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# THREE-DIMENSIONAL THERMOLUMINESCENCE SPECTRA AND THEIR APPLICATION IN THE STUDY OF SOME SEDIMENTARY QUARTZ

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## Abstract

The intensity of thermoluminescence emission is a function of both temperature and photon energy (or wavelength) and therefore lends itself to a three-dimensional (3-D) display, either in the form of an isometric plot or a contour diagram. The technique of 3-D thermoluminescence spectrometry is briefly reviewed and the advantages and applications are illustrated by reference to the spectral properties of samples of Australian sedimentary quartz drawn from the context of thermoluminescence dating. In this context, the temperature of emission, the photon energy and the ease with which the emission is bleachable by sunlight are of significance. It is suggested that, in one of the samples, the energy of the photon emitted from one of the luminescence centres depends on the temperature of emission.

Key Words: Thermoluminescence, three-dimensional spectra, sedimentary, quartz, dating, solar bleaching.

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# Introduction

Thermoluminescence (TL) can be used in the general field of luminescence for the study of the trap levels of individual crystal systems. In recent years, most research into TL has been directed toward its use in applications viz. radiation dosimetry, TL dating and mineralogy. The present paper has its origins within the context of TL dating. However, although the questions addressed have been stimulated by work in that field, the results will be of more general interest.

The intensity of TL emission is a function of both temperature and photon energy (or wavelength), and there is an extensive literature on glow curves (TL intensity vs. temperature) and spectral emission of a wide variety of materials (McKeever, 1985; Townsend and Kirsh, 1989). The three-volume handbook, edited by Horowitz (1984), deals primarily with dosimetry, with little discussion of spectra. Sankaran et al. (1983) review the TL of geological materials. Compilations of TL spectra of minerals have been made by Tarashchan (1978) and Gorobets (1981) in Russian, and some of this is reported in English by Marfunin (1979). Although it deals with cathodoluminescence, Marshall (1988) is a useful source of reference for spectra, since the characteristics of the materials are common to most luminescence phenomena. The paper by Townsend and Kirsh (1989) is particularly to be noted for its discussion of the value of spectral measurements in interpreting and applying TL.

Levy et al. (1971) and Levy (1979) seem to have been the first to point out that TL intensity, temperature and emitted photon energy together lend themselves to a three-dimensional (3-D) display, either in the form of an isometric plot or a contour diagram. This representation adds materially to the ability to interpret the behaviour of the substances being studied. The temperature peaks correspond to traps from which electrons are released on heating and the spectral emission bands carry information about the charge recombination sites. Not every temperature peak emits at the same photon energy. Hence, information about the solid state properties of the material itself, e.g., trap depths, pre-exponential factors, excitation probabilities, or the nature of the luminescence centres and whether they compete, can be found and protocols developed for use in applications. The use of specifically 3-D spectra has been subsequently developed, particularly by the groups at Adelaide (Prescott *et al.*, 1988) and Sussex (Townsend and Kirsh, 1989). Individual 3-D spectra are to be found scattered through the literature but it is only relatively recently that systematic accounts have begun to appear exploiting the 3-D concept (Fox *et al.*, 1988; Townsend and Kirsh, 1989; Prescott *et al.*, 1989, 1994; Rink *et al.*, 1993; Prescott and Fox, 1993; Rendell *et al.*, 1994).

#### **Spectral Measurements**

The designer of a 3-D spectrometer for TL has to address two challenging problems: the variation with temperature and hence with time, and the low intensity level. It is characteristic of TL measurements that the temperature changes monotonically during observations. Consequently, spectral measurements must be made continuously or, in practice, as discrete samples over small specified temperature intervals. Further, in applications such as personnel dosimetry and TL dating, the TL intensity is low because the radiation dose that gives rise to the stored TL energy dose is low. Typically, for the former, using high-sensitivity phosphors, the dose will be a few mGy, while for dating and mineralogical applications, the dose for much the same amount of light will be of the order of tens of Gy.

