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Combined gamma and beta dosimetry, using $Al_2O_3:C$, for in situ measurements on a sequence of archaeological deposits

C.I. Burbidge*, G.A.T. Duller

Institute of Geography and Earth Sciences, University of Wales, Aberystwyth, Wales SY23 3DB, UK

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Abstract

Single crystal chips of $Al_2O_3:C$ (TLD500) were evaluated and employed for measurements of combined gamma, beta, and cosmic dose-rate. A regenerative dose optically stimulated luminescence (OSL) measurement procedure is described, using the closed source of a Risø reader for irradiation. The reproducibility of these measurements was assessed for both irradiation within the reader, and during field deployment. Combined dose-rate measurements using the chips were corrected for beta attenuation by comparison with independent dose-rate measurements. Dosimeters were buried for up to 3 weeks in the field, providing parallel measurements of in situ combined dose-rate for 79 OSL samples.

Keywords: Aluminium oxide; Environmental dose-rate; OSL; Old Scatness; Shetland

1. Introduction

Assessment of the environmental dose-rate is central to luminescence dating. Where the sites to be dated are expected to have local variations in the radiation field (e.g. Brennan et al., 1997), in situ measurement of the environmental dose-rate at the point of luminescence sampling is desirable. Highly sensitive luminescence detectors such as TLD500 allow a large number of in situ measurements to be made in parallel during a relatively short field deployment. TLD500 detectors are single crystal chips (1 mm thick, 5 mm diameter) of carbon-doped aluminium oxide ($Al_2O_3:C$) (Akselrod et al., 1990). The aim of the present work was to evaluate and apply TLD500 for measurement of combined beta, gamma and cosmic in situ dose-rate, through complex and inhomogeneous sequences of archaeological deposits.

2. Measurement protocol

All measurements on the TLD500 chips were conducted using a Risø TL/OSL-DA-10 reader, with Hoya U340 detection filters (50% transmission between 290 and 370 nm). Optical stimulation was from blue LEDs (470 nm), delivering 2.3 mW/cm^2 to the sample. The reader was equipped with a 40 mCi $^{90}Sr/^{90}Y$ beta source, giving a dose-rate of around 1.7×10^4 μGy/s to samples with the shutter in front of the source open. However, doses measured in the field were expected to be less than 100 μGy. Radiation derived from the source with the shutter in the closed position, mainly Bremsstrahlung (X-Rays), provides an appropriate dose-rate (this was the, "old irradiator", as described by Markey et al., 1997). Calibration of this radiation from the closed source was performed, using TLD500 chips, by comparison with a 1 mGy dose from a ^{137}Cs gamma source, administered at the Risø National Laboratory, Denmark. The ^{137}Cs source calibration was by the Danish State Institute for Radiation Hygiene, and is directly tracable to the NPL standard in the UK. The dose-rate from the closed $^{90}Sr/^{90}Y$ source of the Risø reader to a TLD500 chip on a nickel planchette was 0.687 ± 0.003 μGy/s.

All optically stimulated luminescence (OSL) measurements were conducted at 30°C, following a pre-heat to

* Corresponding author. Tel.: +44-1970-622604; fax: +44-1970-622659.

E-mail address: cib99@aber.ac.uk (C.I. Burbidge).

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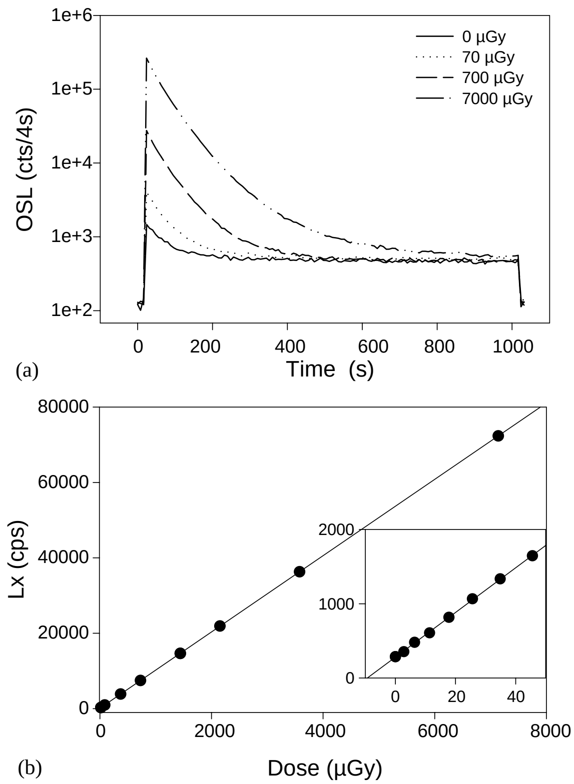

