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High sensitivity TL spectra of quartz and feldspar

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Introduction

During the last three years a new high sensitivity TL spectrometer has been designed and constructed at the University of Sussex (Luff and Townsend, 1993). The basic arrangement of the new instrument is shown in fig.1. The detection system features a pair of spectrometers with gratings blazed for the UV-blue (200-450nm) and blue/green-red (400-800nm) and a pair of IPDs (imaging photon detectors). The performance details are as follows: temperature ranges 20-300K, RT-400°C and wavelength range of 200-800 nm. Heating rates are typically 0.1K s^{-1} in the range 20-300K, and 2.5 °C s^{-1} above RT. The main advantage of the arrangement is that there is simultaneous recording of all wavelengths in the spectral range with photomultiplier photon counting sensitivity. For archaeological or geological TL applications, the effective sensitivity is within a factor of 10 of that of most conventional TL systems using polychromatic light via filters.

Some indication of the sensitivity of the system is that we are easily able to detect spectra from $\text{CaSO}_4\cdot\text{Dy}$ after 1 milligray of x-ray irradiation, and the signal to noise ratio is such that measurements to 10 micrograys are possible. In order to demonstrate the spectral range achieved, a spectrum for $\text{CaF}_2\cdot\text{Cr}_2\text{O}_3$ is given in figure 2. We are now able to measure the natural TL spectra of geological and archaeological quartz and feldspar. The purpose of this paper is to demonstrate the value of the additional spectral information that we have obtained in a series of preliminary experiments.

TL spectra of natural quartz fractions

The basic pre treatment procedures for samples destined for archaeological TL analysis were established in the 1970s and have remained virtually unchallenged since

then. One key procedure is the use of strong etching, with hydrofluoric acid to remove the alpha-damaged surface portions of quartz grains, and to remove any contaminant grains from quartz fractions. The efficacy of the etching method is the issue here, and we therefore present some results of a series of etching tests undertaken with the quartz fractions, in this example from dune sand collected from sand ramps in the Mojave Desert. The quartz fractions were separated using heavy liquid (sodium polytungstate) and then etched for either 40 minutes or 80 minutes in cold 40% hydrofluoric acid. Monolayers of the etched grains were prepared on clean 10mm diameter aluminium discs and the natural TL spectra recorded over the temperature range RT to 400°C. Examples of the resultant emission spectra are shown in figures 3 & 4.

Two samples were chosen to emphasise the range of behaviour which has been seen. After the 40 min etches for both of the samples DL11Q and DL12Q, the TL shows two strong emission bands at 320 and 280nm with, in the case of DL12, broader and weaker features near 400nm and 620nm. After the 80 min etch, the spectra are dramatically different, in DL11Q the intensity of the emission band at 320nm is strongly enhanced whereas in DL12Q the peaks at 280 and 320nm have almost disappeared leaving a broader (noisy) emission peaked at about 350nm and broad emission features peaked at 430 and 620nm.

Several questions are raised by this new spectral information. The first is the extent to which the relatively strong natural TL signals detected at 280 nm and 320 nm result from the presence of feldspar grains or feldspar inclusions or indeed, what evidence do we have that the 280 and 320 nm signals do not come from quartz? Our current spectral measurements made