A conventional dispersive monochromator, using a grating or prism with photomultiplier recording, selects only a small portion of the spectrum at a time. Such instruments are not optically efficient because they accept only a small solid angle at the input because good resolution requires a narrow slit, and they select a narrow wavelength band of the dispersed spectrum at the output. The sensitivity can be improved by upwards of two orders of magnitude by replacing the exit slit and scan system with a detecting array that records all wavelengths simultaneously; this is known as the multiplex advantage. The use of image intensifiers, arrays of charge-coupled devices, intensified diode arrays, position-sensitive photomultipliers and multichannel-plates have been reported (Walton, 1982; Bakas, 1984; Huntley et al., 1988a; Hornyak and Franklin, 1988; Luff and Townsend, 1993; Piters et al., 1993; Rieser et al., 1994).

An alternative solution, which eliminates the slit, is to have an extended source viewed through a succession of interference filters inserted in the beam sequentially (Bailiff *et al.*, 1977; Delunas *et al.*, 1988). The collected light is much increased but wavelength bands are sampled one at a time.

Other things being equal, the sensitivity of an optical system is expressed by its *etendue*, or optical throughput (Jacquinot, 1954, 1960; Steel, 1983; Meyer-Arendt, 1984). This is the product of the solid angle and the beam area and is expressed in units of mm<sup>2</sup>·sterad. It is an invariant of any optical system, apart from absorption losses.

The *etendue* of dispersive spectrometers is necessarily limited because it is difficult to reduce the aperture much below f/2 (which constrains the solid angle) and because the slit has to be narrow for good resolution. The latter is not a critical restriction for TL measurements because the band-width of the emitted radiation is commonly of the order of 10 nm or more and the slit can be made wide enough to admit more light without compromising resolution. The *etendue* of a dispersive spectrometer with f/2 input optics and a 1 cm x 0.1 cm slit is approximately unity. Probably the most sensitive and versatile of present-day dispersive spectrometers is that of Luff and Townsend (1993).

Jacquinot (1960) pointed out that interferometers, such as Fabry-Perot and Michelson, can both accept light from an extended source and code all wavelengths simultaneously into the interferogram. Although the multiplex advantage may not be fully realised, the *etendue* of an interferometer more than compensates for this and the sensitivity of an interferometer is about an order of magnitude larger than that of a multiplexed dispersive spectrometer for the same resolution (Steel, 1983).

Spectrometers for TL measurements, based on interferometry, have been described by Prescott *et al.* (1988) and Haschberger (1991). The former was used to obtain the spectra described in the present paper. Since it has been fully described elsewhere, only a brief account of its characteristics will be given here.

The instrument is a high-sensitivity quartz optics spectrometer based on a modified Twyman-Green, Michelson type, interferometer. It covers the photon energy range 1.5-5.0 eV (800-250 nm), although for practical purposes the lower limit of energy is about 1.7 eV (730 nm) because of interference from incandescence of the sample at the higher temperatures.

The sample, prepared either as grains or a slice a fraction of a mm thick, is placed on an electrically heated plate in an inert gas and, as the temperature is raised, the light output is directed through the interferometer. As the sample is heated, the moving mirror of the interferometer is scanned backwards and forwards in successive temperature intervals. The output is normally recorded on a cooled 9558Q photomultiplier, although a 9635Q photomultiplier, which is insensitive in the red, is also used. In the latter case, the effective

photon energy limit is about 2 eV (610 nm). The output as a function of the mirror displacement is known as an interferogram and is the Fourier transform of the input spectrum, which is found from the inverse Fourier transform of the interferogram.

The apparatus employs photon counting and the data are presented as counts per mg of sample, per unit photon energy and kelvin. For the purposes of comparison, it should be noted that the transformation into events per unit wavelength and kelvin, though straightforward, changes the relative heights of peaks in the 3-D spectrum and may cause small shifts in peak location. The same is true of a transformation from events to irradiance (power). The spectra have been apodised and corrected for instrument response, which was determined using high pressure deuterium and incandescent lamps calibrated by the National Measurement Laboratory (Commonwealth Scientific and Industrial Research Organisation, Australia) and checked by combining the responses of individual components. The instrumental resolution is given by  $\lambda/\Delta\lambda = E/\Delta E = 25$ . Being largely determined by relative off-axis path length in the interferometer, it is almost independent of photon energy. The theoretical maximum etendue is 55 mm<sup>2</sup>·sterad, which includes the loss inherent in the Michelson design; reflection losses reduce this by a further factor of about 30%. The resolution can be improved if necessary, with the sacrifice of etendue.

