

LETTER TO THE EDITOR

Optical Dating of Quartz: A Comment on Stokes and Gaylord (1993)

Stokes and Gaylord (1993) present some interesting results concerning the optical dating of quartz sand from the Ferris dune field, Wyoming. However, we have recently discovered that the procedure used by Stokes and Gaylord results in a systematic age underestimation of 10–40% when applied to quartz sand from Australia, Africa, and Europe in the age range 0–60,000 yr. We caution readers that the specific method used by Stokes and Gaylord may also cause the age of the Ferris dune field to be underestimated.

Optical dating is a trapped-electron-based technique that can be applied to unburnt sediments to determine the time that has elapsed since the constituent mineral grains were last exposed to sunlight (Huntley *et al.*, 1985). One of the steps used in the optical dating procedure, termed the “preheat,” involves sample storage at an elevated temperature prior to optical stimulation in the laboratory. Stokes and Gaylord employed a preheat of 160°C for 16 hr (following Stokes, 1992) in preference to a preheat of 220°C for 5 min (introduced by Smith *et al.*, 1986). The latter preheat has been tested on quartz sand from Africa and Europe and has yielded a set of optical dates that are consistent with independent age estimates (Smith *et al.*, 1990a,b). Using the same optical stimulation source and preheat ovens as Smith *et al.* (1990a,b) and Stokes and Gaylord, we have recently tested both preheats on a suite of samples from Australia for which comparative ^{14}C ages are available. Our results revealed that the 160°C preheat induces a dose-dependent increase in sample sensitivity, with a concomitant underestimation of the sample equivalent dose (the dose accrued since the last exposure to sunlight), and hence the sample age, by 10–40% (Roberts *et al.*, 1993; Roberts *et al.*, in press a). Only the 220°C preheat yielded optical ages in agreement with thermoluminescence (TL) dates for the same sample and multiple ^{14}C dates for associated charcoal. We subsequently obtained the same result for samples from Morocco and England and are therefore hesitant to accept as reliable the dates presented by Stokes and Gaylord.

The optical dates presented by Stokes and Gaylord appear to be corroborated by ^{14}C ages. This is especially so for their three oldest optical dates: these range from ~8100 to 8800 yr and are stratigraphically bracketed by calibrated ^{14}C ages of 6700–8600 yr. The optical dates in

the 4000–4300 yr age range are less-well constrained by independent age control, being bracketed by calibrated ^{14}C ages of 2000–5400 yr. It is possible for a systematic age underestimation of 10–30% to go undetected for these latter optical ages. The degree of underestimation resulting from the use of the 160°C preheat need not be constant at any one site: at an Australian site, values ranged from 30 to 40% for samples collected from different depths within a single stratigraphic profile (Roberts *et al.*, in press a).

The recognition of age underestimation is complicated by the fact that Stokes and Gaylord assumed that the optically stimulated luminescence (OSL) signal grows linearly with added dose (see their Figs. 3a and 4a). However, quartz OSL exhibits a nonlinear dose response which may be approximated by a saturating exponential function (Smith *et al.*, 1990b; Roberts *et al.*, in press a,b). A linear fit in the low-dose region is only an approximation to the true form of the growth curve and is apt to overestimate the equivalent dose (Wintle, 1987; Grün and MacDonald, 1989). Even a subtle nonlinearity in an additive-dose growth curve can have a significant effect on the extrapolation used to determine the equivalent dose. Due to this effect, any age underestimate induced by the 160°C preheat may be offset by Stokes and Gaylord’s use of a linear fit over a small range of applied doses. For example, we fitted a single saturating exponential curve to the dose points shown in Fig. 4a (sample OX_{OD}833/1) and weighted each point by the reciprocal of its variance (Brumby, 1992; Lyons *et al.*, 1992). The variance was calculated from the standard error bars displayed in Fig. 4a and the number of aliquots stated in the caption to Fig. 3. The equivalent dose we determined for this sample was ~24 grays, or 75% of that obtained by Stokes and Gaylord using a linear fit. This result is not intended to be definitive, but indicative of the degree of uncertainty arising from the use of linear growth curve fits. The difference may be even greater if a saturating exponential growth curve was fitted to a larger range of applied doses. For sample OX_{OD}833/1, Stokes and Gaylord used a maximum applied dose of less than double the equivalent dose, whereas a maximum applied dose of 10 times the value of the equivalent dose is more appropriate for saturating exponential growth curves (Grün and Rhodes,

1992). At least for this sample, the overestimation of the equivalent dose by use of a linear fit may compensate sufficiently for any underestimation induced by the 160°C preheat to obscure any age discrepancy introduced by that preheat. Perhaps this explains the apparent concordance between the optical age of this sample, as reported by Stokes and Gaylord, and the comparative ¹⁴C ages.

A linear fit may not even be appropriate for the near-surface sample shown in Fig. 3a, which shows signs of supralinear growth (i.e., a concave-upward growth curve). We have observed such behavior for a quartz sand from northern Australia following a preheat at 160°C (Roberts *et al.*, in press a) and suspect also that the 160°C preheat induced supralinear growth in two quartz samples from the Netherlands (Stokes, 1991, see their Fig. 5). A linear fit to small data sets that exhibit supralinearity at small added doses may result in erroneous equivalent dose and age estimates.

