Dose Normalization in Fine Grain Dating of Loess

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In fine grain dating it is usually assumed that a series of fine grain discs does not need to be normalized because the disc to disc scatter in the TL intensity is better than ±5% (Aitken, 1985). However it has recently been observed that in a group of eight discs prepared from fine-grain loess the natural TL showed a scatter such that the standard deviation was ±11% of the mean and this was reduced to ±4.5% after a dose normalization procedure commonly used for coarse grains.

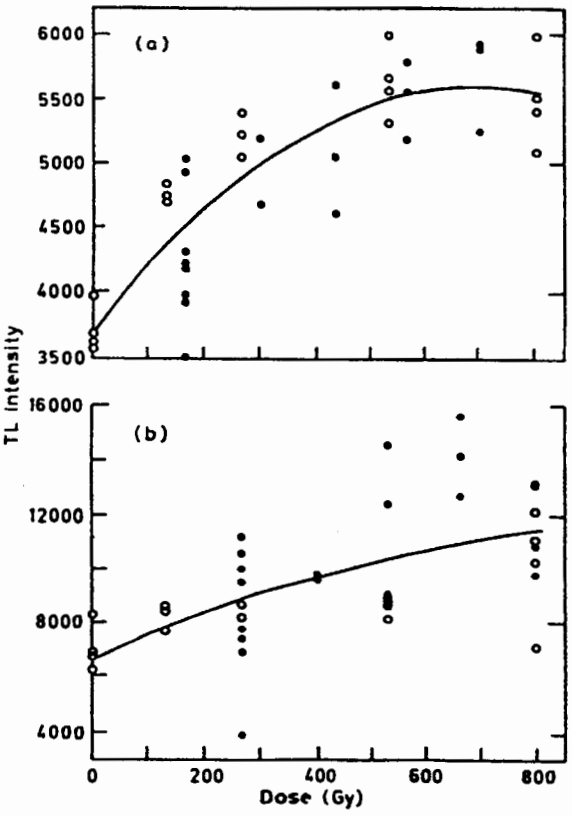

Figure 1

Relationship between the TL generated by the normalizing dose of 6 Gy and the total radiation dose received by the sample prior to normalization. The solid line represents a best-fit quadratic function. Data points are indicated by black circles for natural samples and open circles for bleached samples. N.B. the suppressed zero on the TL intensity axis.

In the Adelaide TL laboratory it has been the practice in dating sediments using 100 μm quartz to dose-normalize all the discs used in the natural and bleached growth curve determinations. After the discs have been glowed they are all given the same normalizing dose, routinely chosen as 6 Gy. The TL so generated in the 350°C peak, the one which is used in the equivalent dose analysis, is then determined. Since the discs in each series have received varying amounts of radiation and there is usually a small amount of pre-dosing, this TL will also vary with dose, and when plotted against the total radiation dose received (Fig.1) can be fitted by a suitable function. The normalization factor for a given sample at each increment of radiation dose is calculated as the fitted value divided by the sample value. This factor is then a function of dose.

The loess samples came from the Belan Valley in India and were estimated to be about 30,000 years old. The material was predominantly present as fine grains from which discs were prepared by the standard techniques, each disc holding about 1 mg of powder. The total bleach procedure was carried out and the data were analysed by the slide technique (Prescott et. al., 1993).

A preliminary determination of the equivalent dose without normalization gave 170 Gy for sample 1. In plotting the TL generated by the normalizing dose against the total radiation received, it was assumed that the naturals had received the equivalent dose of 170 Gy with further increments up to 600 Gy. In the case of the bleached samples, it was assumed that bleaching had set the dose to zero, with increments up to 800 Gy. (In fact three days of bleaching in full sun had reduced the TL to 15% of the natural.) The plot was best fitted with a quadratic relationship as shown in Fig.1a. The downturn in the function at the highest dose plotted has no physical basis, but is an artifact which does not affect the general conclusions. A full analysis of the normalized data yielded an equivalent dose of 200 Gy as shown in Fig.2a. There was a very convincing equivalent dose plateau in the region of the glow peak.

For the second sample, the standard deviation in the raw data was 17% of the mean and this was reduced

to 6.5% by the normalization process described. The normalization TL intensities are plotted in Fig.1b and the growth curve in Fig.2b.

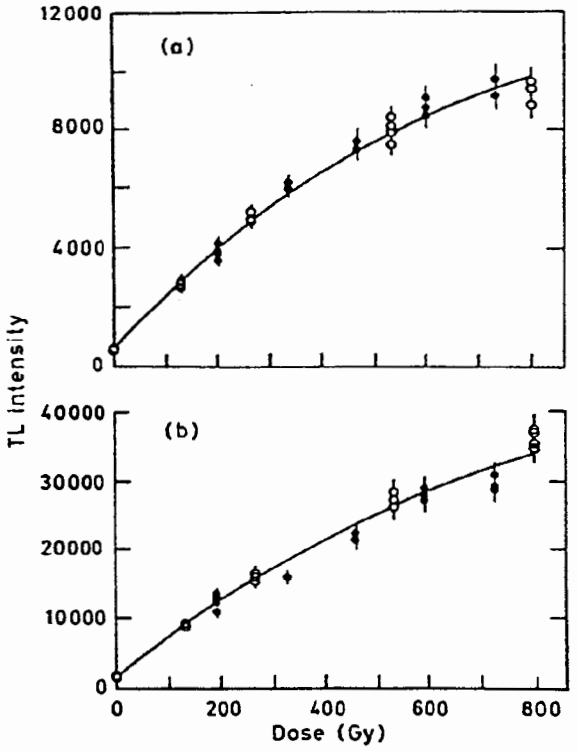

Figure 2

Growth curves generated from the (N+dose) data, denoted by black circles, and the (B+dose) data, denoted by open circles, using the slide technique. The data refer to the two samples of loess from Belan taken from the region of the peaks in the respective glow curves. The solid line represents the best-fit saturating exponential function.

It is not known why the scatter in the TL of these fine grains is greater than might be expected. It does not appear to be associated with varying masses of powder on the discs. As this is a mixture of minerals, it is possible that most of the TL is coming from a small number of grains belonging to one mineral, presumably quartz, which is not present as the major constituent. Dose normalization should work for this situation, as it does here. The fact that the normalization TL plotted against radiation dose follows a quadratic relationship may indicate some degree of saturation. In both samples the growth curve obtained in the age analysis was exponential, also showing a tendency to saturate.