#### Quartz

Many of the samples studied by the Adelaide laboratory have been chosen because of their relevance to TL dating, in which the two most common minerals used are feldspar and quartz.

Feldspars are alumino-silicates of alkali and alkaliearths, in which the cation concentration ranges continuously from potassium at one end (orthoclase) through sodium (albite) to calcium (anorthite) at the other. Because of the wide range of compositions, the range of observed spectral types is large (Prescott and Fox, 1993).

Quartz, by contrast, has a single mineral composition with a simple structure and large band gap, and might be expected to show relatively little variability from sample to sample. However, while there are a number of common features, the spectra turn out to be quite variable. McKeever (1984, 1985) has reviewed the TL of quartz up to those dates with further discussion in McKeever (1991). It is worth noting that David *et al.* (1977a, 1977b) found many of the spectral features described below, including changes on heating, although the work was done with samples that had received very high doses.

In TL dating, it is assumed that the TL was set to zero at some time in the past either by heating, as in the case of volcanic or ceramic samples, or by exposure to sunlight, as in the case of sediments. Heating is almost guaranteed to reset the TL clock completely, but sunlight exposure usually leaves a residual TL amount which depends on the nature of both the quartz and the exposure. Until recently, TL dating of sediments was mostly based on an empirical division into samples that have been "total bleached" or "partial bleached" and the main dating techniques were developed based on this rather crude division (Aitken, 1985). There is a 375°C component which emits in the blue at 2.6 eV (470 nm) which is not very susceptible to bleaching; its properties have been extensively discussed by Hornyak et al. (1992). Another peak at 325°C emits in the blue and ultra-violet and bleaches to essentially zero level in a few minutes of bright sunlight (Spooner et al., 1988; Prescott and Fox, 1990). The temperatures 325°C and 375°C are conventional; the peaks occur near these temperatures in most samples at a heating rate of 20 Ks<sup>-1</sup> but the actual temperatures depend on the heating rate, and to some extent, on the sample. A phenomenological description might be "rapidly-bleaching" and "slowly-bleaching," as suggested by Franklin and Hornyak (1990).

However, another broad division in quartz spectra has been described by Hashimoto in a series of papers, first reported in English in Hashimoto et al. (1986), and listed in Hashimoto et al. (1993). They have found that individual grains of natural quartz commonly show either red emission at 2.0 eV (620 nm) or blue emission at 2.6 eV (470 nm). The former come from volcanic ash layers, whereas the latter originate in plutonic rocks (pegmatite, quartz vein and granite). Dune sands reflect the origin of the quartz. They find changes in spectra following different annealing treatments, as do Rendell et al. (1994). Hashimoto et al. (1993, 1994) have advocated the use of the red component for dating. Miallier et al. (1991) have found red emissions to be common and they, too, have argued that the red is superior to blue for TL dating. Red emission has been reported by Huntley et al. (1988a, 1988b) to be common in sedimentary quartz. Rink et al. (1993) have studied the 3-D spectra of a range of igneous quartz and hydrothermal vein quartz. In broad agreement with Hashimoto (loc cit), they find that spectral types are characteristic of the mode of genesis and thermal history. In particular, volcanic quartz exhibits very intense red emission over a wide temperature range; and granitic quartz has emission bands of variable intensity across the uv, blue, violet and red.