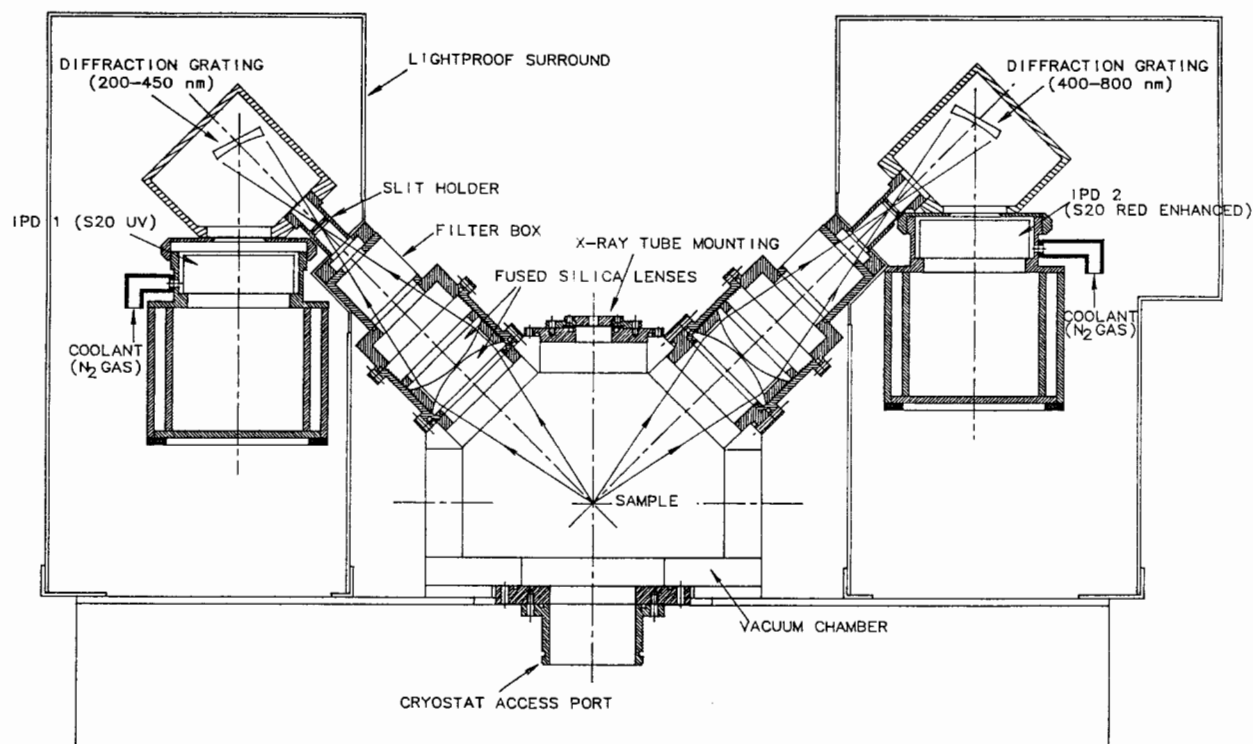


Figure 1
Schematic design of the new TL spectrometer.

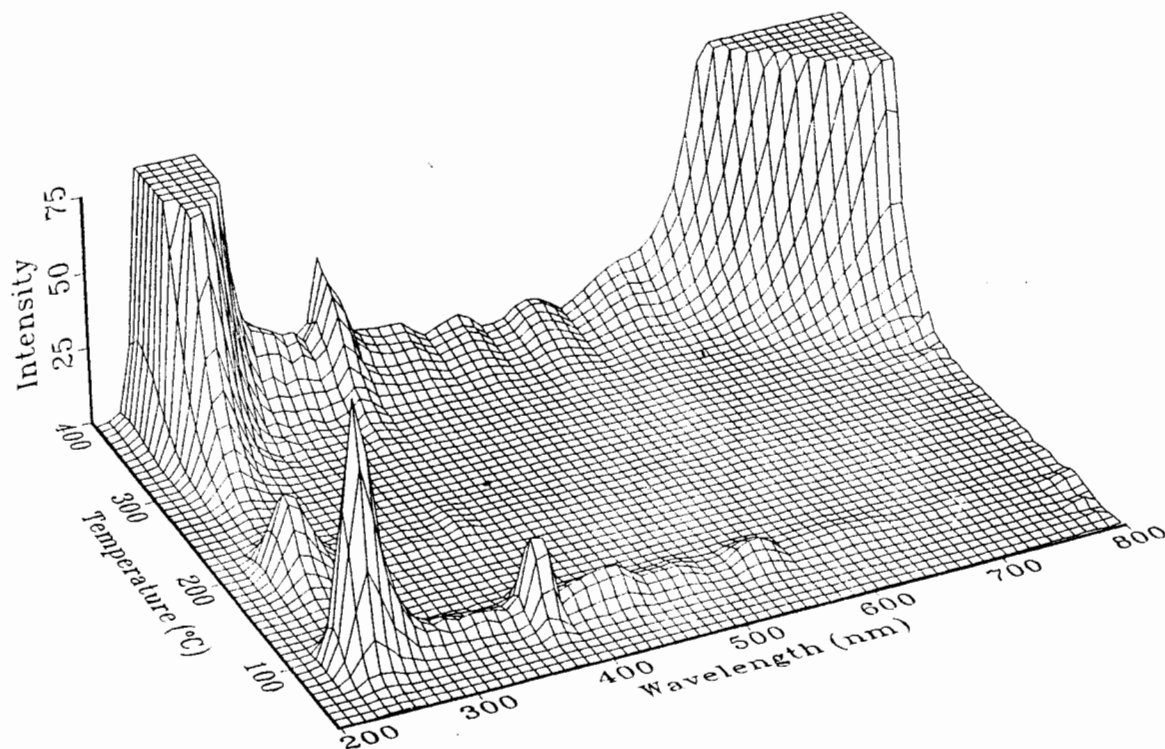


Figure 2.
TL emission spectrum for $\text{CaF}_2:\text{Cr}_2\text{O}_3$ after x-ray irradiation.