Fig. 1. (a) Decay of the OSL signal from a TLD500 chip following different periods of irradiation under the closed source of the Risø reader. Note the increased background at 1 ks stimulation following 7000 µGy applied dose. For comparison, burial doses measured in this project were around 70 µGy. (b) Linear growth in OSL signal with dose from the closed source, in the range 0–7000 µGy. Inset is the response for 0–40 µGy, showing the ~ 9.5 µGy "zero dose" response caused by irradiator cross-talk.

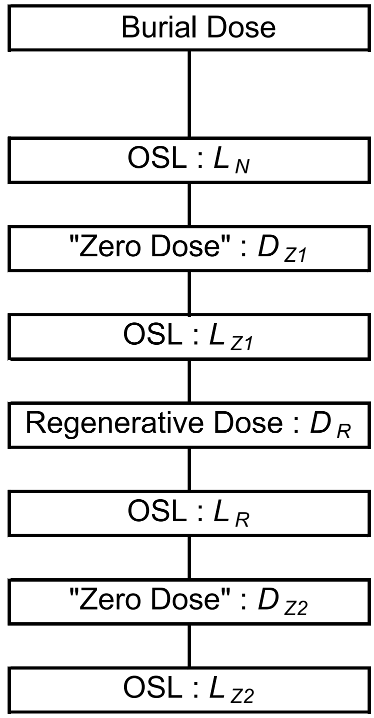

Fig. 2. Summary of the measurement procedure for TLD500 chips. All OSL measurements involved a pre-heat to 100°C at 5°C/s, which was held for 10 s. The sample was then heated to 30°C, held for 10 s, and then the 1 ks optical stimulation was started.

100°C, held for 10 s. Decay of the OSL during stimulation with the blue LEDs was found to be relatively slow, and in order to prevent the build up of a residual OSL signal during repeated measurements, a standard stimulation time of 1 ks was adopted. This prevented buildup of background for initial signals up to around 10^5 counts per four-second channel, equivalent to more than 700 µGy (Fig. 1a). Growth of the OSL signal with dose between 0 and 7000 µGy (Fig. 1b) was linear, with an intercept on the signal axis that indicated a "zero dose" of approximately 9.5 µGy. The "zero dose" arose from the approach and departure of the chip from the position of the source in the reader. Crosstalk for three carousel positions either side of the source was greater than 5% of the direct (closed source) dose-rate. This crosstalk, and the inability to turn off the source, meant that it was practical to analyse only one chip at a time.

A standard measurement procedure for evaluating the dose received by the chip is outlined in Fig. 2. The linearity of growth in OSL signal with dose meant that only a single regeneration dose (D_R) was required. A dose of 81 µGy was adopted for general use, as this was similar to the doses expected from field deployment. Measurements of the "zero dose" (L_{Z1} and L_{Z2}) were inserted after measurement of both the OSL due to the burial dose (L_N) and due to the regeneration dose (L_R), to account for irradiator crosstalk, while at the same time providing a check on sample sensitivity. The average ($L_{Zave} = (L_{Z1} + L_{Z2})/2$) of the two "zero dose" measurements was used in D_e calculation. Since L_N did not include a component from crosstalk, L_{Zave} was only subtracted from L_R in the calculation of the equivalent dose (D_e):

$$D_e = D_R L_N / (L_R - L_{Zave}). \quad (1)$$

D_e is the equivalent Bremsstrahlung X-ray dose, delivered under the conditions of these experiments, required to give the same OSL signal ($L_R - L_{Zave}$) as the OSL (L_N) from the burial dose.