The scatter in luminescence data from coarse grain feldspar samples has been discussed in a recent paper by Huntley and Berger (1995) who attribute some of the inter-sample variation to two external causes, different radiation doses and different extents of bleaching at deposition. As the dose normalization worked well for the Belan loess when differences in total radiation dose were taken into account, using the total bleach assumption, it is concluded that incomplete bleaching is not a factor here. This result is to be expected for samples of loess which should be completely bleached in transit.

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Semi non-destructive, single aliquot ESR dating

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Introduction

In ESR dating of tooth enamel, the specimen is usually partly destroyed: enamel is separated from dentine, ground and about ten aliquots are produced for the establishment of the dose response curve (DRC). Furthermore, some enamel and dentine material is used for uranium analysis. This partial destruction of samples is not feasible if ESR dating is to be applied on valuable specimens, such as fossil hominids.

In principle, it is possible to measure ESR signals of large samples that have not been pretreated by:
1) inserting the whole specimen into an ESR spectrometer (cavity with an aperture; see Ikeya 1993, p.480),
2) placing a small ESR spectrometer close to or around the sample (Ikeya & Ishii 1989, Ishii & Ikeya 1990), or
3) ESR imaging of surfaces (Furusawa et al. 1991).

If a complete tooth is measured in the cavity, it will not be possible to separate the enamel and dentine components of a composite ESR spectrum (this may be overcome by applying a magnetic field gradient which allows the recording of spatially resolved ESR spectra). Therefore, subsequent ESR age estimates are most likely erroneous because ESR dating of dentine is fraught with severe age underestimations due to continuing crystallisation of the hydroxyapatite constituents (Grün & Schwarcz 1987). Imaging has a considerably lower ESR sensitivity (10^{-10}) compared to measuring the same sample in the cavity, and therefore only very high spin concentrations can be detected by imaging. If a tooth of a larger sample (e.g. a tooth sitting in a mandible) is to be measured, the whole specimen will have to be irradiated which in turn may impede subsequent investigations.

In most cases, fossil teeth have cracks and it easy for an experienced curator to remove small pieces of enamel from a tooth and later re-insert these fragments into place without any visual damage. Three bovid teeth excavated at the Florisbad archaeological site, South Africa, were selected for testing the validity of using single enamel pieces for the establishment of dose response curves. A relatively large enamel piece was separated from each of these three teeth and a smaller segment was cut from the centre of each sample, the outer portion was used to establish DRCs by the conventional multi-aliquot powder method (see e.g. Grün 1989).

Two main problems that had to be addressed were:
1) The sample had to be mounted into the cavity in a reproducible position. Unlike powders, single pieces show a very strong angular dependence of the ESR signals (the dating signal in tooth enamel is an axial species, therefore anisotropic). Gamma irradiation will generate free radicals in most materials used for fixing the sample. These signals are usually in the range of g=2 and will interfere with the dating signal. Therefore, the sample has to be removed from a holder for gamma irradiation and then reproducibly positioned into the cavity.
2) As it is not possible to remove any surface layers, the external alpha dose rate has to be considered.

Experimental and Results

For the insertion of the sample into the cavity a sample holder was designed as shown in Figure 1. It was made of silica tubing which contained small E' signals. These signals disappeared after annealing to 1100 °C for 10 h. Constant insertion depth is ensured by the glass O-ring which sits on top of the cavity. Vertical alignment is achieved as the holder is long enough to protrude from the lower ends of the cavity and can be held in place by the teflon clamps at the top and bottom end of the cavity. The tube fixed at the top is used for angular alignment of the sample holder with marks on the magnet pole shoes.

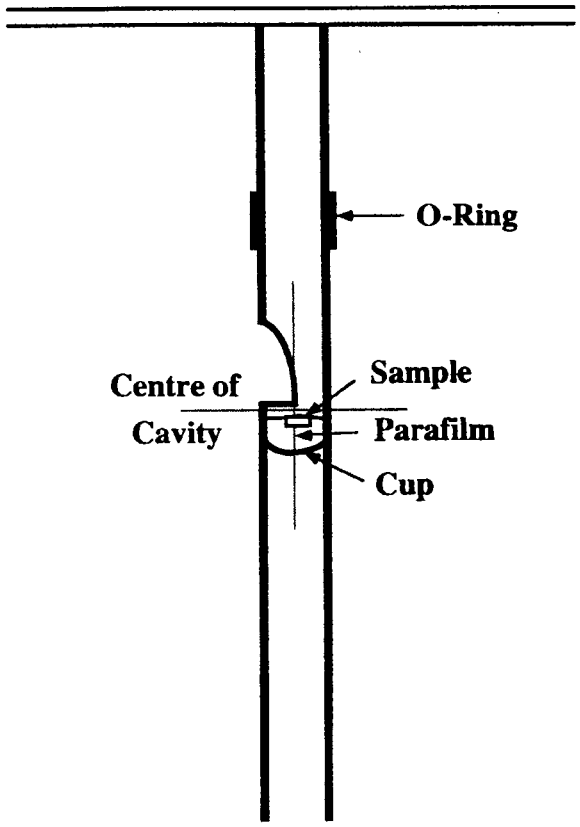

Figure 1

Sample holder for measuring single pieces (not to scale). The holder is made of a round bottom silica tube, extended by a further silica tube. Above the cup, a triangular slot provides access. The sample is pressed into warm parafilm, which will turn into a flexible melt with negligible ESR signals (see Figure 2). The O-ring is a piece of a silica tube fused to the sample holder. The top cross tube can be aligned with markings on the pole shoes of the magnet.

Some parafilm was melted at 120 °C over night in the cup of the silica tube and the samples were then pressed into the hot melt. This created an imprint into the parafilm that is very stable at low temperatures and enables the easy removal and re-insertion of the sample. Apart from its high viscosity at room temperature, parafilm was chosen because it shows only minor ESR signals (see Figure 2). The holder assembly ensures that the sample sits very close to the centre of the cavity.

Preparation and ESR measurement of the powder samples were carried out without any special treatment. Because there was relatively little material available, only 9 and 7 aliquots (with the minimum acceptable routine weight of 15 mg) were prepared for samples 1159 and 1160, respectively, instead of the 10 aliquots that are usually measured (as for 1164A with 25 mg). The weights of the single pieces were 16.54, 23.07 and 17.10 mg for samples 1159, 1160 and 1164A, respectively. The irradiation doses of the powders were: 0, 10.2, 18.5, 34.4, 86.9, 127.6, 196.8, 338.0, 465.0 and 669.5 Gy. The cumulative doses for the single pieces were: 0, 9.3, 27.8, 55.1, 97.1, 187.3, 277.6, 367.8, 468.1, and 558.3 Gy. The powders were measured about 3 weeks after irradiation, single pieces were measured twice, within a few hours after irradiation and after heating at 60 °C for about 13h.

