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.

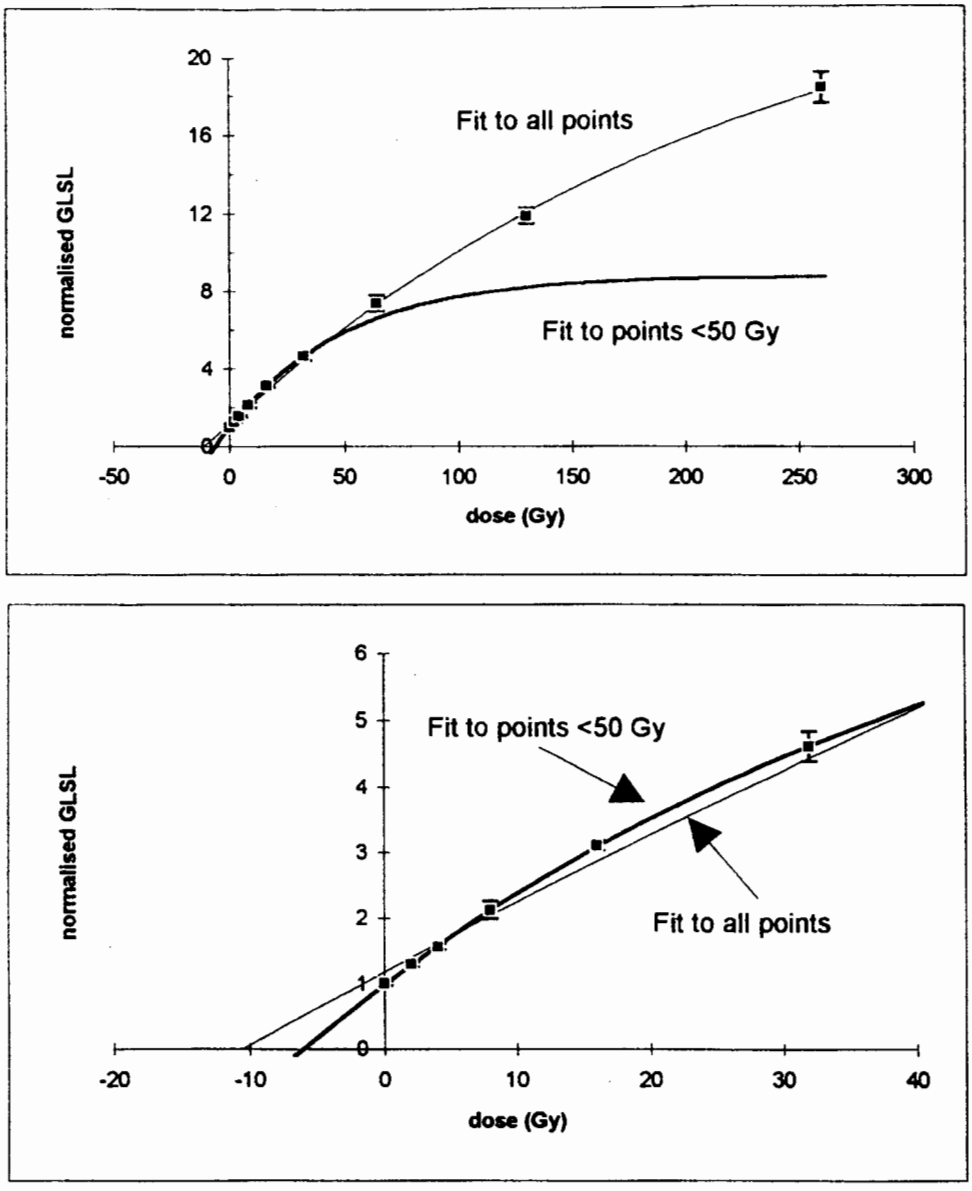

Figure 1

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.