3. Zeroing of the OSL signal

In the experiments described in this paper, TLD500 chips were placed behind window glass to bleach the OSL signal before exposure to environmental radiation. A residual luminescence signal in $Al_2O_3:C$ may be produced by phototransfer resulting from UV exposure (Colyott et al., 1996). Window glass is a poor transmitter of UV and should minimise this effect, providing a convenient way of zeroing the OSL signal in these chips, in the field or laboratory. Eleven explicit measurements of residual OSL signal, following bleaching behind window glass, were made on separate chips. The mean residual D_e was 1.1(±0.1) μGy. This is larger than the value of < 0.4 μGy found by Bøtter-Jensen et al. (1999), but less than 2% of the burial doses with which the residuals are associated.

4. Dose recovery and reproducibility

Three experiments, designed to differentiate between sources of error in the laboratory and the field, were conducted to assess the accuracy and precision of measurements made using the TLD500 chips. Accuracy in D_e determination was assessed by giving known doses to chips, then measuring D_e as if the chips contained an unknown burial dose (we term this process "dose recovery").

First, repeated dose recovery measurements were performed on a single chip without removing it from the Risø reader. The chip was subjected to 40 measurement cycles as described in Fig. 2, with burial dose replaced by the recovery dose of 81.42 μGy. The regenerative dose D_R was also 81.42 μGy, giving 80 repeat measurements. The first measurement of OSL signal was 2.5% higher than the mean of the other measurements, and was excluded from calculations. For the remaining measurements however, the OSL response increased by $< 0.02\%$ per measurement cycle. Correction for sensitivity change during D_e determination was therefore unnecessary. Using Eq. (1), these OSL measurements can be used to calculate 40 separate recovered D_e values (Fig. 3a). The first point was again omitted from calculations. The mean recovered D_e (81.47 μGy) was within 0.1% of the dose given (81.42 μGy), and the standard deviation of individual recovered D_e values about the mean was 0.7% (0.54 μGy).

In a second experiment, ten measurement cycles were performed on a single chip, giving ten recovered D_e determinations. Before irradiation with the recovery dose (80.83 μGy), the chip was placed in sunlight behind window glass for 1 day. After the recovery dose was given, the chip was removed from the reader. It was then replaced in the reader, and the measurement sequence run separately. Recovered D_e values for this experiment are presented in Fig. 3b. The mean recovered dose was 81.63 μGy, with a standard deviation of 1.72 μGy (2.1%). The difference from the known dose (80.83 μGy) indicates a residual signal, following bleaching, equivalent to 0.8 μGy, although this is within errors.

In the third experiment, six chips were used to make repeat measurements of the dose-rate in homogeneous sand, in the laboratory. A portion of dried sand from Banc-Y-Warren Quarry, Cardigan, UK, was sieved to obtain the < 355 μm fraction. This was used to fill two containers (each polythene, 3 cm high, 6 cm diameter). One container was sealed with polythene film, and into the other were inserted six dosimeters (Fig. 4). The dosimeters were TLD500 chips protected from light by jackets. The jackets were made of