All measurements were carried out on a Bruker ECS 106 spectrometer with a 15 kG magnet and a rectangular 4102 ST cavity. The powder samples were recorded with the measurement parameters routinely applied in this laboratory: accumulation of 8 scans with 1.015 Gpp modulation amplitude, 10.24 ms conversion factor, 20.48 ms time constant, 2048 bit spectrum resolution (resulting in a total sweep time of 20.972 s), 120 G sweep width and 2 mW microwave power. The single pieces were recorded with the same measurement conditions except that the spectra resulted from 128 accumulated scans.

The main differences between the multi-aliquot powder and single sample approach are:
- different ESR spectra (see Figure 2),
- the powder samples are all irradiated at the same time, whereas the single samples are successively irradiated and measured.

Figure 2 shows the single piece and powder ESR spectra of the three samples. It can be seen that all powder spectra are basically identical apart from an occurrence of a radiation induced peak at lower magnetic fields (around 3410 G). The main differences in the single piece spectra occur at the lower end of the main peak (especially for the natural samples), and in the position of the lower dip of the main peak relative to the dip of the second peak. In the powder spectra, the second dip is always below the first one, whereas in the single piece spectra the second dip occurs above the first dip. This may be due to the fact that all enamel pieces were inserted into the parafilm melt in the same way (the outside of the tooth was always horizontal, facing down) and the relative position of the two lower dips is most probably the result of preferential growth of hydroxyapatite crystals within the enamel layer.

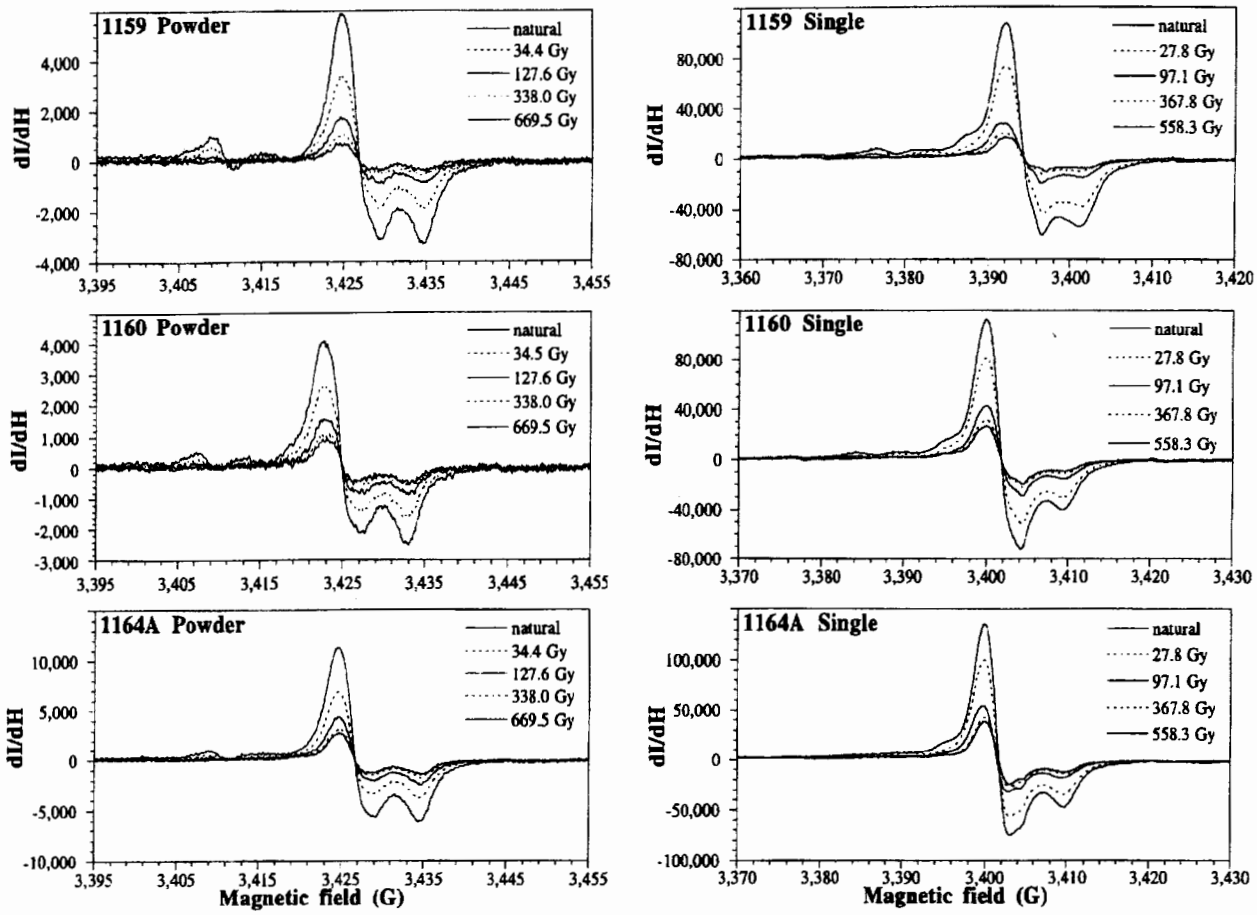

Figure 2

Powder and single piece spectra for three tooth enamel samples

The reproducibility of the ESR signals that were due to the positioning of the single piece samples in the cavity was within 2% (repeatedly inserting the whole holder assembly into the cavity). The reproducibility of mounting the sample into the parafilm and positioning the assembly into the cavity was statistically indistinguishable from the previous approach. Samples were first measured with a single piece standard in order to address the problem of equipment stability. However, the equipment stability was better than the reproducibility of the standard, hence, no normalisation was carried out.

There have been several papers reporting that unstable signals occur after irradiation of hydroxyapatite (e.g. Houben 1971, Ostrowski et al. 1974 and Caddie et al. 1985), decaying within about one week. However, no such effect could be seen in the ESR spectra recorded one hour after irradiation and those recorded 4 days after irradiation. Some heating was carried out after irradiation (at 60 °C between 8 hours and 100 hours). No quantitative effect could be observed from this treatment either.

The dose response curves of the powders and the single pieces are shown in Figure 3. The DRCs are normalised on the natural ESR intensities (set to 100) of the sample pairs. The data were fitted with a single saturating function (see Grün & Brumby 1994). The visible differences between the respective dose response curves are mainly due to the normalisation process which causes maximum deviations at the highest dose points.

