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

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

Introduction

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

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

Luminescence dating procedures

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

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

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

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

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

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

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

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

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

Preheat experiments on $Ox_{OD}K166$

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

1. OSL shine-down curves

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

These results show that the OSL signal is eroded slightly (6.5%) by the 220°C preheat whereas the 160°C preheat induces an 8.6% increase. While the 220°C preheat result is consistent with expectations from thermal erosion of the OSL signal (Spooner, unpub. data), the 160°C preheat result suggests that sensitisation has occurred. This sensitisation may be as much as 15% if both preheats initially eroded the OSL signal (i.e. the 325°C TL peak) to the same extent.

2. TL glow curves

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

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

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

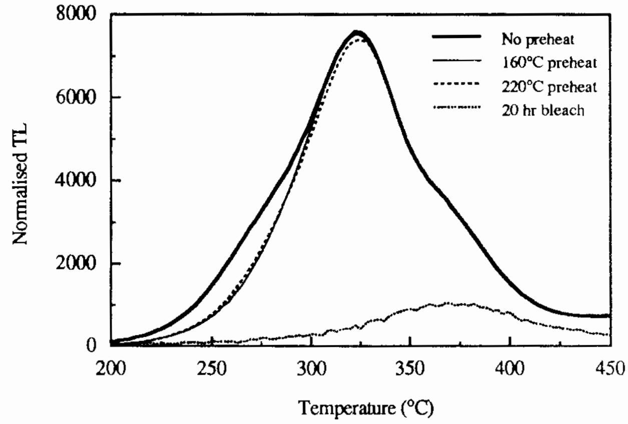

Figure 1.
Glow curves for natural (unpreheated), preheated and bleached aliquots of Ox_{OD}K166. Glow curves for aliquots preheated at 160°C for 16 h or at 220°C for 5 min are shown. At glow curve temperatures <300°C, the 160°C curve overlies the glow curve obtained for aliquots preheated at 190°C for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

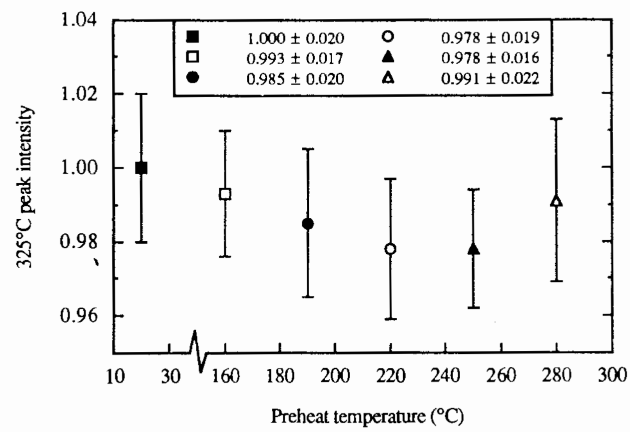

Figure 2.
TL intensity of the 325°C peak (315-335°C peak area integration) for natural and preheated aliquots of Ox_{OD}K166. The intensities are normalised by the mean intensity of the naturals, which were stored at room temperature (~20°C). The legend lists the mean normalised intensity ± standard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

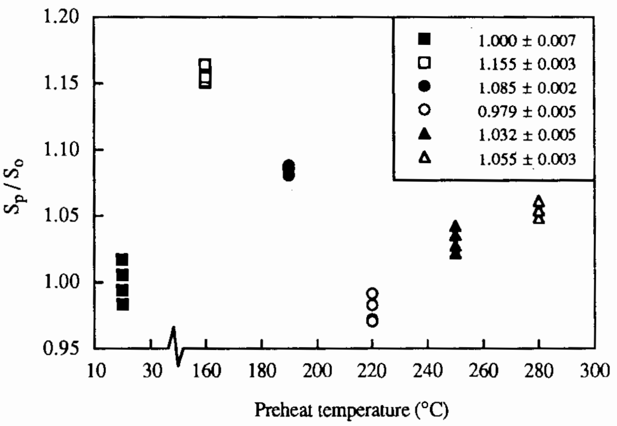

Figure 3.
Sensitivity of the 110°C TL peak (105-115°C peak area integration) for natural and preheated aliquots of $Ox_{OD}K166$. The sensitivities are expressed as a ratio of S_p (peak intensity after preheat) to S_0 (peak intensity before preheat), normalised by the mean ratio of the naturals, which were stored at room temperature (~20°C). The legend lists the mean normalised S_p/S_0 ratio ± standard error for each treatment.

