Infrared stimulation of quartz

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The ability of infrared light to stimulate luminescence from old natural quartz was reported by Godfrey-Smith et al (1988). More recently the Oxford group have reported otherwise and have been using the lack of any infrared response as an indication of the success of their quartz separation procedure (see for example Spooner and Questiaux, 1989; Smith et al, 1990). This note describes the result of an experiment attempted in order to address these differing observations.

Obvious explanations of the discrepancy are (a) that it is simply a matter of intensity and sample sensitivity, and (b) that the quartz for which the phenomenon was observed was not pure either due to inclusions within the quartz grains of, for example, feldspars, or due to an unsuccessful separation. One method of testing these ideas is to measure the spectrum of the luminescence emitted under infrared stimulation. If a contaminant is the cause then likely candidates are K-feldspars which, Hütt et al (1988) showed, have a remarkably high sensitivity to infrared stimulation. The emission spectra of these is primarily a band at 400 nm while plagioclase feldspars show predominantly a band at 570 nm (Huntley et al, 1991). Zircons, which are also 'bright' have bands at 485 and 580 nm (Godfrey-Smith et al, 1989). The only quartz for which an emission spectrum is available so far, and which is the quartz on which the present experiments are done, shows a band at 365 nm (Huntley et al, 1991). There is then the possibility for the luminescence spectrum to give some indication of the mineral responsible.

The sample for which the results of measurements are reported here is quartz separated from the East Naracoorte dune in southeast South Australia. It is about 800,000 years old (Idnurm and Cook, 1980). The material is calcarenite, for this sample being about 81% $CaCO_3$ and 16% quartz with minor amounts of feldspars and heavy minerals; the potassium content is 0.2%. 100 μm grains were separated using HCl, sieving, HF, magnetic separation, and density separation using sodium polytungstate. X-ray powder diffraction showed only lines attributed to α-quartz. Infrared irradiations were performed using an array of 16 Telefunken model TSUS-5402 diodes (950 nm emission) mounted in a ring over a TL oven similar to the arrangement described by Poolton and Bailiff (1989). The spectra were determined by successively placing different Schott longpass filters between the sample and photomultiplier tube (equipped with a KG-1 and two BG-38 filters to absorb scattered IR from the diodes), and using the measured luminescence intensity differences to calculate the emission intensity at the appropriate wavelength. This procedure is described in detail in Jungner & Huntley (1991). (In our case there were no mirrors of unknown reflectivities). There was about 2% draining of the sample by each IR exposure (5 s at 10 mW/cm^2) and this was corrected for. In addition, two samples were measured, one from short to long wavelengths and the other in the reverse direction, as a check.

The results obtained from these two samples are the curves shown in figure 1a. There is good agreement between the different results except at the short wavelength end. The differences here could be due to a change in spectrum with draining, or just scatter in the data; more measurements would be required to clear this up. In order to test whether or not the spectrum altered as a result of trap draining one of the samples was measured again after an additional IR exposure of 25 mW/cm^2 for 100 s. This was measured using 25 mW/cm^2 and the result is the lower curve in the same figure; there is some evidence of a change.

It is apparent that the dominant emission from the sample is in the 300-450 nm range and that there is relatively little emission between 450 and 600 nm, above which the BG-38 filters are non-transmitting. The emission spectrum of the same quartz under 647 nm stimulation is shown in figure 1b; this is quite different. Also very different are plagioclase and zircon spectra (Huntley et al, 1989, Godfrey-Smith et al, 1989).

The emission at wavelengths greater than 400 nm appears similar to that of the 400 nm band observed in K-feldspars, an example of which is shown in figure 1b. Explanation of the emission from 330-380 nm is more difficult. While some of this could be due to the quartz we note that Bailiff and Poolton (1991) have shown that feldspars exhibit a variety of different spectra in this region; their spectrum for an albite comes closest to the present spectrum and is shown in figure 1b also.

While we cannot make any definite conclusions it does seem likely that the emission is not from the quartz itself and that possible candidates are feldspar inclusions in the quartz grains. Such inclusions have been observed by Spooner (1987, p.87) in similar quartz grains from the Woakwine Range. We must note, however, that the current information on spectra is limited and that we cannot rule out the possibility that the emission is indeed from the quartz, or from some other mineral as yet unidentified.