Discussion

The D_E values of the powders and single pieces of the three samples agree well within the calculated errors. It can also be seen that the DRCs of the powder/single piece pairs have similar characteristics: for example, the DRCs of sample 1159 are nearly linear ($I_{MAX} >> I(D_{MAX})$; $D_0 >> D_{MAX}$) whereas the DRCs of sample 1160

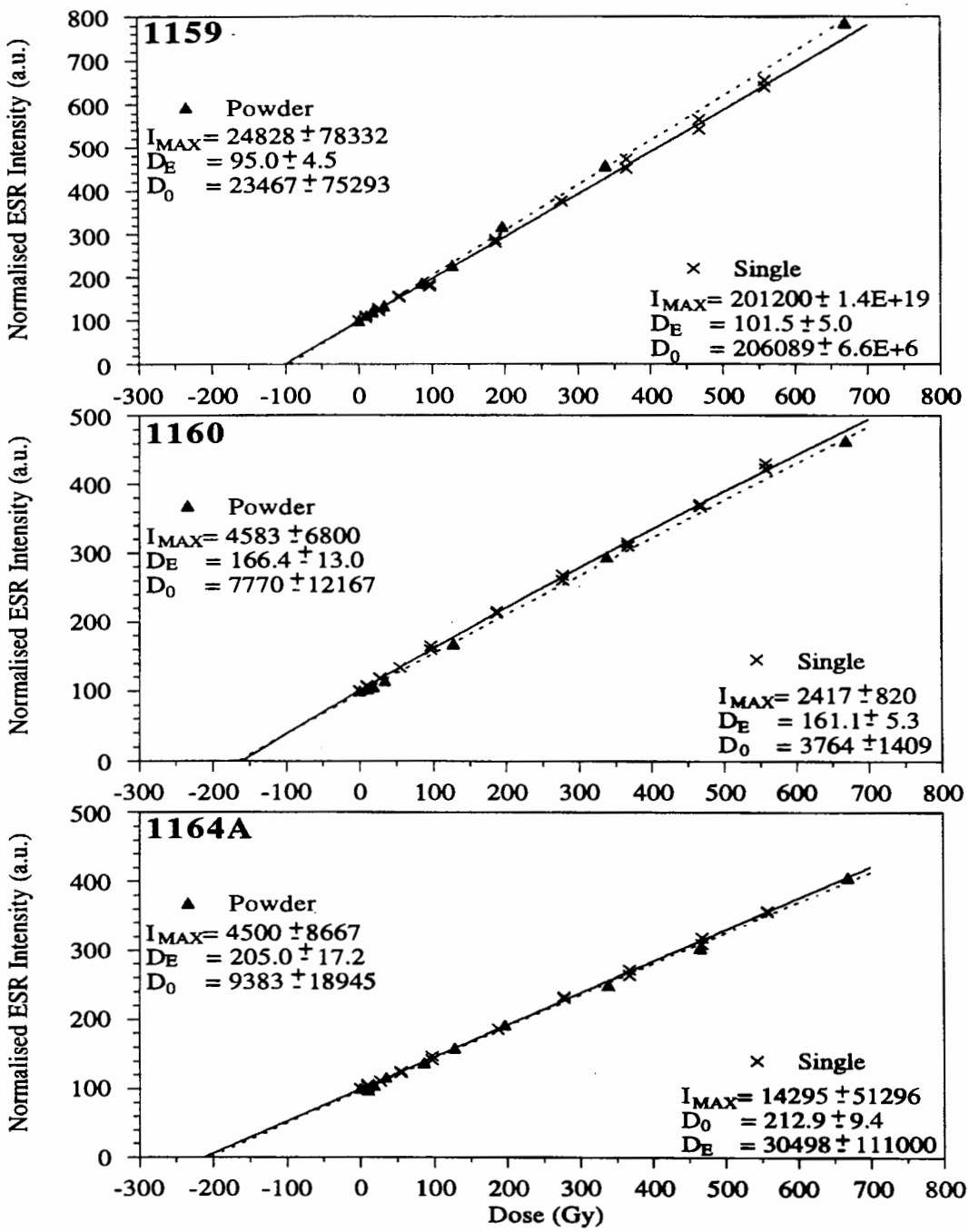

Figure 3

Dose response curves for the multi-aliquot powder and single piece method. The dotted lines are the calculated best fits for the powder DRCs and the solid line is the best fit for the single piece DRCs

Although the comparison between the powders and the single pieces is very good it seems advisable, if single aliquot measurements are carried out, to measure at least 10 dose points on several pieces of the same specimen. Unlike single aliquot measurements in luminescence dating (e.g. Duller 1991, 1994, Murray et al., 1995 instead of in press), the single aliquot ESR approach involves significantly more work than the routine multi-aliquot method. This is due to the fact that the multi-aliquot ESR approach involves only one gamma irradiation session (we use a sample holder that allows the irradiation of ten aliquots of 16 samples simultaneously). Gamma sources are usually located in some distance away from the dating laboratory whereas beta dosing for luminescence dating is usually carried out by an automated irradiation facility in the dating laboratory.

Compared to the powder samples, it is not possible to remove the volume from the single pieces that has been irradiated by external alpha rays. If we assume that the sample has a minimum thickness of 500 μm, the average effective "α" -doses from the Th- and U-decay chains are 0.65% and 0.54% of the effective infinite matrix doses, respectively (Grün 1987). Assuming an "α" -efficiency of 0.227, which is the highest value so far measured for tooth enamel (Chen et al. 1994), the alpha dose contribution to the total external dose rate (alpha + attenuated beta + gamma) is 1.6% for both the Th- and U-decay chains. The alpha dose contribution of thicker samples will be proportionally smaller. The calculation shows that external alpha dosage seems to have a minor influence on the D_E -value.

For the internal dose rate it is necessary to assess the U concentrations in enamel and dentine. Presently, we envisage using ICP-MS on samples with a mass of about 5 mg. Although this means that ESR dating is still not entirely non-destructive (in the sense that no material is lost for analysis) we shall be able to come very close to it.

Conclusion

Dose determination of tooth enamel can be carried out semi non-destructively on a single piece of enamel. This piece can be separated from a larger specimen and can later be reinserted into its original place. For dose rate estimation, however, it is still necessary to destroy some, though small, amount of the specimen.

