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Pre-heats, Palaeodoses and Paradigms in the Optical Dating of Quartz: Some Comments on Roberts et al. (1993) 'Cautions on the use of Extended Duration Pre-heats in the Optical Dating of Quartz'

Stephen Stokes

Research Laboratory for Archaeology and School of Geography,
Oxford University
6 Keble Road, Oxford, OX1 3QJ.

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In a recent issue of Ancient TL, Roberts et al. (1993) described a series of experiments on the OSL of quartz extracted from an archaeological sediment context in Australia. Their discussion ultimately indicated that for their sample, long duration, relatively low temperature (c. <200°C) pre-heats result in palaeodose underestimation. I question their assertion that differences between the OSL growth curves presented are attributable to dose-dependent sensitivity changes, and their implication that palaeodoses generated using an extended duration pre-heat will consistently be in error.

As a result of their study, I have embarked on my own intercomparison of long versus short duration pre-heats on 45 samples. These data are presented in a companion paper (Stokes, 1996). My investigation indicates that there is no tendency for one or other of the pre-heats to give a systematic over or under estimation. Further to these new data, I additionally think that the following points relating to Roberts et al (1993) should be considered in the context of their claims.

a. Despite their suggestion, the two pre-heat procedures are not kinetically equivalent. If, as Roberts et al. (1993) acknowledge, the bulk of the OSL in quartz is derived from the 325°C TL peak would it not be appropriate to predict the extent of thermal depletion from E and s values of that trap? Instead they arbitrarily assign an E value of 1.61 eV. Wintle (1975) calculated a depth of 1.69 eV for the 325°C trap. 160°C, 16 hour and 220°C, 5 minute pre-heats would therefore be expected to reduce trapped charge populations by 12.5 and 16.1% respectively. When considered in isolation, erosion of the 325°C trap should be greater for the 220°C pre-heat than for the 160°C, treatment and not equal.

As a test for the occurrence of the post pre-heat 'OSL excess' such as that observed by Roberts et al. (1993), I have measured the initial OSL of 28 samples, both prior to and following pre-heating (23 of which were administered both pre-heats). This ratio is plotted in Figure 1. Of the 23 comparable samples, 8 exhibited a post 160°C pre-heat OSL which was in excess of that following the 220°C pre-heat, all others exhibited greater intensities following the 220°C procedure. While a minority (c. 25%) of samples indicate higher OSL intensities following pre-heating, the bulk of the samples indicate nett depletion, in some cases by up to 60%. Clearly, the pattern of trapped charge re-adjustment caused by pre-heating is considerably more variable and complicated than inferred by Roberts et al. (1993), and their data on a single sample is probably misleading. The presence of non-quartz microinclusions in some samples may further complicate the picture.

b. Given the remarkably high degree of precision which they claim, it must be asked why their suggestion of a 15% sensitisation effect is not observable in the TL data presented in their Figures 1 and 2. The influence of a clearly subordinate 375°C peak is not considered to be a satisfactory explanation, particularly given that they acknowledge that the mean TL intensities for the pre-heated aliquots are indistinguishable, yet still use these data to infer sensitisation.

c. In relying on the 110°C TL peak for evidence of dose-dependent OSL sensitivity change the authors assume (as did Stoneham and Stokes, 1991) that the changes are synchronous. Further investigations (e.g., Jungner and Botter-Jensen, 1994; Stokes, 1994) clearly showed this not to be the case, and that

OSL sensitivity changes in quartz are triggered by the bleaching to which aliquots are subjected subsequent to additive dose dating measurements. Of course, although the samples on which I have worked included one from NW Australia (OX$_{OD}$778M8), the Roberts et al. sample may have different characteristics.