#### Rapidly-bleaching components in quartz

One of the difficulties in applying TL methods to



Figure 1. TL spectra for sedimentary quartz sample TC2S/2.5b, taken with a 9635Q photomultiplier which is red-insensitive. This and other figures are averages of at least 4 spectra and the relative intensities are correct. In this figure, the maximum contour is set at 16 for the natural spectrum. (a) Sample glowed in its natural state (equivalent dose 64 Gy) but with addition of 1 Gy immediately before glow-out, to include the 110°C peak which is normally absent in natural samples due to thermal decay; (b) Natural sample after bleaching by a 30 minute exposure to a solar spectrum transmitted through a long wave-length pass filter cutting to 1% at 475 nm; (c) Difference of (a) and (b) showing the rapidly-bleaching components.

#### 3-D Thermoluminescence Spectra



Figure 2 (at left). TL spectra of sample SK11S/W. (a) Natural sample (equivalent dose 60 Gy). Maximum contour is 10; (b) Difference of natural and bleached spectra. Any rapidly-bleaching component is little different from background.

Figure 3 (at right). TL spectra of TC2S/2.5b. This is the same sample as shown in Figure 1, but in the present case, a 9558Q photomultiplier was used which covers the whole available spectrum. (a) Natural sample. The red (2 eV) component of the spectrum, not seen in Figure 1, is prominent and more intense than the 2.6 eV component. Maximum contour is 10; (b) Difference of the natural and bleached spectra showing that the red component is rapidly bleaching, as well as that at 3 eV seen in Figure 1.

sedimentary material is that there will always be uncertainty in the degree to which the material was reset to zero in the past. A variety of protocols are in use, or have been suggested, to take advantage of the existence of a rapidly-bleaching component on the assumption that this, at least, will have been completely or nearly completely reset. There is an additional assumption that there is, in fact, such a component (Wintle and Huntley, 1982). Such a component has been identified with the 325°C peak and it is usually reported to emit at 3.3 eV (380 nm) (Spooner et al., 1988; Franklin and Hornyak, 1990; Prescott and Fox, 1990; Scholefield et al., 1994; Franklin, 1994) although, as will be seen, the photon energy is probably smaller. Franklin and Hornvak (1990) and Prescott and Mojarrabi (1993) have suggested dating procedures, specifically using the properties of the 325°C peak.

## Quartz spectra

As an illustration of the application of 3-D TL spectrometry, the present paper reports a study to determine the extent to which the 325°C peak is found in quartz used for TL dating. While it appears to be relatively common, nevertheless, samples have been reported which do not share the bleaching properties of the "conventional" 325°C peak (Morris and McKeever, 1994) or do not show the corresponding emission (Rink et al., 1993). We have found samples in which there does not appear to be any identifiable 325°C component, bleachable or otherwise. Accordingly, we have measured a considerable number of samples of sedimentary quartz with particular attention to identifying rapidly bleaching components. In view of the advocacy of the use of the red component, cited above, some attention was paid to the bleaching in this spectral region.

Figure 1 shows 3-D spectra for sample TC2S/2.5b, an aeolian sand from Tennant Creek in the Northern Territory, Australia. This sample is typical of many Australian dunes sands from the arid interior of Australia and appears to be similar to sands from the Kalahari desert (Franklin and Hornyak, 1990). The field context and some TL properties of the quartz in TC2S/2.5b have been discussed in Hutton *et al.* (1994) and Scholefield *et al.* (1994). Figure 1 shows the spectra in both isometric display and in the form of a contour diagram.

The spectrum in Figure 1a is from the sample in its natural state; it was collected with care not to expose it to light. Immediately before glowing, a radiation dose of 1 Gy was added so that the 110°C peak, which is absent in natural samples because of thermal decay, appears in the figure. A peak near 330°C emitting at 2.6 eV (470 nm) is prominent; this is the commonly-observed "slowly-bleaching" peak. An emission of lower intensity at a temperature of about 300°C causes a projection in the contour diagram toward the short wavelength side and can also be seen in the isometric plot; this is the "rapidly-bleaching" peak.

Exposure of the sample to tens of seconds of full sunlight or tens of minutes of sunlight through a longwavelength-pass filter cutting off at 475 nm (yellow bleach) removes this component completely, leaving other glow peaks unaffected. Figure 1b shows the spectrum after 30 minutes yellow bleach.