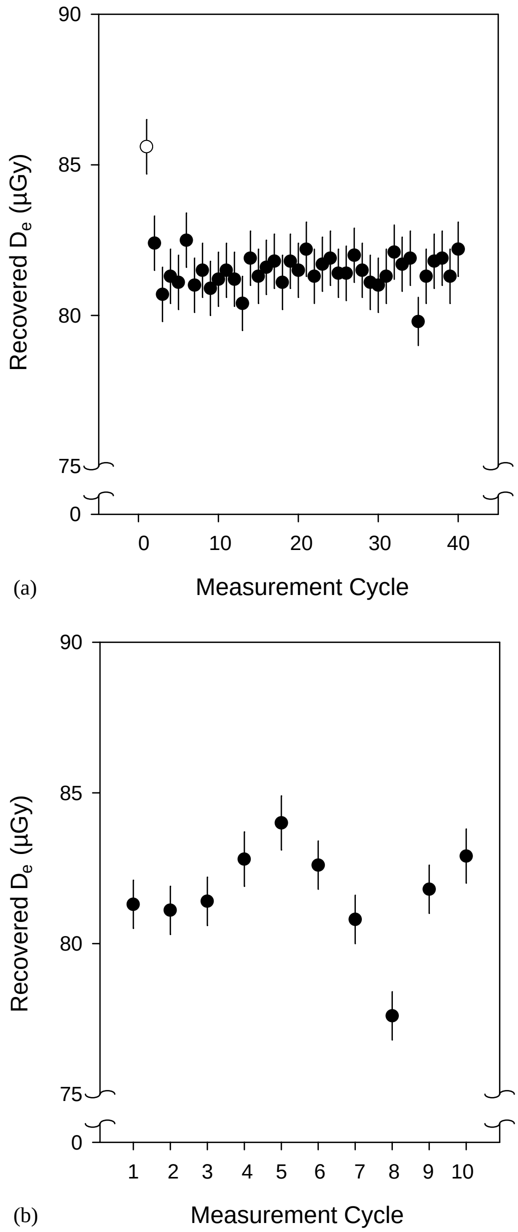

Fig. 3. D_e values from a single TLD500 chip subjected to repeat dose recovery cycles, plotted with errors expected from counting statistics. (a) Forty repeated cycles of irradiation (81.42 μGy), and measurement as in Fig. 2, all undertaken within the reader. (b) Ten repeated cycles of sunlight bleaching behind window glass, irradiation (80.83 μGy), and measurement as in Fig. 2.

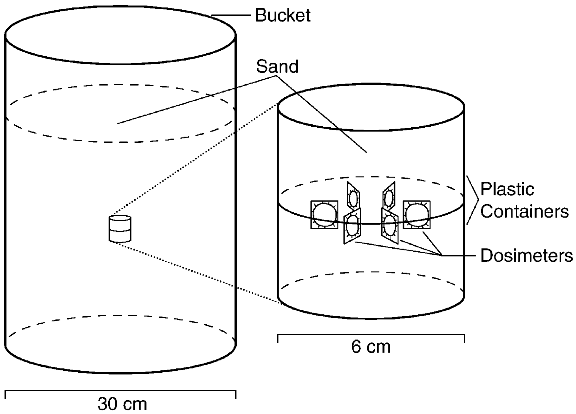

Fig. 4. Experimental arrangement for repeated burial of dosimeters in the laboratory.

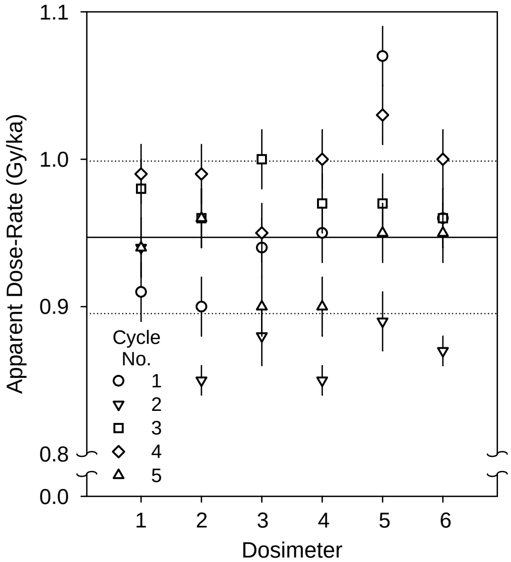

Fig. 5. Apparent dose-rates from six dosimeters (TLD500 chip in jacket), measured from repeated deployment in homogeneous sand, in the laboratory. Five cycles of sunlight bleaching behind window glass, burial, and measurement as in Fig. 2 were undertaken. Burial periods were 33, 33, 37, 37, and 41 days for cycles 1–5, respectively. Residual doses were measured in a separate cycle between cycles 1 and 2. The mean and standard deviation of the complete data set are indicated by the solid and dotted lines, respectively.