Acknowledgements

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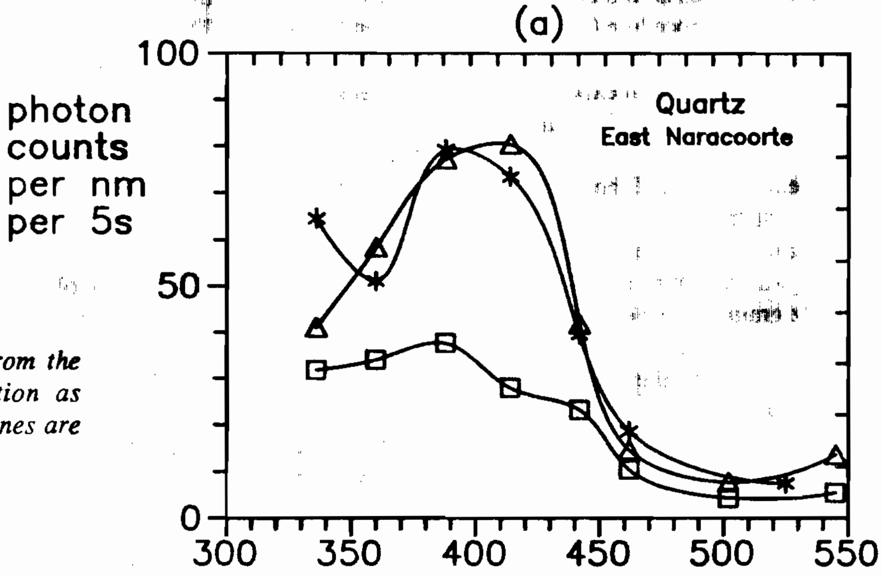
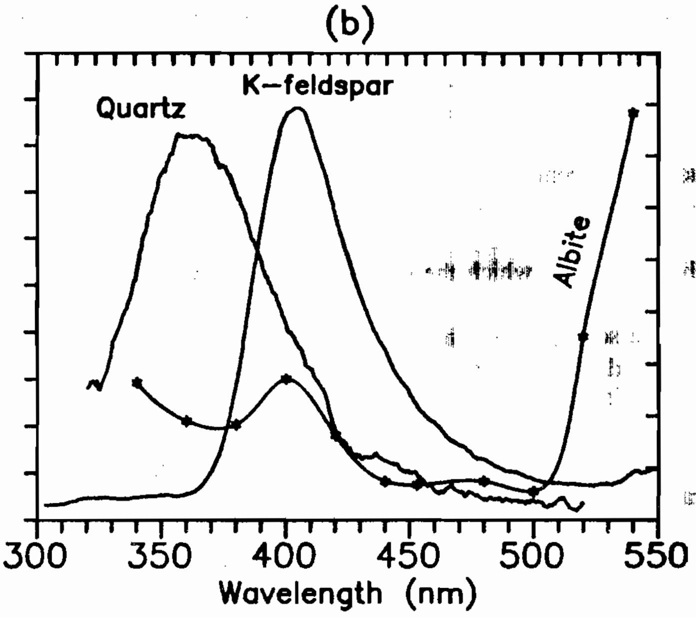
Figure 1.



(a) Emission spectra obtained from the quartz under 950 nm stimulation as described in the text. The solid lines are cubic spline fits through the data.



(b) Emission from the same quartz under 647 nm stimulation and K-feldspar under IR stimulation (Huntley et al, 1991), and an albite under IR stimulation (Bailiff and Poolton, 1991).

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The effect of shallow traps: a possible source of error in TL dating of sediments

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In a recent TL dating study of Eemian and Early Weichselian deposits in Denmark (Kronborg and Mejdahl 1989) severe underestimates of ages were encountered, e.g. ages of 80-90 ka for typical Eemian deposits with an expected age around 125 ka. The same result was obtained with optically stimulated luminescence (Mejdahl unpublished). Similar underestimates for deposits of comparable ages have been reported by Balescu et al. (1991) for K- feldspars from interglacial beach sands. A small part of the underestimate, perhaps 10 ka for ages around 100 ka, might be ascribed to thermal fading (Mejdahl 1988a, 1989). In addition, it has been found that the use of a particular filter, Schott UG11, could cause underestimates (Balescu and Lamothe (1992), Balescu et al. (1992), Grün et al. (1989) and Wintle and Duller (1991)). In the measurements of the Danish sediments where a blue filter, Corning 5-60, was used rather than the UG11 filter the large difference between measured and expected ages is still unexplained.