In summary, the 160°C preheat used by Stokes and Gaylord in their optical dating study of the Ferris dune field is apt to yield age underestimates. At other sites, this preheat has been implicated in equivalent dose, and hence age, underestimates of between 10 and 40% (Roberts *et al.*, 1993, in press a). This value is variable even within the same stratigraphic profile, so that samples collected from several depths at a single site may exhibit different degrees of age underestimation. It is possible that the mid-Holocene optical dates presented by Stokes and Gaylord are too young by 10–30%. This could push back the most recent period of eolian sedimentation by 1000 yr, to as early as 5000–5400 yr, an age range still consistent with the ¹⁴C chronology. The early Holocene optical dates appear to be corroborated by calibrated ¹⁴C ages. This may represent the fortuitous outcome of two compensating effects: age underestimation induced by the 160°C preheat and age overestimation due to the use of linear growth curves.

Optical dating has considerable potential and demonstrated validity as a dating technique, provided the correct methods are adopted (e.g., Smith *et al.*, 1990b; Roberts *et al.*, in press b). We advise readers to treat with caution the mid-Holocene optical dates presented by Stokes and Gaylord, until the same dates are obtained using the 220°C preheat and nonlinear curve fits (e.g., Brumby, 1992) over a larger range of applied doses.

ACKNOWLEDGMENTS

The work described here was carried out at the Research Laboratory for Archaeology and the History of Art, Oxford University, with the kind permission of Professor M. S. Tite. R.G.R. was supported by a grant from the Australian Institute of Aboriginal and Torres Strait Islander Studies, Canberra, and N.A.S. and D.G.Q. were in receipt of bursaries from the RLAHA, Oxford. We extend our thanks to Professor M. J. Aitken and Mr. S. Stokes for discussions.

REFERENCES

- Brumby, S. (1992). Regression analysis of ESR/TL dose–response data. *Nuclear Tracks and Radiation Measurements* 20, 595–599.
- Grün, R., and MacDonald, P. D. M. (1989). Non-linear fitting of TL/ESR dose-response curves. *Applied Radiation and Isotopes* 40, 1077–1080.
- Grün, R., and Rhodes, E. J. (1992). Simulations of saturating exponential ESR/TL dose response curves—weighting of intensity values by inverse variance. *Ancient TL* 10, 50–56.
- Huntley, D. J., Godfrey-Smith, D. I., and Thewalt, M. L. W. (1985). Optical dating of sediments. *Nature* 313, 105–107.
- Lyons, R. G., Brennan, B. J., and Hosking, P. L. (1992). Estimation of accumulated dose and its uncertainties: Potential pitfalls in curve fitting. *Ancient TL* 10, 42–49.
- Roberts, R. G., Spooner, N. A., and Questiaux, D. G. (1993). Cautions on the use of extended duration preheats in the optical dating of quartz. *Ancient TL* 11, 47–54.
- Roberts, R. G., Spooner, N. A., and Questiaux, D. G. (in press a). Palaeodose underestimates caused by extended duration preheats in the optical dating of quartz. *Radiation Measurements*.
- Roberts, R. G., Jones, R., Spooner, N. A., Head, M. J., Murray, A. S., and Smith, M. A. (in press b). The human colonisation of Australia: Optical dates of 53,000 and 60,000 years bracket human arrival at Deaf Adder Gorge, Northern Territory. *Quaternary Geochronology*.
- Smith, B. W., Aitken, M. J., Rhodes, E. J., Robinson, P. D., and Gellard, D. M. (1986). Optical dating: Methodological aspects. *Radiation Protection Dosimetry* 17, 229–233.
- Smith, B. W., Rhodes, E. J., Stokes, S., and Spooner, N. A. (1990a). The optical dating of sediments using quartz. *Radiation Protection Dosimetry* 34, 75–78.
- Smith, B. W., Rhodes, E. J., Stokes, S., Spooner, N. A., and Aitken, M. J. (1990b). Optical dating of sediments: Initial quartz results from Oxford. *Archaeometry* 32, 19–31.
- Stokes, S. (1991). Quartz-based optical dating of Weichselian coversands from the eastern Netherlands. *Geologie en Mijnbouw* 70, 327–337.
- Stokes, S. (1992). Optical dating of young (modern) sediments using quartz: Results from a selection of depositional environments. *Quaternary Science Reviews* 11, 153–159.
- Stokes, S., and Gaylord, D. R. (1993). Optical dating of Holocene dune sands in the Ferris dune field, Wyoming. *Quaternary Research* 39, 274–281.
- Wintle, A. G. (1987). Thermoluminescence dating of loess. *Catena* 9(Suppl.), 103–114.

RICHARD G. ROBERTS¹

*Division of Archaeology and Natural History
Research School of Pacific and Asian Studies
Australian National University
Canberra ACT 0200, Australia*

NIGEL A. SPOONER²

DANIÈLE G. QUESTIAUX

*Research Laboratory for Archaeology and the History of Art
Oxford University
6 Keble Road
Oxford OX1 3QJ, United Kingdom*

¹ To whom correspondence should be addressed.

² Present address: Division of Archaeology and Natural History, Research School of Pacific and Asian Studies, Australian National University, Canberra, ACT 0200, Australia.