black polythene, sealed with black insulation tape, with a total mass thickness of 26 mg/cm^2 (thickness ≈ 0.18 mm, density ≈ 1.4 g/cm^3). The two small containers were taped together to seal in the dosimeters, and buried in the middle of a bucket (plastic, 30 cm high, 30 cm diameter) filled with dried, unsieved sand from the same quarry (Fig. 4). After around 5 weeks the six dosimeters were removed. The chips were measured to obtain the burial dose, as in Fig. 2, and bleached for a few days behind window glass. The chips were then inserted in new jackets, replaced in their original positions in the containers, and the whole process repeated five times. D_e values between 76 and 108 μGy were measured for storage periods between 33 and 41 days. A mean residual dose, from bleaching behind window glass, of 1.3(±0.3) μGy, was measured for the six TLD500 chips between cycles 1 and 2, and was subtracted from all D_e values before calculation of the apparent dose-rate. The mean of all dose-rate measurements was 0.95 Gy/ka, with standard deviation of 5.4% (Fig. 5).

5. Correction of apparent dose-rate

A particular advantage of using TLD500 was the potential for its use in a combined in situ dosimeter, measuring beta, gamma, and cosmic radiations. The simple polythene jackets of the dosimeters were designed to minimise beta attenuation, while maintaining a robust, lightproof cover for the chips.

The photon energy response of $Al_2O_3:C$ is very similar to that of quartz (Akselrod et al., 1990), and thus the gamma dose-rate to quartz can be well represented by measurements on an $Al_2O_3:C$ dosimeter. Energy deposition from the hard component (muons) of cosmic radiation in the ground proceeds, as for gamma radiation, mainly via secondary electron production. Detection efficiency of the hard component was therefore assumed similar to gamma. However, because of their thickness, significant beta attenuation occurs in TLD500, and the beta efficiency of the chips has been reported to be energy dependent in the range < 0.224 to 2.28 MeV (Akselrod et al., 1999).

The fraction of the total dose-rate measured by the dosimeters, F_T, was calculated for samples from the fieldwork site in Shetland (Table 1). Apparent dose-rate, measured using the dosimeters, was divided by the total dose-rate to the samples calculated from independent dose-rate measurements. Beta and gamma dose-rates were found from a combination of thick source alpha counting (TSAC) and beta counting (using a gas-counting system). Cosmic dose-rate was calculated from a fit to the data of Prescott and Hutton (1988, Fig. 1). The independently derived dose-rates were calculated for etched coarse grain quartz, and corrected for in situ water content. F_T therefore provided a direct conversion factor for the dosimeter data.

Some inverse correlation of individual F_T values and apparent dose-rate may be observed for samples from Pit 3 at the Old Scatness site (Table 1), but this is not significant over the range of dose-rates present. The fraction of the total dose-rate, F_T, was also calculated for the sand used in the third reproducibility experiment (Section 4, and Figs. 4 and 5), using similar methods to those for the Old Scatness samples. After allowance for the gamma field external to the bucket, F_T was calculated to be 0.61, higher than the values

Table 1
Dose-rates and fraction of total independent dose-rate measured by the dosimeters. Pit 3 displayed the most homogeneous stratigraphy of those in the present study. Samples from Pit 3 were used to define a site value for the fraction of total dose-rate measured by the dosimeters. Samples from Pit 1, with the least homogeneous stratigraphy, were measured for comparison