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Optical Dating Of Young Sediments Using Fine-Grain Quartz

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Introduction

Coarse grained quartz has been widely used for optical dating due to its natural abundance, lack of fading and, as was found by Godfrey-Smith et al (1988), the fact that the quartz OSL signal bleaches substantially more quickly than that of feldspars. However, the use of coarse grains typically leads to greater scatter of data than for fine grained methods. The use of polymineral fine grain IRSL measurements can overcome the problem of large scatter. However, the associated feldspar signal may exhibit fading, or may in some cases exhibit a low signal intensity (Parish 1992). In this paper it is shown that fine-grained quartz extracted from polymineral fine-grained samples provides an alternative for dating young sediments. A comparison of the dating results and behaviour of the fine grain IRSL and quartz GLSL signals will be made.

Preparation and Measurement Procedures

The samples used in this study were two alluvial deposits (792d and 792e) and two colluvial deposits (962a and 962b). Sample 792d has age constraints of approximately 4500-1300 years old and the date for 792e should lie between 1300-920 years old based on radiocarbon dates. Samples 962a and 962b should both be of a similar age and between 4000 and 1000 years old on the basis of archaeological evidence.

These sediments were treated using routine fine grain procedures (treatment with hydrochloric acid, hydrogen peroxide and separation of the 4-11μm grains by sedimentation). IRSL dating was carried out on this material for three of the samples. The polymineral fine grain fraction was subsequently treated with fluorosilicic acid to remove all non-quartz material as suggested by Jackson et al (1976) and following the preparation procedure of Berger et al (1980). After acid treatment of several days, samples were rinsed and all grains that did not settle in 20 min from a depth of 8 cm of acetone were removed to ensure that no partly dissolved grains remained. This procedure resulted in samples that had no IRSL signal (indicating a lack of feldspars) and that tended to be pale, although samples 962a and 962b were still slightly brown in colour due to an iron oxide coating. Further checks for purity were carried out on sample 792e using infrared spectroscopy which showed the sample to be approximately 95% quartz (there was a trace of possible remnants of feldspars, clay minerals or mica). This quartz purity was confirmed by mapping for potassium and aluminium in the grains using an x-ray spectrometer attached to a scanning electron microscope which showed that less than 10 grains in a sample of approximately 400 contained significant levels of these elements (Rees-Jones 1995).

Measurements were carried out on the fine-grained quartz with stimulation by 514 nm light from an argon ion laser. All discs were normalised by applying an initial short shine to the natural sample and 4-6 discs were used at each dose point and more at the natural point. A preheat of 5 min at 220°C was used. Additive beta and alpha growth curves were determined for all the samples, and intercept corrections (to allow for recuperation, residual signal and any supra-linear growth) were calculated by extrapolating a regeneration growth curve obtained from the discs used for the beta growth curve (Aitken and Xie 1992). Similar measurements were carried out for IRSL dating except for the use of a 160°C preheat, the duration of this preheat being determined by constructing a preheat plateau (Huntley et al 1985). This was 4 hours for samples 792d and 792e and 6 hours for 962a. Curve fitting was carried out using the Elsec fitting program, which uses a least squares fit for linear fitting and a procedure for exponential fitting based on that described in Smith (1983).

Dating Results

The results for the polymineral IRSL and quartz GLSL measurements are shown in table 1. The fine grained quartz signals from all samples showed lower intercept corrections and lower a-values as compared to IRSL, as well as the low scatter on individual dose points expected for fine

grains (fig. 1 and 2). The fine-grained quartz dates calculated for 792d, 792e and 962a are in agreement with the polymineral fine grain IRSL dates and are consistent with archaeological evidence. Polymineral grains could not be prepared from 962b, but the fine-grain quartz date is consistent with the archaeological evidence.

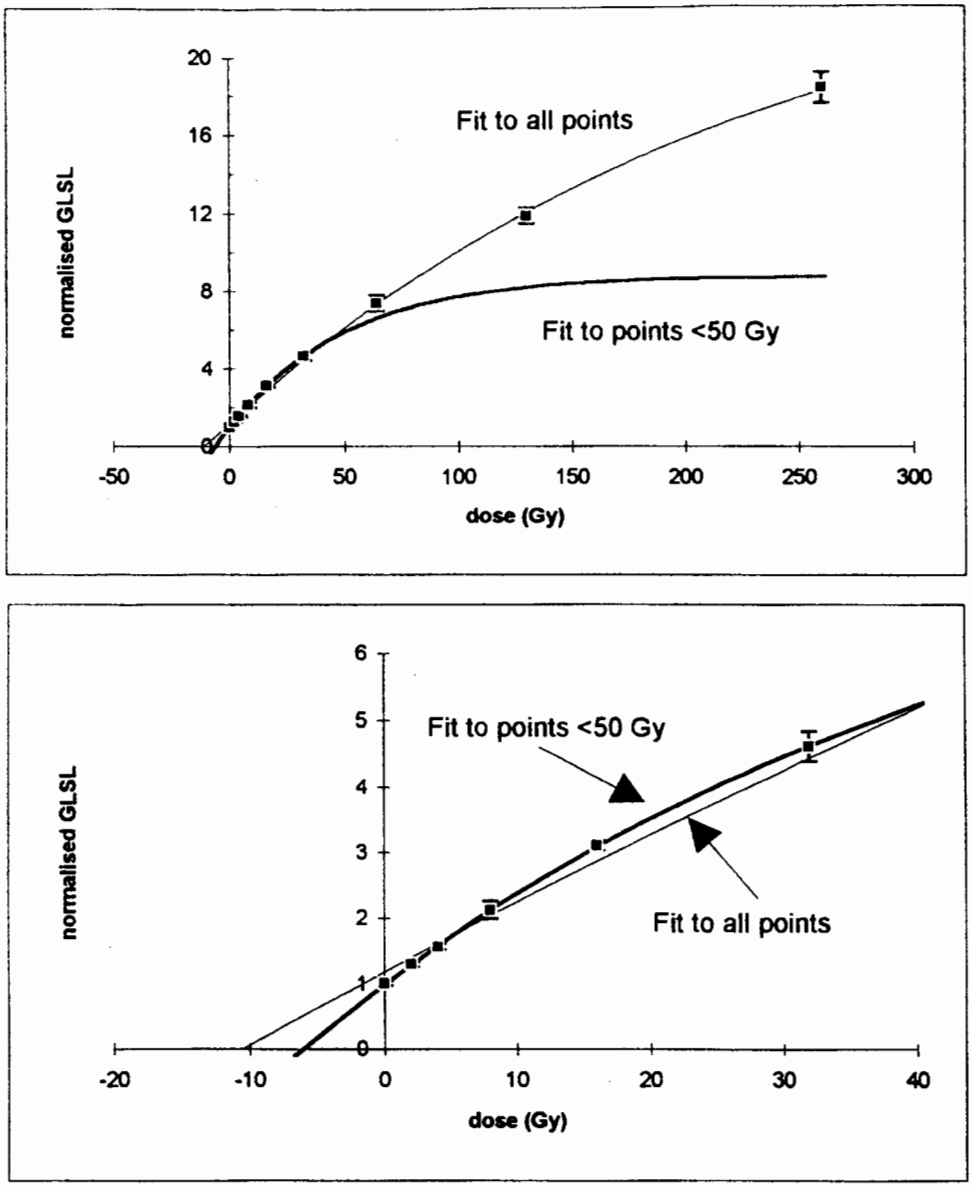

Figure 1

Additive beta growth curves for fine-grain quartz from sample 792d, showing the saturating exponential curve extrapolated through all dose points and to the dose points below 50 Gy. The dose points shown are the average of 6 discs and the error bar the standard deviation of the 6 measurements. Figure 1a shows the total dose range and figure 1b shows an enlarged view of the dose points less than 50 Gy.