If this second spectrum is subtracted from the first, the difference is the rapidly bleaching component; this is shown in Figure 1c. The effect of bleaching on the component near 300°C is clearly seen by comparing these three figures. In the subtracted spectrum, the difference peak (representing the rapidly-bleaching component) is seen to be broad, peaking at about 3.0 eV (410 nm). There is also a previously unsuspected minor peak near 370°C at about 2.5 eV. It is thought to be real and not due to imperfect subtraction.

It should be noted that these spectra were obtained with a 9635Q photomultiplier, which is insensitive in the red, in order to bring out the detail at the other end of the spectrum. The 2.6 eV peak is correctly located but emission in the red is suppressed. In fact, this sample also emits strongly at wavelengths near 2 eV, as can be seen in the spectrum of the same sample in Figure 3 (discussed below) which is taken with a 9558Q photomultiplier.

Virtually identical spectra, with some differences in detail, are found for PJ1ES/75, an aeolian quartz from the Cleland Hills in the Northern Territory, Australia (Prescott and Purvinskis, 1993); LW1S/1 from Lake Woods in the Northern Territory (Hutton *et al.*, 1984); WK1S/4, from the Woakwine Range as representative of some twelve former coastal dunes near Robe in South Australia (Huntley *et al.*, 1993); and EB1S/1.9 from a dune on the East Bank of the River Murray north of Blanchetown, South Australia (Prescott, 1983).

In all of these examples, the success of the dating method used (Franklin and Hornyak, 1990; Prescott and Mojarrabi, 1993) was predicated upon the existence of a rapidly-bleaching component emitting at 325°C and a photon energy near 3 eV, such as is shown in Figure 1. Although this particular property is common in quartz, we have found examples where there is no obviously identifiable rapidly bleached component. An example is given in Figures 2a and 2b. Sample SK11S/W comes from a deep palaeosol coastal dune at North Stradbroke Island, Queensland (Tejan-Kella *et al.*, 1990). The sample shows the emission near 2.6 eV above 300°C but there is little evidence of a rapidly-bleaching component similar to the one shown in Figure 1. In SK11S/W, there is a component at about 280°C which is thermally unstable on a long term basis, which is seen in samples irradiated in the laboratory but which does not clearly show in the natural sample in Figure 2.

Samples from the mound springs near Lake Eyre at the western edge of the Great Artesian Basin in the far north of South Australia (e.g., BB1S and BC1S) also have little, if any, rapidly-bleaching peak.

From the point of view of TL dating, such samples present difficulties unless there is a reasonable belief that the sample had been totally bleached in the past, although there may be a small amount of rapidly-bleaching component (which cannot be distinguished in the 3-D spectra) but which allows a date to be obtained. Morris and McKeever (1994) discuss a sample that appears not to bleach completely in the 325°C region and relate this to bleaching mechanisms.

Most of the samples of sedimentary quartz, already referred to, also emit in the red at around 2 eV (620 nm) at temperatures above 300°C. Such a spectrum is shown in Figure 3. Figure 3a shows the spectrum of the natural sample for TC2S/2.5b but now covering the full spectral range. The 330°C and 300°C peaks of Figure 1 are present but not resolved in the presence of strong red emission at 2.0 eV and 310°C. This peak is particularly interesting in that it bleaches rapidly, as Figure 3b shows. This shows the bleachable component, obtained in the same manner as for Figure 1c above by subtracting a bleached spectrum from an unbleached spectrum. The red peak is prominent but the rapidly-bleaching component at 2.7-3 eV (450-420 nm), previously discussed in connection with Figure 1, cannot be seen on the contour diagram and not clearly in the isometric plot. However, when seen in more detail it appears to emit at a different temperature from the red peak, which implies that there may be more than one rapidly-bleaching trap.

SK11S/W also possesses a rapidly-bleaching red peak at about 2 eV but a different temperature from TC2S/2.5b.

Work is currently in progress to extend our studies of the bleaching of the red component of quartz.