Site	Sample	Apparent dose-rate to dosimeter (Gy/ka)	Dose-rates to coarse-grain quartz[a]		Cosmic dose-rate (Gy/ka)	Total independent dose-rate (Gy/ka)	Measured fraction F_T[b]	Corrected dose-rate to dosimeter[c] (Gy/ka)
			Beta (Gy/ka)	Gamma (Gy/ka)				
Aber/41 OSB00 Pit 3	T5	1.17 ± 0.06	1.31 ± 0.04	0.70 ± 0.04	0.19 ± 0.01	2.20 ± 0.06	0.53 ± 0.03	2.43 ± 0.14
	T8	1.14 ± 0.06	1.31 ± 0.04	0.66 ± 0.03	0.19 ± 0.01	2.16 ± 0.05	0.53 ± 0.03	2.36 ± 0.14
	T11	1.12 ± 0.06	1.44 ± 0.04	0.70 ± 0.04	0.18 ± 0.01	2.32 ± 0.05	0.48 ± 0.03	2.33 ± 0.14
	T18	1.11 ± 0.06	1.34 ± 0.04	0.69 ± 0.04	0.17 ± 0.01	2.20 ± 0.05	0.51 ± 0.03	2.31 ± 0.14
	T24	1.43 ± 0.08	1.91 ± 0.05	0.96 ± 0.05	0.17 ± 0.01	3.04 ± 0.07	0.47 ± 0.03	2.96 ± 0.18
	T27	1.40 ± 0.08	1.97 ± 0.05	1.00 ± 0.06	0.16 ± 0.01	3.12 ± 0.08	0.45 ± 0.03	2.91 ± 0.17
	T30	1.57 ± 0.09	2.30 ± 0.06	1.12 ± 0.06	0.16 ± 0.01	3.58 ± 0.09	0.44 ± 0.03	3.27 ± 0.19
	T33	1.62 ± 0.09	2.31 ± 0.06	1.15 ± 0.06	0.16 ± 0.01	3.62 ± 0.09	0.45 ± 0.03	3.38 ± 0.20
							Mean[d] = 0.48 ± 0.01	
Aber/41 OSB00 Pit 1	K12	1.05 ± 0.06	1.26 ± 0.03	0.66 ± 0.05	0.19 ± 0.01	2.12 ± 0.06	0.49 ± 0.03	2.18 ± 0.13
	K14	1.06 ± 0.06	1.16 ± 0.03	0.59 ± 0.04	0.19 ± 0.01	1.94 ± 0.05	0.54 ± 0.03	2.20 ± 0.13
	K18	1.08 ± 0.06	1.40 ± 0.04	0.72 ± 0.04	0.19 ± 0.01	2.30 ± 0.05	0.47 ± 0.03	2.24 ± 0.13
	K30	1.38 ± 0.08	1.81 ± 0.05	0.88 ± 0.05	0.17 ± 0.01	2.86 ± 0.07	0.48 ± 0.03	2.87 ± 0.17
	K33	1.47 ± 0.08	1.83 ± 0.05	0.96 ± 0.06	0.17 ± 0.01	2.96 ± 0.08	0.50 ± 0.03	3.06 ± 0.18
	K36	1.59 ± 0.09	1.95 ± 0.05	1.00 ± 0.06	0.17 ± 0.01	3.11 ± 0.08	0.51 ± 0.03	3.30 ± 0.19

[a]Dose-rates calculated from TSAC and beta counting results, corrected for both in situ water content and etching of the quartz grains during the preparation of OSL samples.
[b]Calculated by dividing the apparent dose-rate to the dosimeter, by the total independent dose-rate.
[c]Calculated by dividing the apparent dose-rate to the dosimeter by the mean F_T for Pit 3.
[d]Mean of F_T for Pit 3 samples (=0.48), with expected error $\sigma/\sqrt{n}$ (=0.01).

measured for the Old Scatness samples. The cause of this difference is not clear. However, since F_T did not vary significantly for the samples from Old Scatness (Table 1), the mean value for the most homogeneous section excavated, Pit 3 (mean $F_T = 0.48 \pm 0.01$), was used to correct all the apparent dose-rates measured by the dosimeters at the Old Scatness site.

The factor F_T is primarily a product of beta attenuation, and the fraction of the infinite beta dose-rate measured by the dosimeter, F_β, may also be calculated. Assuming that there is no attenuation of the gamma and cosmic dose-rates (i.e. $F_\gamma = F_c = 1$), $F_T = 0.48 \pm 0.01$ equates to $F_\beta = 0.15 \pm 0.01$ for the Old Scatness samples, i.e. the dosimeters recorded 15% of the total beta dose-rate in their burial environment. The equivalent value for the sand used in the reproducibility experiment was $F_\beta = 0.23$.

6. Field measurements

Seventy-nine combined dosimeters were buried with OSL samples at the Old Scatness Broch archaeological site, Shetland, UK, as part of a project looking at OSL dating of anthropogenic soil sequences. The dosimeters were deployed in vertical columns down each of three pits, excavated in an area containing evidence of cultivation associated with the site. The sections contained similar overall stratigraphies, though with different levels of inhomogeneity. Many layers contained substantial amounts of stone, and uncultivated midden material retained an inhomogeneous structure visible upon excavation.