Sample 792d was dosed to a relatively high level in order to observe the growth characteristics of the signal. The beta growth curve was clearly sub-linear from very low doses, but continued to grow up to the maximum dose point of 260 Gy (fig. 1a), and gave an equivalent dose (De) of 10.4±1.2 Gy when fitted with a saturating exponential function.

When the low dose region is examined (fig. 1b), it can be seen that the exponential function extrapolated over the full dose range does not fit the low dose points. The points up to 32 Gy can be most accurately fitted by a different saturating exponential function to give a De of 6.0±0.4 Gy. This value was used for the age calculation as it produced the best fit to the data close to the natural, which is the region of interest. As the highest dose points were progressively removed, the De value reduced until a dose of 32 Gy was reached, after which removal of further dose points made little difference to the De value calculated. A linear fit could only be made to the dose points up to and including 8 Gy (4 points) giving a De value of 7.2±0.1 Gy. However, this did not reflect the true growth of the signal and was considered inaccurate. In summary, it is apparent that the growth of this signal is not a single saturating exponential function.

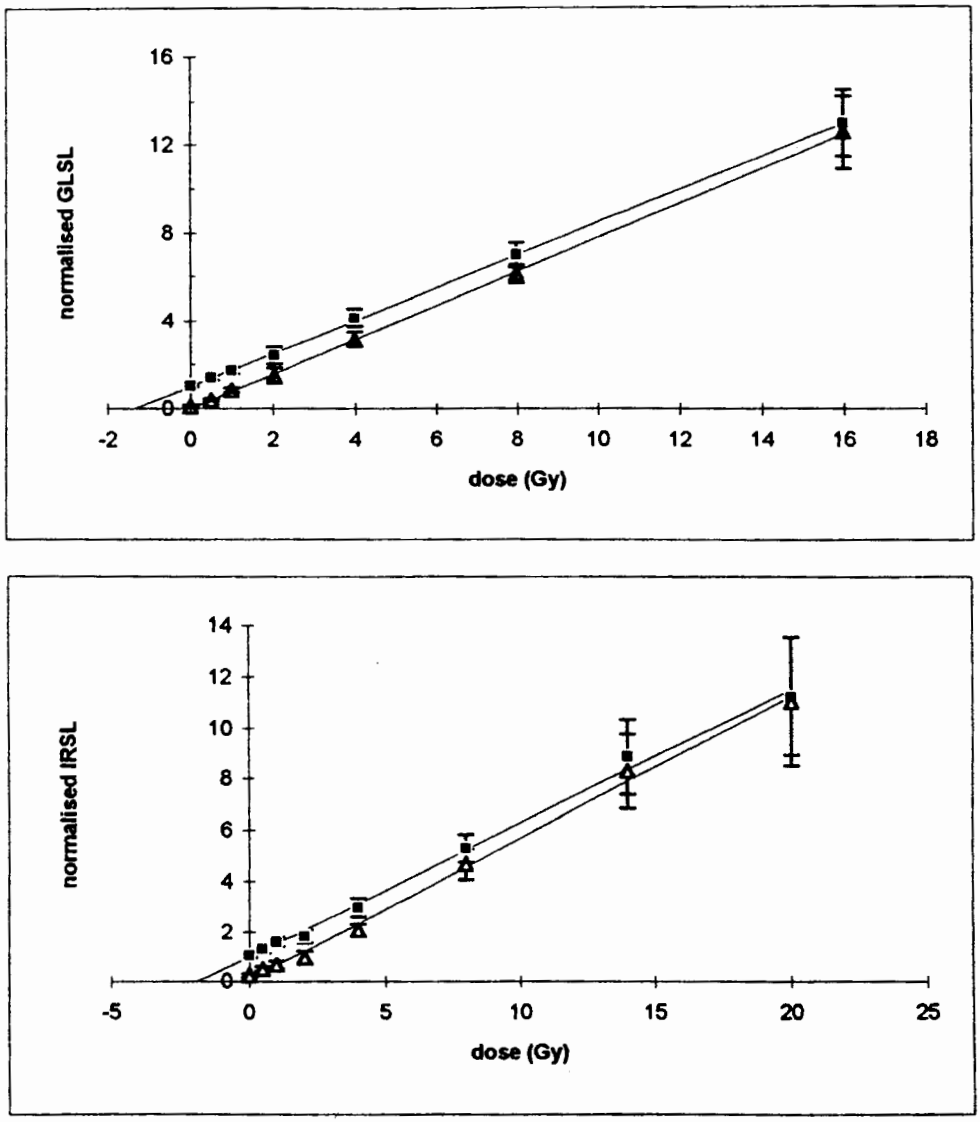

Figure 2

Additive beta (squares) and regenerated (triangles) growth curves for sample 962a, each dose point is the average of 5 discs. Figure 2a showns the growth curves for fine-grain quartz GLSL and figure 2b for polymineral IRSL.

The regenerated beta growth curve showed exponential growth when dosed up to 30 Gy, but a linear fit was made to the dose points up to 7 Gy to give the intercept correction in table 1, which allows for any recuperation and residual signal. For the alpha growth all the dose points up to the maximum of 3200 Gy (equivalent to a beta dose of approximately 100Gy) could be fitted by a single exponential function. The age calculated from the fine grained quartz data for this sample is 3560±290 years, which compares well to the IRSL age of 3640±210 years for the same sample. The IRSL signal was found to be linear up to the maximum added dose of 36 Gy.

sample	signal	natural signal (c/s)	beta D_e (Gy)	Intercept correction (Gy)	a-value	age (a)
792d	IRSL	2681	8.13±0.12	0.60±0.03	0.091±0.001	3640±210
	quartz	2155	6.03±0.41[e]	0.07±0.04	0.032±0.002	3560±290
792e	IRSL	1366	4.58±0.17	0.40±0.01	0.099±0.006	1270±100
	quartz	5417	3.52±0.24[e]	0.12±0.02	0.039±0.003	1420±190
962a	IRSL	150	1.98±0.10	0.43±0.05	0.069±0.004	3230±430
	quartz	233	1.41±0.05	0.11±0.02	0.043±0.002[*]	3020±360
962b	quartz	353	1.53±0.07	0.08±0.02	0.043±0.002	3370±360

[e] saturating exponential fit
[*] assumed value

Table 1
Polymineral fine-grain infrared and fine-grain quartz dating results. Infrared measurements were carried out at a power of 20 mWcm-2 and quartz measurements using a laser power of 5 mWcm-2.