Wintle and Packman (1988) and Shlukov et al. (1992) pointed out that a substantial underestimate of luminescence ages might result from the large difference between natural dose rates and those used in laboratory irradiation of samples combined with presence of shallow traps in the minerals. When minerals are irradiated in nature such traps maintain only a very small equilibrium population of trapped charge, but during laboratory irradiation they become filled and may go into saturation. If the shallow traps saturate at a lower dose than that required for saturation of the deeper traps used for dating, additional charges become available for the deeper traps with the result that from a certain dose level the growth of the luminescence signal with dose will be faster for laboratory irradiation than for irradiation in nature.

In the present work potassium feldspar was studied. After laboratory irradiation this mineral has a TL peak at about 150 °C (heating rate 8 °C/s) in addition to the two characteristic peaks at around 230 °C and 330 °C present in laboratory irradiated as well as natural samples (see e.g. Bøtter-Jensen et al. 1991, fig.4, and Mejdahl 1990, figs.7 and 8).

The situation arising in dating experiments is illustrated in fig.1 where the natural growth curve has been simulated (fig.1, B) by holding the samples at a temperature of 130 °C during laboratory irradiation (60Co gamma), thus keeping the traps corresponding to the 150 °C peak empty as in nature. After the irradiation the samples were kept at a temperature of 100 °C for one week in order to eliminate the effect of anomalous fading. It is seen that when a growth curve obtained by laboratory irradiation of samples held at room temperature (fig.1, A) is used for estimating the palaeodose rather than the "natural" growth curve (fig.1, B) then the palaeodose will be underestimated, especially when using the regeneration technique. A growth curve for the 150 °C peak is shown in fig.2. It is interesting to note that this peak shows initial sublinear growth, but does not appear to saturate until about 900 Gy. This is well above the dose level at which curves A and B in fig.1 deviate.

Shlukov and Sakhovets (1987) and Shlukov et al. (1992) in a study of quartz circumvented the dose rate problem as well as the problems of thermal fading and "radiation fading", i.e. the eviction of charges from traps caused by irradiation (also called radiation quenching), by constructing a natural "master curve" which in its simplest form is an exponential function:

$$S = S_o\ (1-\exp(-D/D_s)) \qquad (1)$$

where
S is the luminescence signal. S_o is the signal at saturation. D is palaeodose. D_s is a parameter to be determined.

The expression (1) was found to be valid for a specific mineral from a certain region. S_o was determined by measuring the signal from infinitely old samples (about 30 Ma) from the region and D_s was determined by measuring the signal from a sediment of known age. The method assumes that minerals from different sediments have the same growth characteristics and that the signal was bleached to its minimum value at the time of deposition.

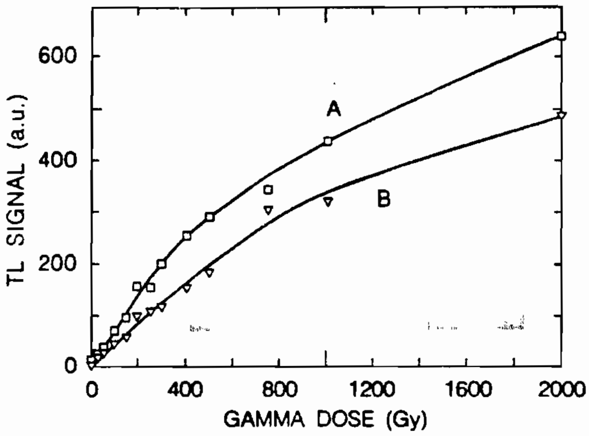

Figure 1.
TL growth curves for the 330 °C peak in K-feldspar samples from a Danish site, Stensigmose (R-909008). Natural samples were exposed to a Philips fluorescent lamp, TL05 for 50 h and irradiated with ^{60}Co gamma radiation at a dose rate of about 70 Gy/min. The samples used for curve A were irradiated at room temperature and those for curve B at a temperature of 130 °C. Prior to the TL measurements the samples were kept at 100 °C for one week in order to eliminate the effect of anomalous fading.

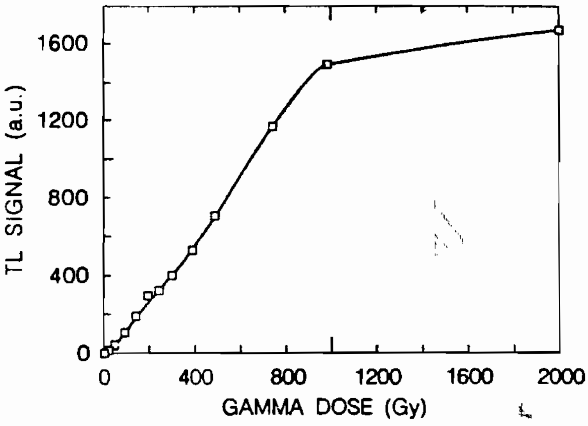

Figure 2.
TL growth curve for the 150 °C peak in a K-feldspar sample from Stensigmose.

