Pergamon

Radiation Measurements Vol. 27, No. 5/6, pp. 749–768, 1997

Printed in Great Britain
PII: S1350-4487(97)00206-0
1350-4487/98 $19.00 + 0.00

LUMINESCENCE TECHNIQUES: INSTRUMENTATION AND METHODS

LARS BØTTER-JENSEN*
Risø National Laboratory, DK 4000 Roskilde, Denmark

Abstract—This paper describes techniques, instruments and methods used in luminescence dating and environmental dosimetry in many laboratories around the world. These techniques are based on two phenomena – thermally stimulated luminescence and optically stimulated luminescence. The most commonly used luminescence stimulation and detection techniques are reviewed and information is given on recent developments in instrument design and on the state of the art in luminescence measurements and analysis. © 1998 Elsevier Science Ltd. All rights reserved

1. INTRODUCTION

Luminescence arises from stimulation, either thermal or optical, of minerals that have been previously exposed to ionising radiation. During exposure, radiation energy is accumulated and stored in the crystal lattice; this energy is stored in the form of electrons that have been trapped at defects in the lattice. During stimulation, the trapped charge is released and as a result the luminescence signal becomes zero. Radiation-induced luminescence should be distinguished from other luminescence phenomena, e.g. photoluminescence, phosphorescence, etc. which are not dose dependent and thus not relevant to dating or dosimetry.

Thermally stimulated luminesence, usually called thermoluminescence (TL), has been used extensively since the early 1950s to measure nuclear radiation doses (Daniels *et al*., 1953), following the commercial availability of sufficiently sensitive and reliable photomultiplier (PM) tubes. TL was subsequently applied to archaeological dating in the early 1960s (e.g. Aitken *et al*., 1964, 1968a; Mejdahl, 1969) and to geological dating in the early 1980s (e.g. Wintle and Huntley, 1980).

Optically stimulated luminescence (OSL) was introduced for dating by Huntley *et al*. (1985), who selected the 514 nm line from an argon laser to stimulate luminescence from quartz. This technique was subsequently taken up by other laboratories using both quartz and feldspar, and a variety of stimulation light sources (Hütt *et al*., 1988; Aitken and Smith, 1988; Spooner and Questiaux, 1990; Poolton and Bailiff, 1989; Bøtter-Jensen *et al*., 1991; Bøtter-Jensen and Duller, 1992). An immediate advantage of OSL over TL is that it is normally measured at or close to room temperature and is thus a less destructive method. OSL also measures only the component of the trapped electron population that is most sensitive to light. In geological dating, this is important because this component is most likely to be emptied (or "reset") during transport prior to deposition and burial.

More recently, luminescence techniques similar to those used in dating have been adopted for retrospective dose assessment, i.e. reconstruction of radiation doses received by the general population after nuclear accidents. Typically, radiation doses are determined from TL or OSL measurements carried out on quartz and feldspar samples extracted from bricks, tiles, pottery or porcelain items collected in nuclear accident areas such as Chernobyl (e.g. Godfrey-Smith and Haskell, 1993; Bailiff, 1995; Bøtter-Jensen *et al*., 1996).

In the following sections these different luminescence dating and dosimetry techniques and methods are described and information is provided on recent achievements in instrument development and in luminescence detection and analysis.

2. THE PM TUBE, ELECTRONICS AND SAMPLES

Both TL and OSL are normally detected using a photomultiplier tube which, after 40 years, still constitutes the vital component in a luminescence measurement system. The photomultiplier is a vacuum tube that includes a photosensitive cathode, a number of electron multiplying dynodes and an anode normally held at about 1000 V. Light photons interact with the photoelectric cathode material (e.g. potassium–caesium), causing the emission of electrons which are then attracted to the positive voltage of the first dynode. Depending on the dynode material (e.g. antimony–caesium), two or

*To whom all correspondence should be addressed.

three electrons are then emitted for each electron striking it. These electrons are again attracted by the next dynode, and so on, resulting in several million electrons reaching the anode for each electron emitted from the cathode. Thus a light photon reaching the photocathode is converted to an electrical pulse at the anode. However, not all photons are converted to pulses and, additionally, the photomultiplier is not equally sensitive to photons emitted at different wavelengths. This results in a quantum efficiency of up to 25%, depending on the wavelength. Typically, a bialkali PM tube, such as EMI 9235, has a selective response curve with a maximum detection efficiency peaking around 400 nm, which is suitable for the luminescence emission spectra from both quartz and feldspars. Other types of PM tubes, such as EMI 9658 and RCA 31034, are available with an extended sensitivity in the red region (S-20 cathode) which is particularly suitable for the investigation of the red-emission from some feldspar types (e.g. Visocekas, 1993). S-20 cathode PM tubes normally need cooling to reduce the dark noise, using commercially available Peltier-element coolers. The quantum efficiency versus photon energy or wavelength is shown for bialkali and S-20 PM tubes in Fig. 1.