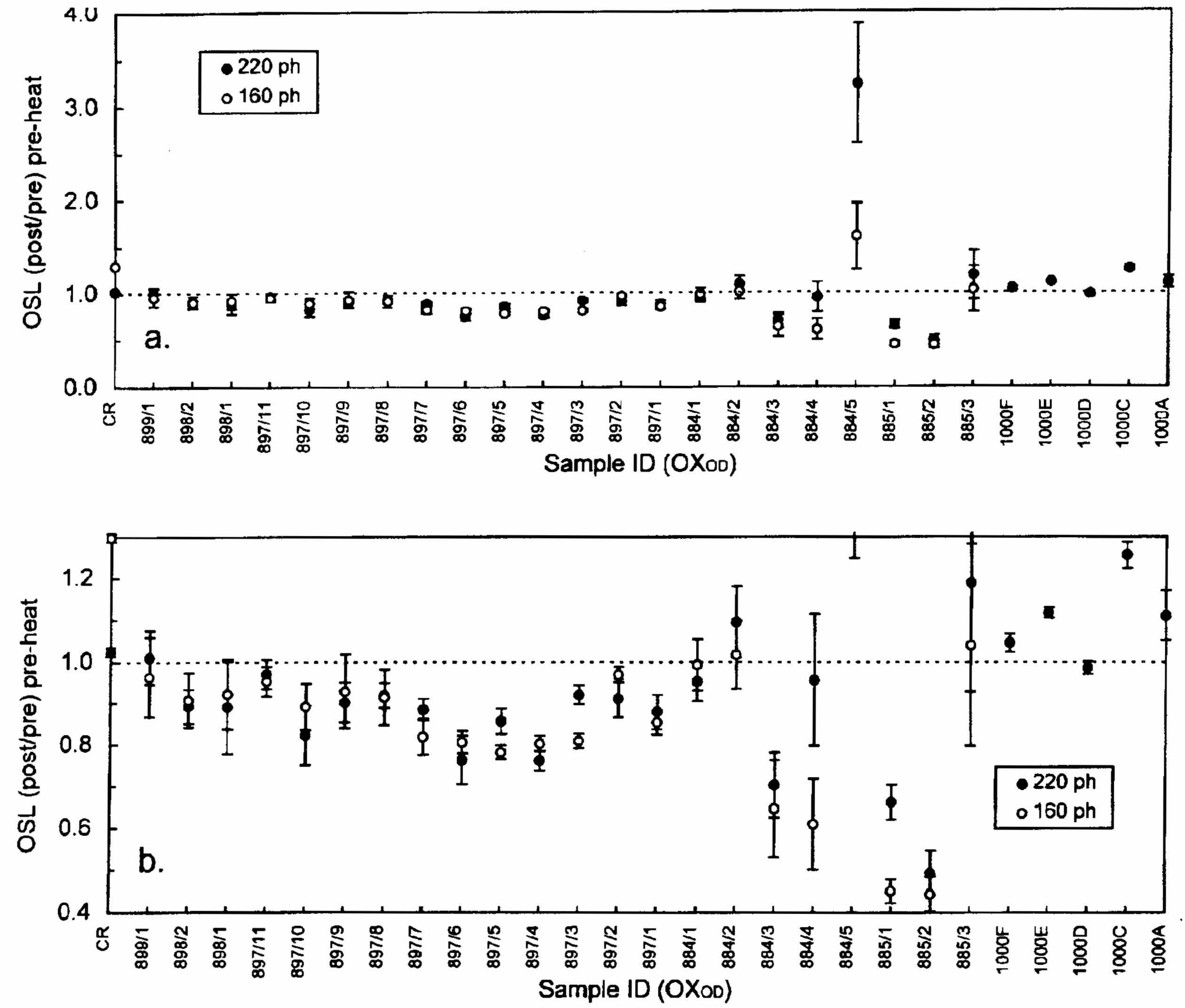

Figure 1.

Ratio of the natural OSL intensity before to that following 220°C, 5 minute (filled symbols) or 160°C, 16 hour (open symbols) pre-heat (ph) treatments for a selection of 28 samples (CR = chaperon rouge). a. All data plotted; b. Data plotted for a restricted range of y values.

d. In contrast to other workers (e.g., Smith and Rhodes, 1994; B.W. Smith, pers comm.), Roberts et al. dismiss thermal charge transfer as a significant process. While the first two lines of argument which they use are marginal to the discussion at best, their third (and 'most direct') line of evidence is somewhat problematic when it is considered in the context of other data presented in the paper. Despite subsequently indicating that regenerative procedures yield palaeodose estimates similar to those generated using additive dose methods, the data in their Table 2 indicates a 91.8/11.2 fold increase in sensitivity upon bleaching. Such a bleach-induced sensitisation would result in a regenerated palaeodose estimate somewhat closer to 0.12 Gy than the 23.5 Gy which they quote. Even if the numbers which they present were sensible, the experimental strategy is neither direct nor compelling evidence to negate charge transfer.

e. Other researchers have documented that dose-dependent sensitivity changes can occur for quartz (and other minerals) whether the 160°C, 16 hour or the 220°C, 5 minute pre-heat treatments are used (e.g, Rhodes, 1990; Duller, 1992; Stoneham and Stokes, 1991; Jungner and Botter-Jensen, 1994; Stokes, 1994). All these previous studies have quantified the degree of dose dependent sensitivity change by comparing ratios (after/before dating) of normalisation factors as a function of added dose. While this does not give a direct insight into the timing of the sensitivity changes, it does provide a means of establishing the occurrence and rate of sensitivity change as a function of added dose. If Roberts et al. wish to suggest that dose dependent sensitivity change is responsible for the apparent effects which they describe then they should at least provide the reader with a measure of its extent and demonstrate its occurrence only for the extended duration pre-heats.