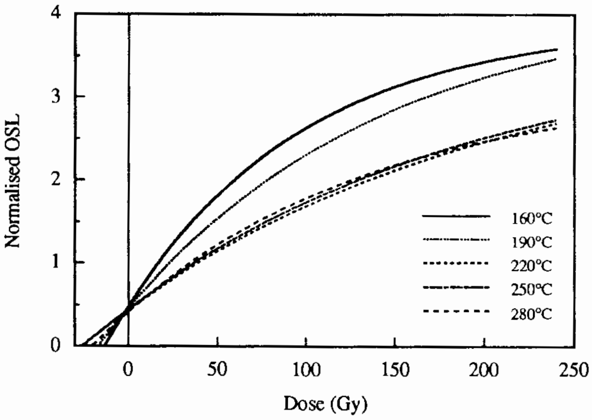

Figure 5.
OSL growth curves for preheated aliquots of $Ox_{OD}K166$. Each growth curve is fitted to eight naturals and four aliquots at each of six dosage levels (5, 10, 20, 60, 120 and 240 Gy) using a weighted saturating exponential function (Brumby, 1992). The OSL intensity for each aliquot is calculated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

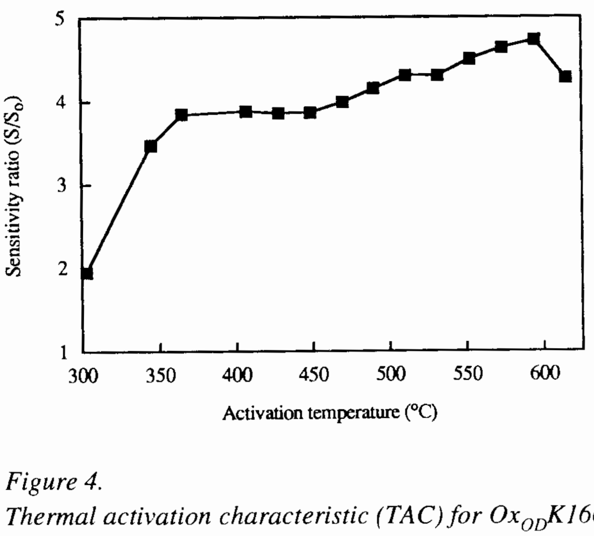

Figure 4.
Thermal activation characteristic (TAC) for $Ox_{OD}K166$, derived using a single natural aliquot and the multiple activation procedure (Aitken, 1985).

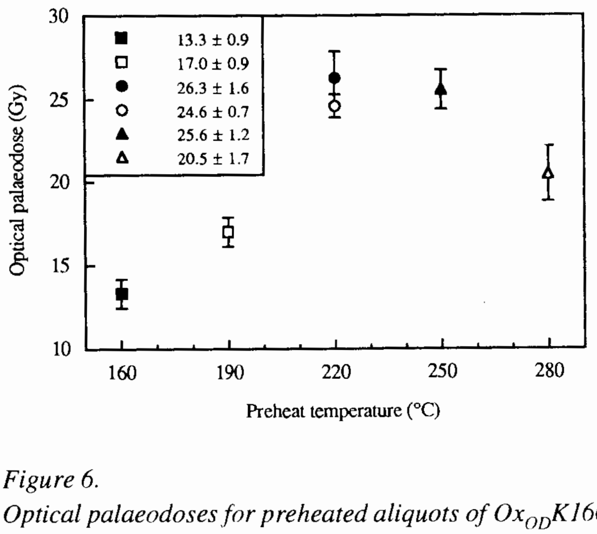

Figure 6.
Optical palaeodoses for preheated aliquots of $Ox_{OD}K166$ derived from the growth curve fits in Fig. 5. The legend lists the mean palaeodose ± standard error for each treatment, along with the palaeodose (24.6 ± 0.7 Gy) from a subsequent trial using 52 aliquots and the 220°C preheat. The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

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

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

3. 110°C TL peak sensitivity

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

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

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

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

4. Thermal transfer of charge

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

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

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

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

Palaeodose determinations on $Ox_{OD}K166$

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

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

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

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

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

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

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

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

Conclusions

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

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

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