In principle, the PM tube can be operated in two modes. One method is based on smoothing the pulses arriving at the PM anode and thereby generating a DC current signal that, if amplified and fed to a recorder, is able to directly produce a TL glow curve (see Section 3.1). Digitising the DC signal may be performed using a current-to-pulse rate converter system which allows a wide response range of the order of 7 decades, and the possibility of offsetting the dark current to zero (Shapiro, 1970). However, a more sensitive mode is to directly count the single pulses generated from light photons interacting with the photocathode, and using a fast

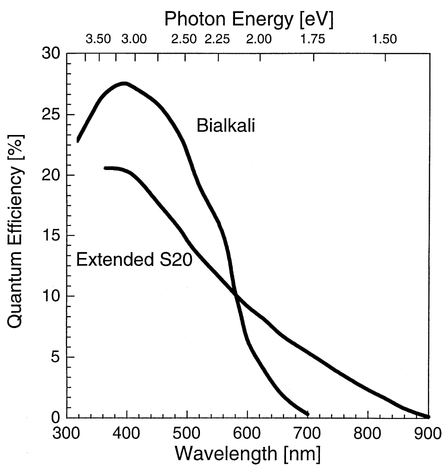

Fig. 1. Quantum efficiency versus photon energy or wavelength for bialkali and S-20 (extended red sensitivity) PM tubes.

pulse amplifier and a pulse height discriminator to feed a ratemeter or scaler (e.g. Aitken *et al.*, 1968b; Aitken, 1985). Modern bialkali PM tubes, such as EMI 9235QA, are now available with a dark count rate of less than 20 cps at room temperature. A further advantage of the single photon counting technique is that the counts accumulated during a measurement can be directly converted into absolute light intensity without knowledge of the PM amplification factor; this facilitates comparison between different systems.

Samples for luminescence measurements are typically prepared either as multiple mineral fine grains (< 10 microns) or pure mineral coarse grains (> 100 microns) on standardised 0.5-mm thick steel or aluminium discs of diameter 10 mm. Alternatively, samples can be prepared in 10-mm depressed cups made of 0.1-mm thick nickel or platinum foils. During TL and OSL measurements the discs or cups are placed on a heater element plate or lifted into a focused stimulation light beam, respectively.

3. THERMALLY STIMULATED LUMINESCENCE

3.1. *Glow curves*

Thermally stimulated luminescence, or thermoluminescence (TL), is observed by heating a sample at a constant rate to about 500°C and recording the luminescence emitted as a function of temperature. A schematic diagram of a TL reader is shown in Fig. 2. The TL signal is characterised by a so-called "glow curve", with distinct peaks occurring at different temperatures, which relate to the electron traps present in the sample. Defects in the lattice structure are responsible for these traps. A typical defect may be created by the dislocation of a negative ion, providing a negative ion vacancy that acts as an electron trap. Once trapped, an electron will eventually be evicted by thermal vibrations of the lattice. As the temperature is raised these vibrations become stronger, and the probability of eviction increases so rapidly that within a narrow temperature range trapped electrons are quickly liberated. Some electrons then give rise to radiative recombinations with trapped "holes", resulting in emission of light (TL). The lifetime for trapped electrons varies, depending on the depth of the trap; low-temperature traps (shallow traps) are thermally drained more quickly at room temperature than deep traps. A typical glow curve obtained from a sedimentary K-feldspar is shown in Fig. 3. The temperature peaks corresponding to different electron traps can be clearly seen, and the lower curve is the black-body radiation signal observed when the sample is heated a second time with no additional radiation.