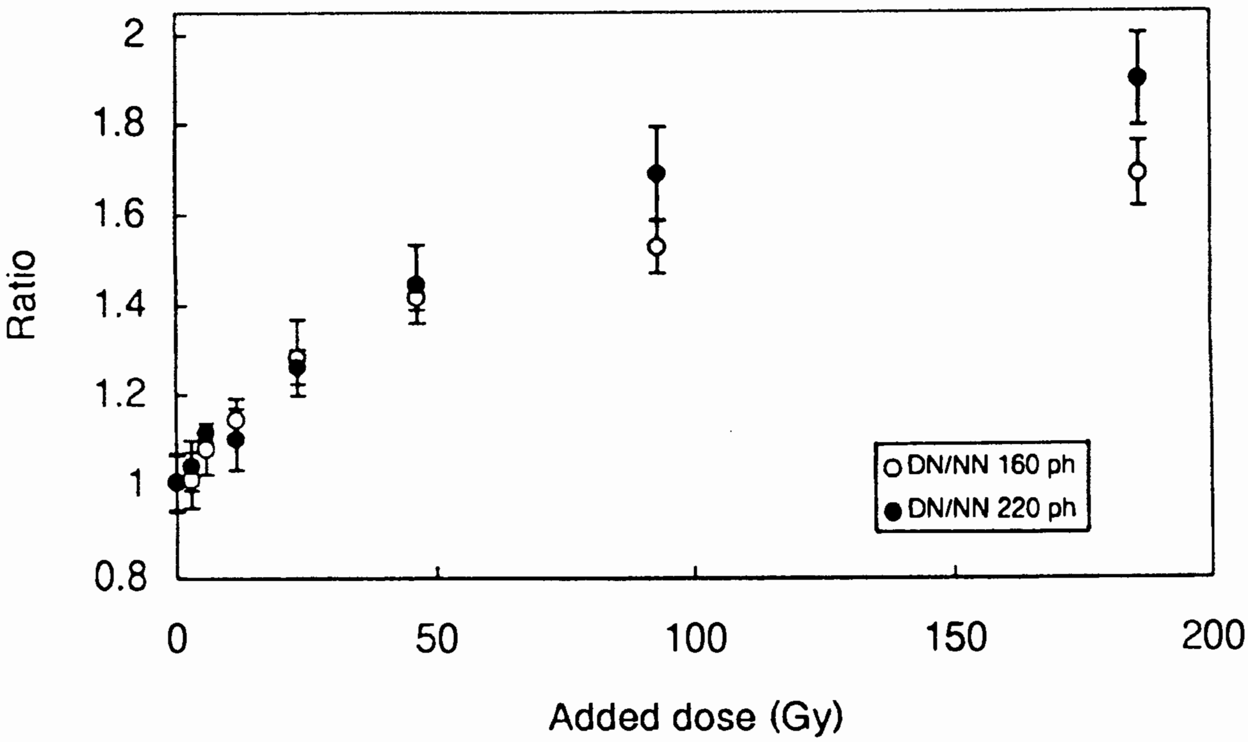

Figure 2

Ratio of $\left(\dfrac{\text{dose normalisation (DN)}}{\text{natural normalisation (NN)}}\right)$ *versus added dose for a dune sample from Chaperon Rouge, Morroco (CR in Figure 1). Filled symbol - 220°C, 5 minute pre-heat, open symbol - 160°C, 16 hour pre-heat. Each data point plotted is an average of 8 aliquots (scaled to unity for average natural aliquot intensity); error bars - 1 standard deviation. When a linear least squares fit is applied to the data in the additive dose range 0-45 Gy, dose dependent sensitivity changes of 0.95 and 0.92 %.Gy*$^{-1}$ *are estimated for the 220°C, 5 minute and 160°C, 16 hour pre-heats respectively. Aspects of the behaviour of this sample have also been described by Rhodes (1990), Roberts et al. (1994) and Smith and Rhodes (1994).*

I have generated such data for one of the samples which they subesquently describe (Roberts et al., 1994). The ratio of normalisation factors measured before and after dosing, pre-heating and laser exposure, plotted as function of added dose is provided in Figure 2. Both sample treatment procedures clearly indicate dose dependent sensitivity changes of the order of 1 %.Gy^{-1} (when measured over a small initial range of added dose). In both cases, at higher doses the degree of sensitisation reduces, as also noted by Stoneham and Stokes (1991).

Clarification of the points raised above would facilitate a more reasoned assessment of the information presented in Roberts et al. (1993). While they present important data which may indicate that palaeodose estimates may partly be controlled by the selection of pre-heat procedures, at least for the sample which they describe and for five subsequent samples (Roberts et al., 1994), to reach the unsubstantiated assertion that it is caused by dose-dependent sensitivity changes is most unhelpful in the collective long term goal of developing robust procedures for routine use of the optical dating method as a Late Quaternary geochronometer.

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