A source of variability in the thermoluminescence of quartz

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Variability in the TL of quartz extracted from sediments is well known, and is now widely attributed to the presence of a small proportion of "bright" grains (for example: Benkő (1983), Huntley and Kirkey (1985)). Following the quantification of this "disc-to-disc" scatter for samples of Australian sedimentary quartz (see Fig. 1) a search was conducted to locate and , if possible , characterise the reasons for the effect.

Previously, examination by X-ray fluorescence spectrometry of the purity of different samples of separated 100 μm quartz grains (following TL measurement) had revealed the presence of levels of K, Al, and Zr significantly above those found for pure silica. This result suggested that contaminant grains of feldspar or zircon were surviving the separation steps. Subsequently, a scanning electron microscope study of the loose, separated 100 μm quartz fraction showed no grains other than quartz, so demonstrating that the disc-to-disc variability was not due to inadequate separation.

As the irregular surfaces of the loose grains prevented more detailed examination, thin sections of various samples were prepared - this mode of sample presentation permits the application of electron microprobe techniques. Individual sectioned grains were inspected using an electron microprobe analyser for features capable of accounting for the levels of K, Al, and Zr detected by the earlier X-ray fluorescence analysis. Many "microinclusions" of various accessory minerals, some of which were of species normally expected to contain radionuclides, were detected. These microinclusions may or may not contain radionuclides - the detection limits of approximately 0.1 wght. % were insufficient to resolve this question. Nonetheless , this suggests the existence of a natural thermoluminescence (NTL) component induced by radioactivity contained in microinclusions and absent in artificially induced thermoluminescence (ATL). A consequence of this may be reduced disc-to-disc scatter of ATL. This supposition is consistent with behaviour shown in Fig. 1 , where statistical testing (F test, 95% confidence level) confirms the significance of the reduction in distribution variance from NTL to ATL. Further details will be given in Spooner and Hutton (in preparation).

Conclusion

The disc-to-disc TL variability in the separated quartz samples considered here was shown as not attributable to contamination by grains of other minerals surviving the separation procedure. Electron microprobe analysis subsequently revealed microinclusions of accessory minerals inside sectioned quartz grains from these samples. Microinclusions were found not to be uncommon in the samples examined, though generally representing a small fraction of the grain volume. However, it is probable that in cases where abundant microinclusions possess high concentrations of radionuclides or high intrinsic TL sensitivity, they will be troublesome in TL dating applications.

Further work on grains bright in luminescence is planned using an image-intensifier-camera set similar to that of Huntley and Kirkey (1985) .

Notes on experimental procedures

1. Sample preparation procedure : digestion in warm dilute HCl; 5 minute wash with dilute NaOH , ultrasonic bath ; dry sieved for the 90-125 μm fraction; magnetic separation (Franz magnetic separator); etched for 40 minutes at ~20 °C in 50% HF acid .

2. Preparation of thin sections : ~100mg sample embedded in araldite disc (2 parts LC191 resin, 1 part LC177 hardener); disc base ground flat and glued to glass substrate ; excess araldite cut from the disc leaving ~0.5mm layer of araldite and grains attached to substrate; grinding wheel, then progressively finer grades of emery paper reduce this to ~30 μm. Thickness during final grinding (grade 600 emery paper) is monitored by polarising microscope; polishing is by progressively finer diamond grits (6 μm, 3 μm, 1 μm) on three separate laps for ~20 minutes/grade. Each finished thin section is ~30 μm thick, 20 mm diameter and displays polished sections of ~12,000-14,000 90-125 μm grains.

3. Electron microprobe analysis.
   Instrument: JEOL 733 Superprobe electron microprobe analyser, with attached KEVEX 7000 series energy dispersive system.
   Conditions : accelerating voltage 15kV , electron beam current 5nA.
   The X-ray spectra were acquired with a take-off angle of 40° , "live" counting time of 60 seconds and detection limits of ~0.1 wght.% . Data was corrected using standard *ZAF* procedures (Z - backscatter effect, *A* - absorption of radiation in the sample, *F* - fluorescence). Analyses were with beam diameters ranging from 20 μm to spot, and the effective beam penetration depth was ~3 μm. The thin sections received a 20 nm carbon coating prior to examination.

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PI. Reviewer's Comments (M.J. Aitken)
The identification of microinclusions likely to have a radioactive component carries us one step further in quartz TL technology. Of course the writing has been on the wall ever since Sutton and Zimmerman (1978) made fission track studies of etched quartz grains (obtained by heavy liquid separation), and found uranium-rich areas (~several hundred ppM) of less than 10 microns in size. However, they found anti-correlation with cathodoluminescence sensitivity and hence presumed the resultant TL would not be substantial.

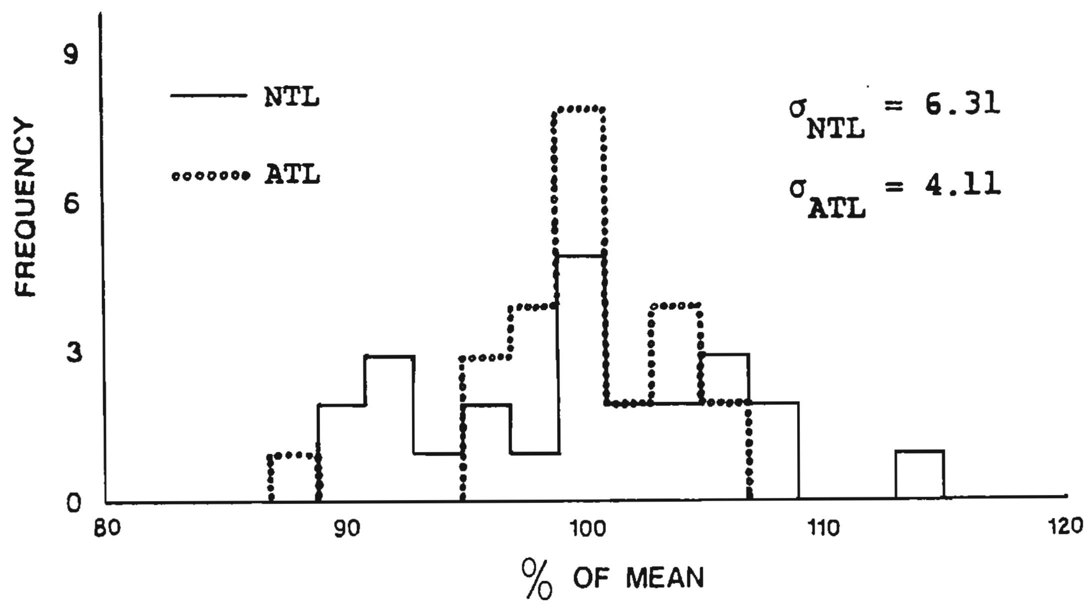

Figure 1. Distributions of the NTL and ATL of the 325 °C peak of Lake Woods quartz (laboratory reference Lake Woods (site 3, toe of dune , 1m) - further details may be found in Hutton , Prescott and Twidale, (1984), Aust. J. Soil Res., 22, 15-21.). The data is from a set of 24 similar sample discs, mean mass quartz/disc ~7 mg. The summation interval is 44 °C centered on the peak, and all TL measurements were made at 5 °K/second to 550 °C maximum, with reheats subtracted. Sample ED ~50 Gy. ATL induced by a 21.7 Gy ^{90}Sr - ^{90}Y β-dose administered following NTL measurement, and measured one day after irradiation. Quartz separation was as described in Note 1 above.