on silicate groups (SiO_4), the recombination luminescence which is strongly localised will appear at nominally the same wavelength, no matter which is the host material.

In the case of quartz and silica emission, peaks reported in this blue/UV region include examples at 260, 290, 380, 390, 400, 450, 460, 470 and 480 nm. The models ascribed to them are variations on the intrinsic $e^- - h^+$ recombination, near 470 nm, or perturbations of the SiO_4 group caused by intrinsic defects (oxygen vacancy, E' or E'_{beta} centres) or impurities such as H, Al or Ge. The variations in sample purity and defect states imply that quartz and feldspar signals may be coincident in wavelength. The chemical etching in HF removes the exposed feldspar and the alpha-damaged quartz more rapidly than quartz which only contains isolated point defects. The crucial observation is that changes in spectra are still continuing after 80 min HF etching which implies that the standard HF etch for 40 min may be only partially successful in separating quartz grains from other material.

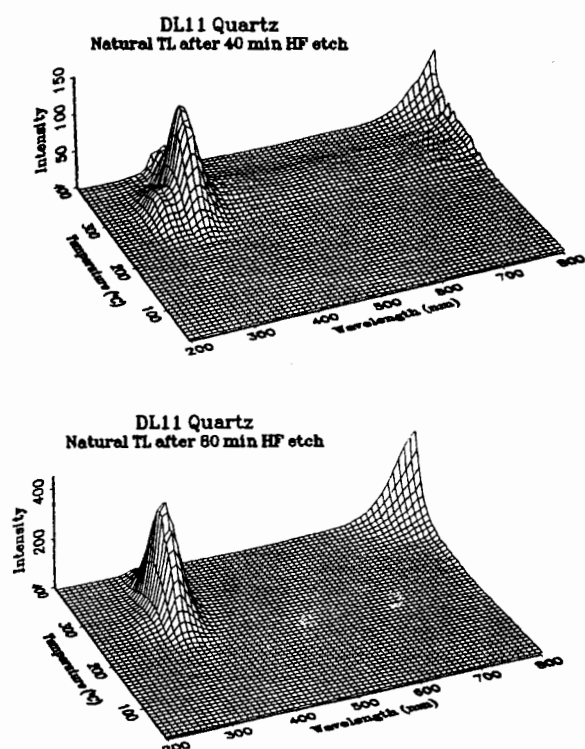


Figure 3.
Emission spectra of natural TL from quartz fractions of sample DL11Q after different etching times.

The second significant observation is that for DL11Q the luminescence peak at 320 nm increases with etching. From a solid state viewpoint this is surprising but may just indicate the loss of surface defects which were offering non-radiative recombination routes. For dating, the differences between the sensitivity to etching for the component emission bands should be noted and their subsequent influence on dating needs careful consideration.

TL spectra of natural feldspar

The results of TL dating of potassium feldspars have been the subject of considerable debate (Balescu & Lamothe, 1992; Rendell, 1992; Wintle & Duller, 1992). During this debate it has become apparent that in the case of some samples, the values of Equivalent Dose obtained were a function of the choice of emission band detected via a particular broad-band filter combination. The choice of filter has until now been a result of educated guess-work. The potassium feldspar fraction was separated from a small sample of material from the Belcroute site in Jersey (Balescu and Lamothe, 1992). The fraction comprises the 355-250 μm grains