All TLD500 chips were bleached until required, and nine travel dosimeters were made up as the buried dosimeters were extracted. In situ dosimeters were placed by extracting the steel tubes (2 cm diameter, 12 cm length) used for OSL sampling, inserting the dosimeter into the sediment at the end of the tube, and replacing the tube in the section. The effect of burial at less than 30 cm into the face of the section is included in the correction factor (F_T). The dosimeters were buried for between 15 and 21 days, and travel was between 2 and 3 days, depending on the date of extraction. Equivalent doses of between 50.8 and 106.0 μGy were measured from chips used in in situ dosimeters. The mean travel dose, 7.7(±0.5) μGy, was subtracted from all results to give burial dose values. Apparent dose-rates were then calculated according to burial time. The third reproducibility experiment (Section 4, above) was considered to simulate field deployment conditions, so a 5.4% expected error was

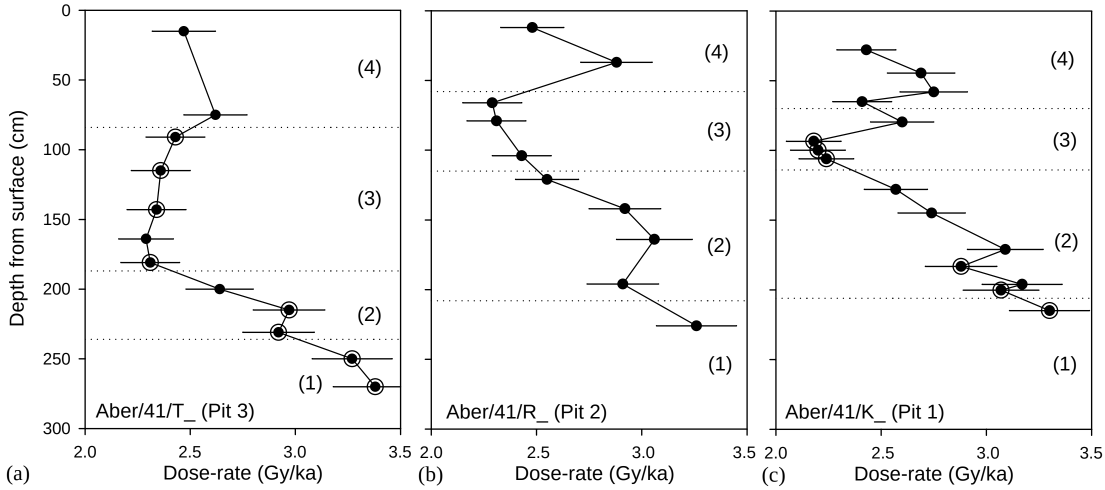

Fig. 6. Corrected dose-rates measured using the dosimeters, down the three sections sampled at the Old Scatness site. ● In situ dosimeter measurement. ◯ Independent dose-rate determination also made for sample (Table 1). Stratigraphic divisions: (1) Quartzose sand. (2) Rich quartzose soils and midden material. (3) Poorer calcareous soils and sands. (4) Mix of (1), (2), and (3), from recent construction activity.

applied to the apparent dose-rates at this stage. Finally, apparent dose-rates were divided by F_T (0.48 ± 0.01), to give corrected dose-rates. Corrected dose-rates measured by the 79 in situ dosimeters ranged from 2.2 to 3.5 Gy/ka, with standard errors of 5.9%.

7. Discussion

Plots of dose-rate versus depth for Pits 1–3, display broad patterns of change down section with superimposed variability (Fig. 6). The broad changes in dose-rate correlate well with stratigraphy and between pits. Change appears smoothest in Pit 3 (Fig. 6a), which contained the least stone and midden. The plots in Fig. 6 display instances where significant ($> 2\sigma$, $> 11.8\%$) differences in dose-rate occur between samples less than 20 cm apart, and sometimes within 10 cm of each other. In this situation, gradients in gamma dose-rate are unlikely to be well represented by measurements on small samples within the laboratory. Conversely, local changes in beta activity may not be well represented by in situ measurements of the gamma field.