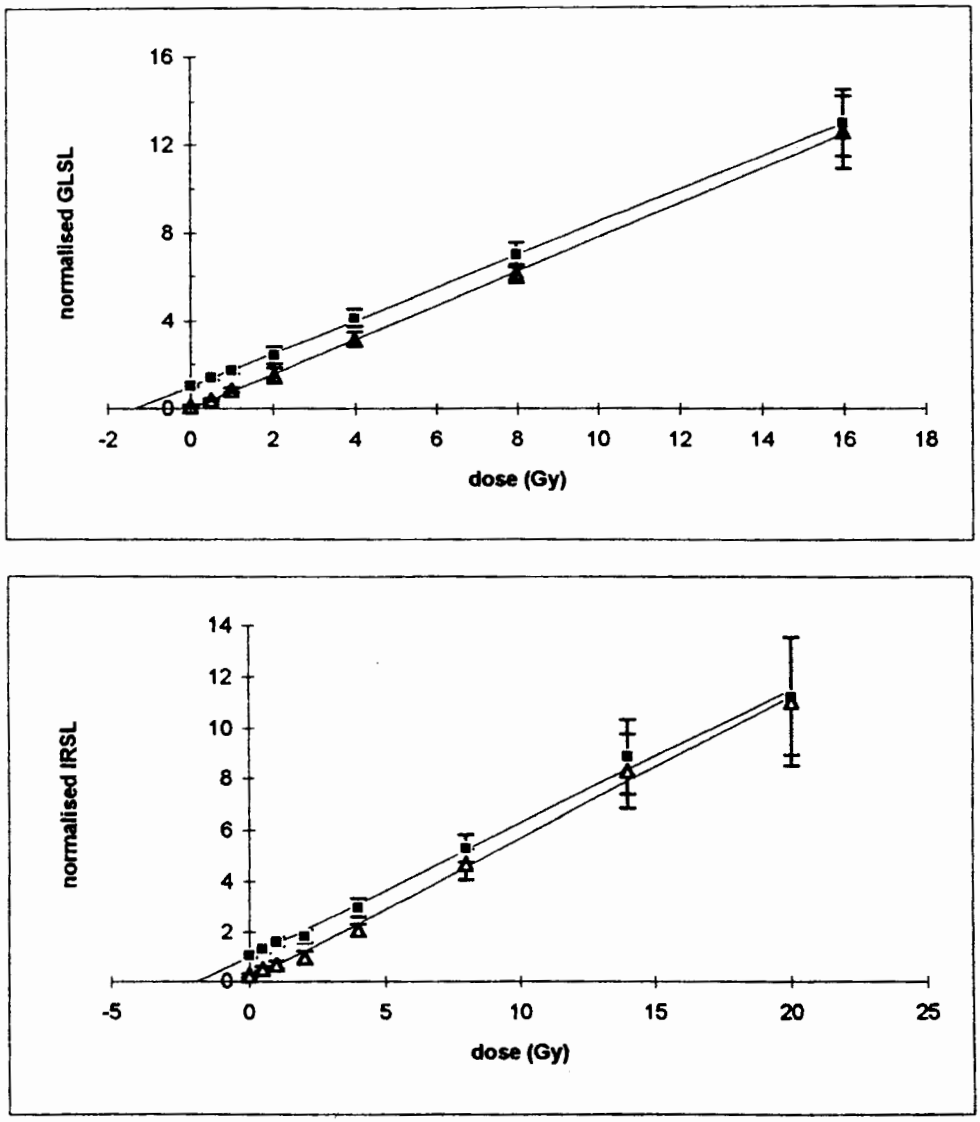

Figure 2

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.

Acknowledgements

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References

Aitken, M.J. and Xie, J. (1992). Optical dating using infrared diodes: young samples. Quat. Sci. Rev. 11, 147-152.

Berger, G.W., Mulhern, P.J. and Huntley, D.J. (1980). Isolation of silt-sized quartz from sediments. Ancient TL, 11, 8-9.

Fragoulis, D. and Stoebe, T.G. (1990). Relationship of anomalous fading to feldspar inclusions in quartz. Radiat. Prot. Dosim. 34, No.1/4, 65-68.

Fragoulis, D. and Readhead, M.L. (1991). Feldspar inclusions and the anomalous fading and enhancement of thermoluminescence in quartz grains. Nuclear Tracks and Radiation Measurements, 18, 291-296.

Godfrey-Smith, D.I., Huntley, D.J. and Chen, W-H. (1988). Optical dating studies of quartz and feldspar sediment extracts. Quat. Sci. Rev. 7, 373-380.

Huntley, D.J., Godfrey-Smith, D.I. and Thewalt, M.L.W. (1985). Optical dating of sediments. Nature, 313, 105-107.

Jackson, M.L., Sayin, M. and Clayton, R.N. (1976). Hexafluorosilicic acid reagent for quartz isolation. Soil Sci. Soc. Am. J., 40, 958-960.

Parish, R. (1992). The application of sedimentological analysis and luminescence dating to water-lain sediments from archaeological sites. Unpublished PhD thesis, University of Durham.

Readhead, M.L. (1988). Thermoluminescence dating study of quartz in aeolian sediments from southeastern Australia. Quat. Sci. Rev. 7, 257-264.

Rees-Jones, J. and Tite, M.J. (1994). Recuperation of IRSL after bleaching and consequences for dating young sediment. Radiation Measurements, 23, No.2/3, 569-574.

Rees-Jones, J. (1995). Optical dating of selected British archaeological sediments. Unpublished D.Phil thesis, University of Oxford.

Rhodes, E.J. (1990). Optical dating of quartz from sediments. Unpublished D.Phil. thesis, University of Oxford.

Smith, B.W. (1983). New applications of thermoluminescence dating and comparisons with other methods. Unpublished PhD thesis, University of Adelaide.

Smith, B.W., Aitken, M.J., Rhodes, E.J., Robinson, P.D. and Geldard, D.M. (1986). Optical dating: methodological aspects. Radiation Protection Dosimetry, 17, 229-233.

Stokes, S. (1992). Optical dating of young (modern) sediments using quartz: results from a selection of depositional environments. Quat. Sci. Rev. 11, 153-159.

Stokes, S. (1994). Optical dating of selected late Quaternary Aeolian sediments from the Southwestern United States. Unpublished D.Phil. thesis, University of Oxford.

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.