The approach in the present paper, which deals with K-feldspar in the 100-300 µm range extracted from natural sediments, was to attempt to suppress the 150 °C peak by holding the samples at an elevated temperature during irradiation, thereby simulating the natural situation. This idea was proposed by Wintle and Packman (1988) in a study of fine-grain loess samples. Using a range of temperatures up to 123 °C and a dose of about 130 Gy they studied the shape of the resulting glow curves, but found no effect beyond a glow curve temperature of 270 °C. As suggested by A.G. Wintle (personal communication) it is conceivable that the dose used was too low for a clear effect to be seen.

The suppression of the 150 °C peak by irradiating a sample at 130 °C is illustrated by comparing the glow curves in figs.3 and 4. Two natural samples of K-feldspar (lab.no. R-909008) from a Danish site, Stensigmose were exposed to a Philips fluorescent lamp, TL05 for 50 h and then given a gamma dose of 200 Gy. One sample was irradiated at room temperature and the other at a temperature of 130 °C. TL glow curves for the two samples measured immediately after the irradiation are shown in figs. 3 and 4, respectively. The heating rate was 8 °C/s and the filters used were BG39 and HA3. The effect of suppressing the 150 °C peak is clearly seen. 130 °C was chosen as the lowest temperature that would cause an effective suppression of the 150 °C peak. It was verified that holding the samples at 130 °C caused no draining of the higher temperature peaks over the period of irradiation.

The effect of holding samples at an elevated temperature during irradiation was tested on a series of K-feldspar samples of different ages from the Thule area, Greenland (Kronborg and Mejdahl 1990). Figs.4 and 5 show plots of palaeodoses and TL ages, respectively, obtained by irradiating samples at 130 °C against the values obtained by irradiating at room temperature. The irradiation was carried out at a dose rate of about 70 Gy/min using a Co-60 gamma source. Palaeodoses were measured by means of the partial bleach technique of Mejdahl (1988b).

The regression lines for the dose and age plots have the equations:

$$Y = 0.87 + 1.35X \quad (2)$$

and

$$Y = 0.70 + 1.35X \quad (3)$$

respectively, i.e. they are almost identical. The correlation coefficients are high, 0.978 for both plots, but it should be emphasized, nevertheless, that with only nine points the results must be regarded as preliminary. It should be noted that even though the regression lines have been extrapolated to intercept the axes, the relationships cannot without further studies be extended to regions above and below those covered by the points.

The validity of the method on a regional basis was confirmed by extensive tests on sediments from the great Russian plain. Detailed studies of sediments from other regions including Caucasus, The Carpathians, The Urals, Altai and The Far East revealed certain regional

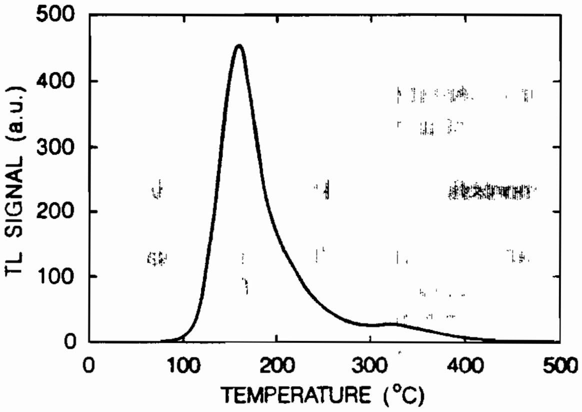

Figure 3.
TL glow curve for a K-feldspar sample from Stensigmose, Denmark (R-909008). The sample was exposed to the Philips TL05 lamp for 50 h and then given a gamma dose of 200 Gy at room temperature. The TL signal was measured immediately after the irradiation. Heating rate 8 °C/s, filters BG39 and HA3.

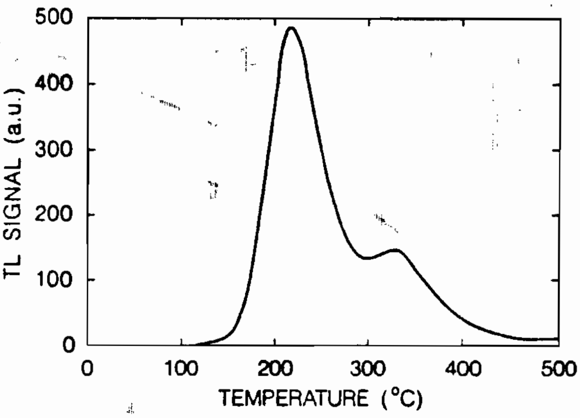

Figure 4.
TL glow curve for a K-feldspar sample from Stensigmose, Denmark (R-909008). Experimental conditions as in fig.3 except that the sample was held at 130 °C during gamma irradiation.