Similar behaviour was also found for sample 792e which showed sub-linear growth from very low doses (less than 12 Gy), and a reasonable linear fit could not be made to any portion of the growth curve. The data, up to the maximum dose point of 30 Gy, could be fitted by a single saturating exponential function. In contrast the IRSL beta growth curve was clearly linear over the same dose range. The two resulting ages are in agreement for both signals, but with a larger error for the fine-grained quartz due to the nature of the exponential fit (table 1).

The fluorosilicic acid treatment and measurement of the fine-grained quartz signal was found to have major advantages when routine fine grain IRSL dating of samples 962a and 962b was attempted. For the sample 962a problems in calculating the intercept correction for the IRSL signal were experienced due to supra-linear growth and for 962b it proved impossible to separate polymineral fine grains from the bulk sample. This latter problem was due to the fine grains being bonded together with a cement of iron oxide and calcium carbonate which was only broken down by the fluorosilicic acid. The fluorosilicic acid treatment therefore enabled a check on the IRSL date for 962a and dating of sample 962b to be carried out.

The natural GLSL signals for these samples were low, due to the low De values and possibly due to some remaining iron coating, indicated by the reddish-brown appearance of the samples. However, the GLSL sensitivity was still an improvement on the IRSL sensitivity for sample 962a. The beta and alpha growth curves for these samples were linear (fig. 2a shows the beta growth curve of 962a) up to the maximum added beta dose of 16 Gy. The alpha De for sample 962a could not be measured due to lack of material, so the a-value was assumed to be the same as for sample 962b. The GLSL signal for sample 962a can clearly be seen in fig. 2a to have a much lower intercept correction and less scatter than that

962a can clearly be seen in fig. 2a to have a much lower intercept correction and less scatter than that of the IRSL signal in fig. 2b. Using the GLSL signal from fine grained quartz very precise low palaeodoses have been produced for these samples (table 1). The errors on the GLSL dates are larger than is usual for fine grains due to systematic errors in evaluating the low annual dose. The ages produced are consistent with each other and the IRSL date for 962a of 3230±430 years and fall within the limits from archaeological evidence.

Discussion

The fine-grained quartz GLSL has been found to have much lower intercept corrections for dating than IRSL. The reason for this is thought to be due to little or no recuperation of the GLSL signal on preheating. Additional experiments have shown that the GLSL signal of sample 792e did not undergo significant recuperation during preheating for 5 min at 220°C or during preheating at 160°C, which contrasts with the behaviour of the fine grain IRSL signal found by Rees-Jones and Tite (1994). This finding of little recuperation of the quartz luminescence signal agrees with the findings of Stokes (1992), who observed that very low De values (<0.3 Gy) could be measured for modern samples and concluded that little recuperation of the quartz signal occurs.

Two of the four samples measured in this study (792d and 792e) had GLSL growth that could not be fitted by a linear function, even the dose points of less than 15 Gy, but were more accurately fitted by a saturating exponential function. The shape of signal growth did not change when a 160°C preheat of 1-16 h was used or when a constant background subtraction value of 35 c/s, rather than the count rate in the last 50 s of the shine-down curve, was used. It may be that saturating exponential growth occurs at lower doses for fine-grained quartz than coarse grains, which would normally be expected to be linear up to 30-50 Gy (Smith et al 1986, Rhodes 1990). However, Readhead (1988) and Stokes (1994) have found quartz samples that exhibit sub-linear growth at lower doses than this. Too few fine-grained quartz samples have so far been measured to draw any firm conclusions. It should also be noted that the signal from coarse quartz grains and fine grains prepared by crushing the same material shows no difference in growth characteristics (Rhodes pers. comm.).

Conclusion

A pure quartz fraction may be separated from fine grained sediment using fluorosilicic acid treatment. Dating this fraction using GLSL has a number of advantages over using fine grain IRSL. These advantages include low intercept corrections, due to little recuperation; absence of fading as the signal comes from quartz (Fragoulis and Stoebe 1990 and Fragoulis and Readhead 1991); higher signal output for many samples as quartz has a greater natural abundance than feldspars (although feldspars are usually brighter); more rapid bleaching for quartz than for feldspars; and probable suitability of one preheat for all samples, as a single mineral species is measured. One disadvantage is that the signal can show saturation of the GLSL signal at low doses so that linear fitting is not possible, this may result in lower precision than for fine-grain IRSL dating. When a linear fit cannot be made to the GLSL quartz data then the GLSL signal will not produce very precise dates for young samples. Quartz GLSL has also been found to suffer from sensitivity changes on bleaching (Stokes 1994), but this should not be a major problem for additive dating of the fine grain signal as this would only affect intercept corrections which are low.

Although treatment with fluorosilicic acid increases the time required to prepare a sample, the advantages mentioned above mean that sample measurement time is reduced as there is no need to monitor for fading and probably no need for a preheat plateau test. This treatment is particularly suitable for young samples where intercept corrections are large with IRSL dating and linear growth of the fine grained quartz signal occurs. It is also suitable for samples that contain few feldspars or for those which contain feldspars that undergo fading. The treatment by fluorosilicic acid was also found to be of use for samples such as 962b where polymineral grains cannot be separated due to a cement of calcium carbonate and iron oxide.

This method of separating fine grained quartz has great potential for use in optical dating and is worth pursuing further.

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I would like to thank Steve Stokes, Professor Tite and D.J. Huntley for their help with this paper and RLAHA for funding the work.

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Rewievers:
D.J. Huntley
A.G. Wintle

Comments (D.J. Huntley)

It is very nice to see that there can be advantages to using silt-sized quartz grains, and I hope that this work will engender further study.

The high-dose response of Fig. la is interesting in that it would permit dating of older samples than is thought possible for quartz. I am wondering if this may, however, be due to a small amount of feldspar remaining; a detailed comparative study of the α and β responses may be informative in this respect.

