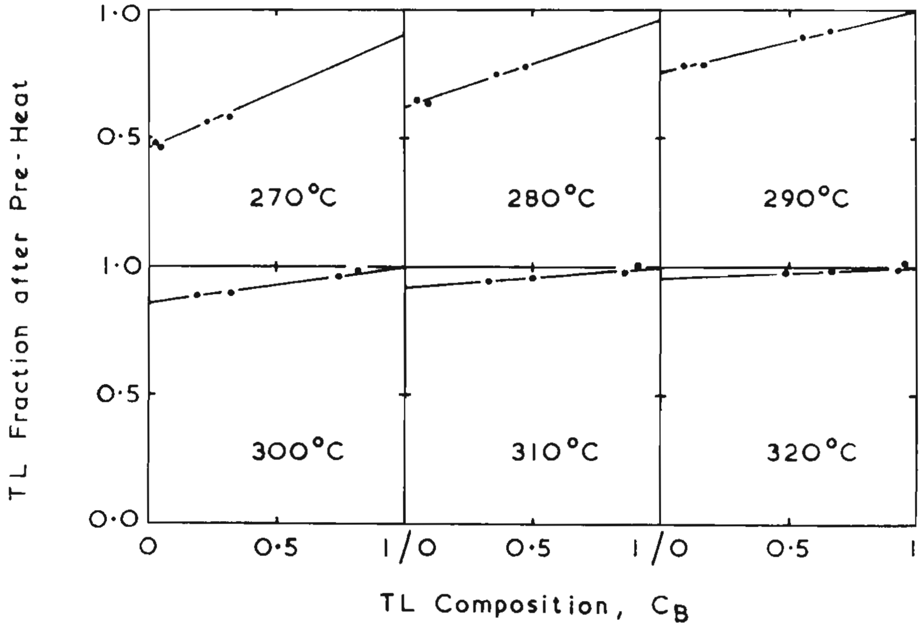

Figure.4. Data, in six temperature intervals of 10°C width between 270°C and 320°C, giving vthe fractions of TL surviving a 230°C pre-heat treatment plotted against TL signal composition, $C_B = I_B^N / (I_A^N + I_B^N)$, as measured from the glow curves I to IV. Straight line extrapolation of these data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

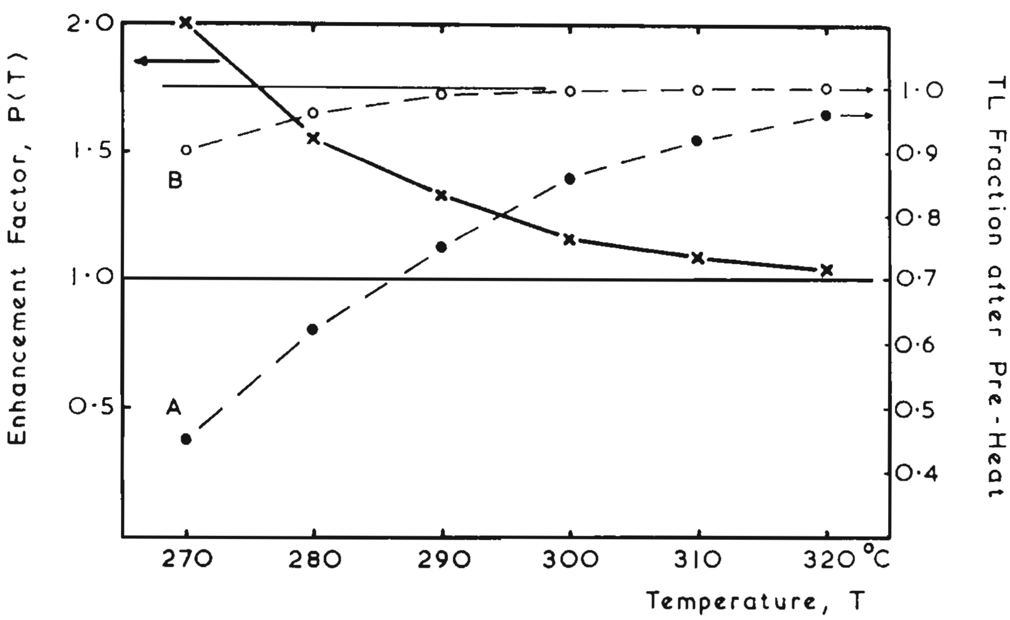

Figure.5. Temperature variations of the fractions of signals A and B surviving a pre-annealing at 230°C for 1 minute, (broken lines), as extrapolated from data given in fig.4. From these is calculated the enhancement factor, P(T), (continuous curve) which gives the increase in the ratio of signal B to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