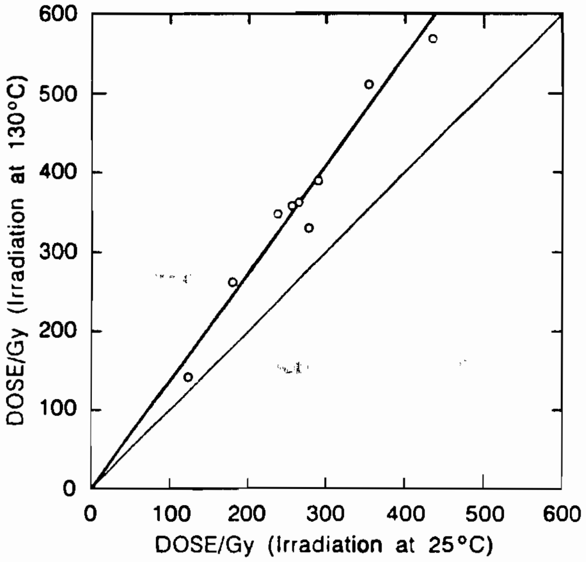

Figure 5.
Plot of palaeodoses for K-feldspar samples obtained by holding samples at 130 °C during gamma irradiation against those obtained by irradiation at room temperature for a series of samples from the Thule area, Greenland.

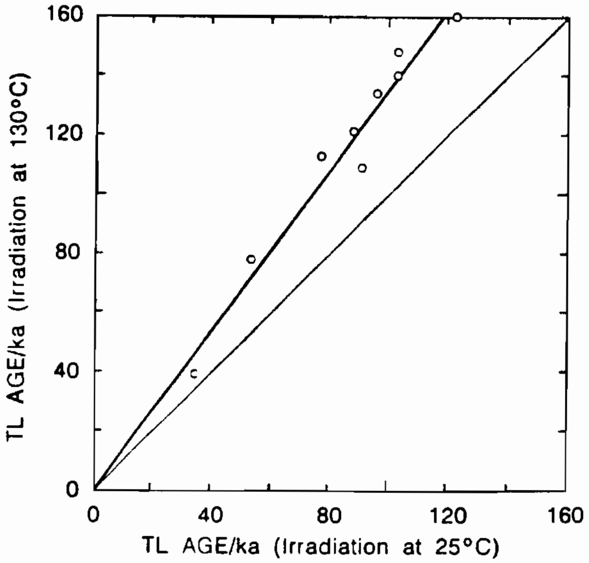

Figure 6.
Plot corresponding to fig. 5, but with TL ages rather than palaeodoses.

variations, e.g. in saturation levels. A general relationship appeared to be: the younger the massif, the lower the saturation level.

Because there are no data points for doses smaller than 100 Gy, the magnitude of the corrections (if any) for young feldspar samples is unclear. TL dating results for Late-Glacial feldspar samples from the Nordic region obtained by Mejdahl (1991a) indicate that there should be no systematic age underestimate for samples of this age. However, Grün et al. (1989) and Dijkmans et al. (1992) reported age underestimates for similar sands. It is unclear whether these uncertainties were caused by the effect examined in the present paper or by the choice of filters (Wintle and Duller 1991).

For feldspar samples older than 100 ka it is to be expected that thermal fading will constitute a significant source of error (Mejdahl 1988b, 1989).

The results shown in figs. 5 and 6 demonstrate that the effect of shallow traps could well be the main cause of the underestimates encountered. The figures further indicate that a simple linear correction procedure should apply, at least over a certain part of the dose and age ranges. Obviously, more extensive tests are required to verify the general validity of this approach, especially whether the same relationships are valid for other regions. The possibility of a sensitivity change as a result of the heating should also be investigated.

It should be noted that because the effect discussed in the present paper relates to trap filling and not to any particular method of releasing the luminescence signal, it may be expected to affect OSL dating as well.

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Notices

UK Luminescence and ESR Seminar

Change of venue and dates: British Museum, 15-16 December 1992

The location and location of the Seminar have been changed since the planned dates clash with those for a number of other meetings due to be held at the end of September. The Seminar will now take place at the British Museum.

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