Many of the significant changes down the Old Scatness sections occur as part of a steep, but relatively continuous, increase in dose-rate through unit (2) (Fig. 6a–c). The trend continues beyond the range expected for gamma gradients across boundaries (∼ 70% of the change in 20 cm. Aitken, 1985, Fig. H1), and is taken to represent gradual change in matrix composition. However, some of the significant changes in dose-rate (Fig. 6) occur in isolation. This implies that the different dose-rate was recorded either close to an inclusion of different radioactivity, or within an isolated volume of different matrix radioactivity. Examples can be seen at the boundary between units (1) and (2) in Fig. 6a, and within and between units (3) and (4) in Fig. 6c.

Results from samples K12, 14, and 18 (Table 1, Fig. 6c unit (3)), also serve to illustrate the effects of local changes in radioactivity. Sample K14 was from a thin layer of sand, bounded by two soil layers containing K12 and K18. The dose-rate measured for K14 by TSAC and beta counting is significantly lower than those for K12 and K18, but the dose-rates measured by the dosimeters are similar for the three samples. Relative to actual dose-rates in situ, the lower beta dose-rate derived from TSAC and beta counting for K14 should be accurate, but the gamma dose-rate will be too low, since the effect of the adjacent higher radioactivity layers will not be measured. Conversely, the dose-rate measured using the dosimeter will be too high. The dosimeter is a more efficient detector of gamma radiation than beta, so the lower beta dose-rate of K14 will not have a proportional impact on the overall dose-rate measured. In situ measurement using a gamma spectrometer would result in an even greater overestimate, since beta radiation is not detected at all.

The low beta detection efficiency (relative to gamma) of the dosimeters biasses measurements towards detection of the gamma field. Relative beta response could be improved by the use of a thinner detector (Akselrod et al., 1999), but at the expense of overall sensitivity and hence the required burial time. However, thin $Al_2O_3:C$ dosimeters tested to date have either been fragile (20 mg/cm^2 single crystal, Akselrod et al., 1999), been fixed to a beta-thick backing (Göksu et al., 1999, Akselrod et al., 1999), or used as loose granular $Al_2O_3:C$ (Soumana et al., 1994). A sufficiently thin and robust detector, that is convenient to deploy and measure, might instead use a flexible plastic substrate to bear the phosphor.

8. Summary

An OSL measurement procedure for TLD500 chips was designed, using Bremsstrahlung from the closed source of a Risø reader for laboratory irradiation. Using this procedure, known doses (of 81.02 μGy) were recovered to within 0.1%, and scatter of repeated measurements in the reader was less than ±0.7%. An experiment to simulate repeated field deployment indicated reproducibility of ±5.4%, in measurements of apparent dose-rate.

Seventy-nine TLD500 chips in polythene jackets were used to measure combined photon (gamma plus cosmic) and beta dose-rate in situ, for small, closely spaced OSL samples, in a 3-week burial period. Beta attenuation was corrected for by comparison with independent dose-rate measurements on samples from the more homogeneous contexts. Uncertainty in corrected dose-rate was ±5.9%.

The deposits studied contained significant variations in dose-rate, both between and within depositional contexts, over distances of less than 20 cm. Conventional laboratory dose-rate measurements were therefore inappropriate for many samples. In situ dose-rate measurements might also be significantly in error, if it were not ensured that the chip was placed at the location of the material sampled for measurement of D_e.

Since this paper was accepted for publication, data have become available that suggest the calibration of the beta counter in the Aberystwyth laboratory may be incorrect. At the time of going to press, it was still not clear which calibration is correct. The substance of this paper is unaffected by the change in dose rates from beta counting, but if the revised calibration is correct, values of the fraction of the total dose rate (F_T) and of the infinite beta dose rate (F_β) measured by the dosimeters will be different. For the samples from Old Scatness, the mean F_T will be 0.58 ± 0.01 instead of 0.48, and the mean F_β will be 0.27 ± 0.01 instead of 0.15. For the sand from Banc-Y-Warren the F_T will be 0.73 instead of 0.61 and the F_β will be 0.32 instead of 0.23.

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