Readers are asked to consider the relative merits of the "intercept correction" of Aitken and Xie and our "thermal transfer correction". It is not clear to me that either does everything that is required perfectly. Can anyone suggest a method that does? In the present work the intercept corrections found in the IR work appear to be unnecessarily large if they are due to thermal transfer because of an unnecessarily high temperature for the preheat. On the other hand I marvel at those obtained for the quartz. More often than not we have found our efforts to date quartz have been defeated by an overwhelming thermal transfer effect.

Finally, I would like to take this opportunity to encourage everyone engaged in studies of a basic nature to work on samples for which the ages are well established. Only in this way can one determine whether or not one has obtained the correct equivalent dose.

Thesis Abstracts

Thesis title: Die Infrarot-Stimulierte-Lumineszenz als Datierungsmethode für holozäne Lössderivate. Ein Beitrag zur Chronometrie kolluvialer, alluvialer und limnischer Sedimente in Südwestdeutschland.

Awarded by: Ruprecht-Karls-Universität Heidelberg
Author: A. Lang
Date: 1995
Degree: Dr. rer. nat.

Abstract

In the Middle European loess regions the insufficient amount of accurate age data is one of the limiting factors for a process-orientated reconstruction of landscape development.
This study proofs the applicability of IRSL-techniques for dating sedimentary reworking processes of Holocene age. This is accomplished through empirical dating-tests, methodological investigations, and by correlative analysis of sedimentological and physical properties of the sediments.
Samples for which independent age control was available were taken from aeolian, fluvial, limnic, and colluvial sediments from SW Germany. The investigations of the polymineral fine-grain fraction led to following results:
All IRSL-measurements were highly reproducible.
An outdoor bleaching-experiment on a rainy winter day showed that even under conditions of low light intensity, the IRSL-signal of a loess sample was reduced to zero after 30 min of exposure. This is confirmed by the ED-plateau-tests which hint to sufficient bleaching for colluvial and limnic sediments during the depositional process.
Results further indicate, that an ED-plateau is necessary for dating fine-grained sediments, but is not a sufficient indicator for datability.
Fading was detected only when a broad wavelength band was used. By using a narrow wavelength window around 410 nm, the influnence of fading on the dating results was eliminated.
With the exception of very young samples ED was determined within a statistical error margin of only ± 3 %.
The application of four independent methods to determine the dose rate allowed a presicion of up to ± 6,5 % in samples with radioactive equilibrium.
The study shows that a multiple method approach is necessary to achieve an acceptable level of reliability.
Based on the origin of the sediments investigated, it can be concluded that not only aeolian, but also colluvial and limnic sediments can be accurately dated by IRSL. The results are especially remarkable in the case of colluvial sediments, because transport distances did not exceed 100 m.
By using IRSL with colluvial sediments, it was possible to define four distinct phases of intense soil erosion in the Kraichgau Hills that correlate in age with the human settlement periods of Bandkeramik, Michelsberg, Celtic and Middle Age cultures.
Results obtained may be valid only for the Middle European loess regions because specific mineral compositions and details of the depositional process vary regionally.

Thesis title: The Use Of An Imaging Photon Detector For Luminescence Dating

Author: C.J. Mcfee, Research Laboratory For Archaeology And The History Of Art,6 Keble Road, Oxford, OX1 3QJ.

Thesis submitted for the degree of Doctor of Philosophy at the University of Oxford, Trinity Term 1995.

Abstract

The individual luminescence (TL and OSL) sensitivities of hundreds of individual feldspar and quartz grains have been measured. Measurements on individual grains were possible by using an imaging photon detector (IPD) which enables the low photon flux from each grain to be resolved and measured. In addition, the imaging capability of the IPD allowed many grains to be measured simultaneously, which is important since without such a facility the measurements would be extremely time consuming. Initially, the resolution of the optics was too poor for imaging and consequently a new optical system was developed which provided sufficient resolution to allow imaging of coarse grains (90-180μm in size).

It was found that a few grains has a high TL sensitivity, that is they exhibited both high natural TL and high second glow TL (measured after administering a laboratory beta dose). Other grains were found to have a high equivalent dose (ED), that is they exhibited only high natural TL and therfore high natural/second glow ratios. Examination of the grains using optical microscopy and SEM was undertaken in an attempt to explain the observed differences in natural and second glow TL. Grains which had both a high natural and a high second glow TL were not correlated with any of the

observed physical features from each grain and hence, differences in the trap and luminescence centre concentrations in each grain (which could not be measured directly) were thought to be the primary cause of the observed differences in TL sensitivities. Grains which had only high natural TL were considered to have been insufficiently bleached prior to deposition.

The infra-red stimulated luminescence (IRSL) from many quartz grains was measured and the origins of the IRSL, whether from indicidual quartz grains or from feldspar inclusions within the grains, was investigated. Finally, the IPD was used to obtain TL images of pottery, rocks and mineral slices and it was established that such images could be routinely used in laboratory to identify areas of luminescence inhomogeneity.

Notices

8th International Conference on Luminescence and Electron Spin Resonance Dating

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22 - 26 April

1996

*****

Australian National University
Canberra
Australia

Scope and Objectives
The Quaternary Dating Research Centre of the Division of Archaeology and Natural History invites you to the *8th International Conference on Luminescence and ESR Dating*, **LED 1996**, to be held at The Australian National University, Canberra, 22 - 26 April, 1996.

LED 1996 will bring together experts from around the world in the fields of luminescence and electron spin resonance dating. The topics covered range from fundamental studies of the basic physical phenomena, dosimetry, advances in equipment technology to the application of the dating techniques in Quaternary research and archaeology.

Prominent scholars will introduce the main topics by invited lectures. Sufficient time will be reserved for the scientific and technical exchange on current and future work. Poster presentations will be briefly introduced by their authors at the beginning of the poster sessions. One day of the conference will be reserved for applications in geography, geology and archaeology to give the Australian scientific community the opportunity to be updated on advances in Luminescence and ESR Dating.

Pre-Conference Field Trips
The luminescence laboratories at Adelaide, Wollongong and Canberra will organise pre-conference 4-5 day field trips. In order to estimate the number of participants, indicate on the reply card if you are interested.

ANU 50th Anniversary Student Prize
To celebrate its 50th Anniversary in 1996, The Australian National University has donated a $2,000 cash prize for the best student abstract. The prize will be awarded at the conference dinner.

Travel Assistance
Concessions on conference fees and accommodation will be available for overseas students who cannot obtain funding from their laboratories or research agencies. Limited travel assistance will be available for professionals.

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Mrs Judy Papps
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