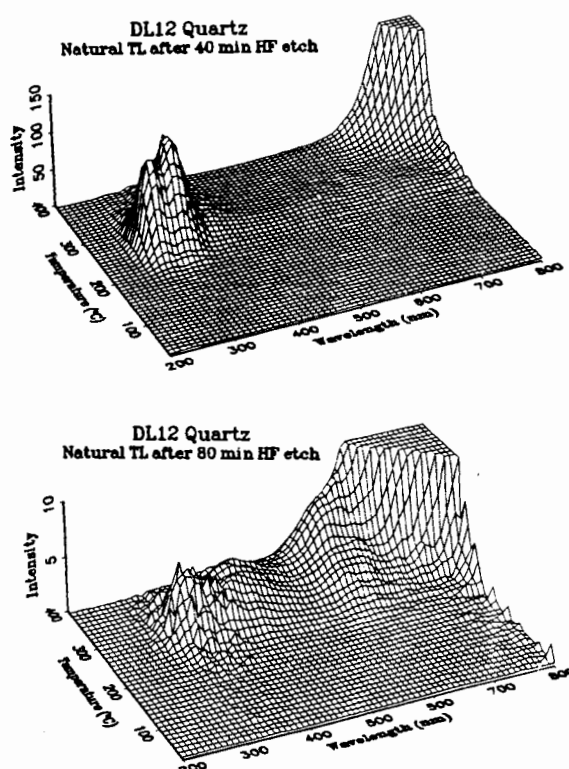


Figure 4.
Emission spectra of natural TL from quartz fractions of sample DL12Q after different etching times.

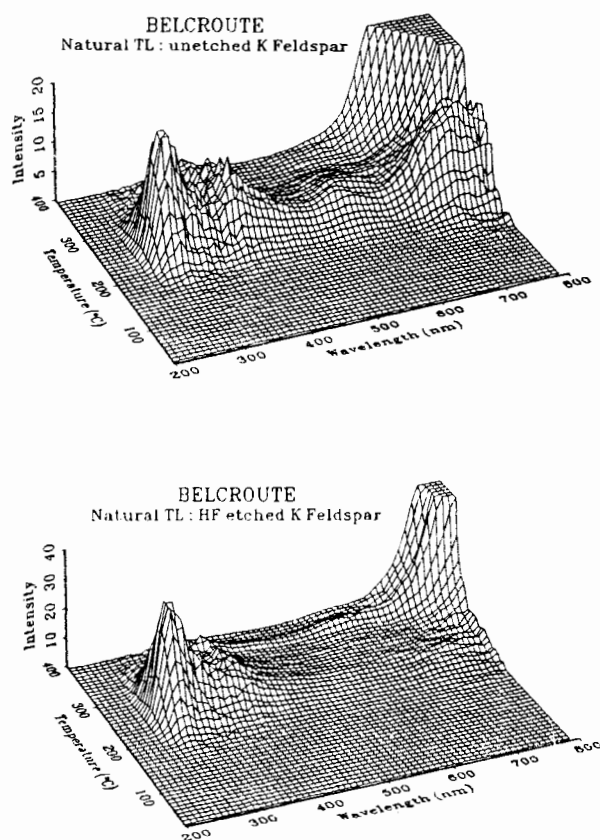


Figure 5.
Emission spectra of natural TL from potassium feldspar fraction of a sand sample from Belcroute before and after etching.

with s.g.<2.58. Spectra of the natural TL are shown before and after etching in 10% hydrofluoric acid (figs.5a and 5b).

The reason for showing both spectra is that, within the luminescence community, some consideration has been given to trying to date separated, but unetched, material. The spectrum of the unetched material (fig.5a) shows emission bands at 280, 340, 380-400, 550 and 730 nm, with glow curve shape varying as a function of wavelength in the blue/UV region. The spectrum of the etched material is dominated by emission in the blue/UV (fig.5b). In particular the intensity of the 280nm emission has increased relative to that at 380-400nm at 260°C, however at temperatures above 320°C, the latter emission band has a higher intensity.

Any simple filter/PM tube combination will thus offer an integrated TL signal which will certainly be unsuitable for kinetic analysis. More importantly, for dating applications a filter view of such signals will be misleading if the ratio of the component glow peaks and spectral features vary with dose, storage, bleaching or re-irradiation. Unfortunately for this material these variations do indeed exist and so one may derive erroneous ED values. With the hindsight of the spectral information, one should pursue normal TL measurements for such materials using a much more precisely selected spectral region, with perhaps a monochromator, rather than a broad band glass filter.

Acknowledgements

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This is an interesting series of observations which needs further study. A methodical investigation of the effects of different filter arrangements on calculated EDs is called for. Regarding figure 3, in addition to the increase in TL emission, there is also an evident increase in the black-body emission. It is not obvious why this should be so.