## Discussion

The samples described here are all from sedimentary sources, mostly aeolian dunes. For this reason, the origin of the quartz is uncertain. On the basis of the studies of Hashimoto *et al.* (1986, 1987), Rink *et al.* (1993) and Rendell *et al.* (1994), the presence of red emission suggests a volcanic origin. Miallier *et al.* (1991) found it also in samples not of volcanic origin. The blue 2.6 eV emission is attributed by Rink *et al.* (1993) to hydrothermal or pegmatite quartz and by Hashimoto *et al.* (1986) to quartz from plutonic rocks or their weathering products. In some of the samples of Hashimoto *et al.* (1986) luminescence of both colours was found, and this is true of most of our samples also. There is no evidence yet to identify the type of quartz in which the rapidly-bleaching component can be found. It is clear, however, that it is not universal although TL dating protocols based upon it will continue to be useful, because they remove the uncertainty in the degree to which the TL was set to zero in the past.

Red emission appears to be more common. Both Miallier *et al.* (1991) and Hashimoto *et al.* (1993) have shown that the lifetimes of the trapped electrons that feed the natural red TL are long enough for TL dating, at least in their samples, and have suggested that it is well-suited for dating. If it can be shown that long lifetime is a general property, then their suggestion will be sustained. The present study has shown that at least some red emission also has the property of being rapidly-bleaching and, as with the  $325^{\circ}C/3 \text{ eV}$  emission discussed above, its use for dating would remove the uncertainty about the degree to which the TL had been zeroed in the past. However, Miallier's samples differ from ours in that they bleached only slowly, even with u-v irradiation.

The electron trap with which the 325°C rapidlybleaching peak is associated has not been identified but there is good evidence that it shares the (H<sub>3</sub>O<sub>4</sub>)° luminescence centre with the 110°C peak which features prominently in pre-dosing (e.g., Yang and McKeever, 1990; Scholefield et al., 1994). Stoneham and Stokes (1991) establish a connection between optically stimulated luminescence ("OSL" in the parlance of luminescence dating) and the luminescence centre serving the 110°C peak. The 325°C trap is also clearly identified with the trap from which OSL releases electrons (Smith et al., 1986; Smith and Rhodes, 1994). The electrons reach the luminescence centre via the conduction band for OSL (Smith and Rhodes, 1994) and our work in progress strongly suggests that the same is true for TL. It is perhaps necessary to remark that the slowlybleaching blue component at 375°C/2.6 eV belongs to an independent system. It does not share the (H<sub>3</sub>O<sub>4</sub>)° centre and probably is not emptied into the conduction band (Franklin, 1994). Its recombination centre is a hole trap at Al substituting for Si to give (AlO<sub>4</sub>)° (Jani et al., 1984; McKeever, 1991; Rink et al., 1993). The red emission is also independent and has been attributed by Hashimoto et al. (1993, 1994) to Eu<sup>+</sup> or Al but little is known about the traps that feed it.

These results lead to the interesting observation that the energy of the photon emitted from the common  $(H_3O_4)^\circ$  recombination centre depends on the temperature (Scholefield *et al.*, 1994). As Figure 1 shows, the photon energy following release of electrons from the 325°C peak is about 3 eV. For emission at 110°C, the energy is 3.3 eV (Bailiff, 1979; Akber *et al.*, 1988; and Fig. 1c above), whereas the energy for OSL emission at room temperature is 3.4 eV (Huntley *et al.*, 1991). A similar temperature shift in the emission spectrum of  $Mn^{2+}$  in calcite has been described by Townsend *et al.* (1994).

This temperature dependence is not surprising. It follows from changes in the crystal field with temperature and is described in terms of configuration models for luminescence centres (Marfunin, 1979; White, 1989). However, it appears to be a new observation in the context of thermoluminescence. It could be argued that it is now being observed because of the advent of high sensitivity spectrometers, such as those described in this paper. This, and the earlier discussion of dating specifically, also underline the thesis of Townsend and Kirsh (1989) that use of any mineral sample for TL dating should be preceded by a 3-D TL study.

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