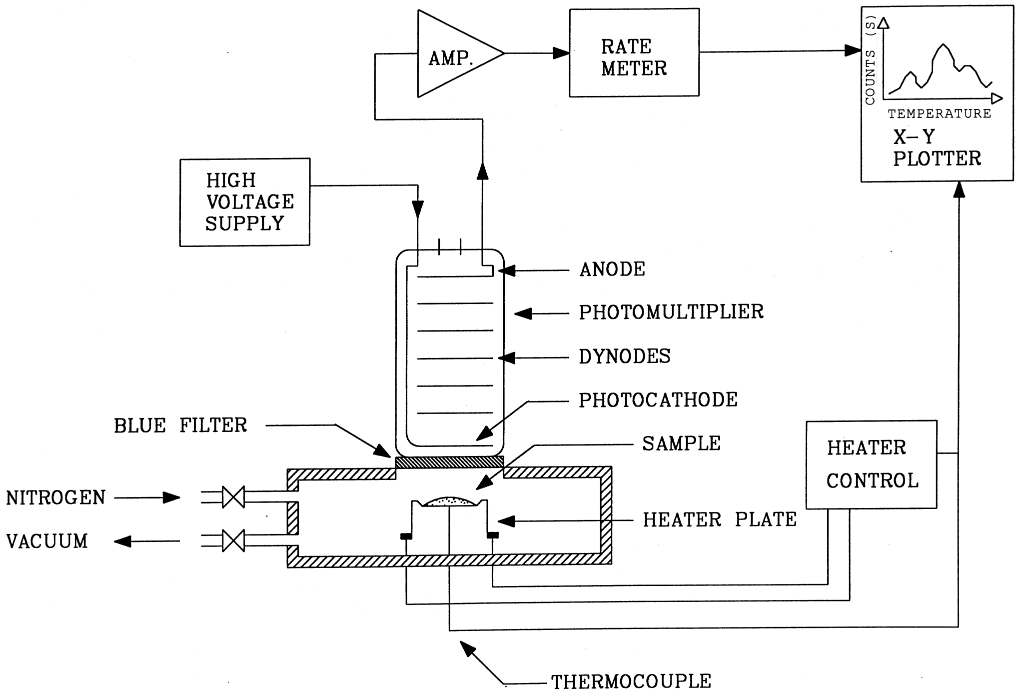

Fig. 2. Schematic diagram of a TL reader system.

3.2. *Heating systems*

In both TL dating and retrospective dosimetry using natural materials, it is important to heat samples at a constant rate in order to get a temperature-resolved glow curve for identification of peak temperatures (electron traps). Linear heating is normally performed using a low-mass heater strip made of high resistance alloys (e.g. nickel and Kanthal) and feeding a controlled current through the heating element. Feedback control of the temperature is achieved using a thermocouple (e.g. Cr/Al) welded to the heater strip (see Fig. 2). Normally, heating is controlled by an electronic ramp that can generate various preheat functions and linear heating rates (e.g. 0.1–30°C/s). The maximum temperature normally used for quartz and feldspar dating is 500°C, but for special investigations of deep trap effects, temperatures up to 700°C have been used (e.g. Valladas and Gillot, 1978).

Other heating systems are used for readout of conventional solid TL dosemeters in radiation protection. The dosemeters may be lifted into a stream of hot nitrogen (300–400°C) and the TL signal released during the resulting non-linear heating (e.g. Bøtter-Jensen, 1978). A CO_2 laser beam has also been used for the non-linear heating of solid TL dosemeters (Bräunlich *et al.*, 1981).

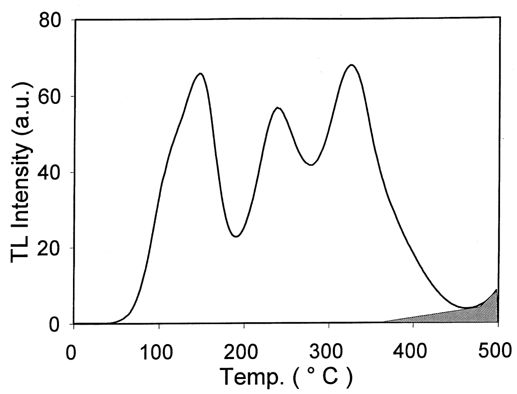

Fig. 3. Typical TL glow-curve from a sedimentary K-feldspar sample given a beta dose of 8 Gy in addition to the natural dose (approximately 200 Gy). The 150°C peak evident in this figure has been created by the recent beta dose; it is not usually evident in the natural signal as it has normally decayed away. The shaded area is the black-body radiation observed when the sample is heated a second time with no additional irradiation.

3.3. *Optical filters*

A limiting factor in TL measurement is the thermal background signal arising from the heating element and sample during heating to high temperatures (black-body radiation). In order to distinguish low TL signals it is important to use blue filters in combination with heat-absorbing filters to suppress the thermal background signal. Typical blue filters used in TL dating routines are Corning 7-59 and Schott UG-11 filters, and an efficient heat-absorbing filter is Pilkington HA-3. The transmission characteristics of these filters are shown in Fig. 4.

It should be noted that the Schott UG-11 filter has a near-infrared transmission window, which is the reason it cannot be used alone for either TL or infrared stimulated luminescence. An additional filter is needed with characteristics to suppress the breakthrough, e.g. Schott BG-38 or BG-39 filters.

3.4. *TL stability*

Although a TL glow curve may look like a smooth continuum, it is composed of a number of overlapping peaks derived from the thermal release of electrons from traps of different stabilities. The lifetime of electrons in deep traps is longer than that of electrons in shallow traps. Normally traps giving rise to glow peaks lower than 200°C are no use for dating, as electrons can be drained from these traps over a prolonged time even at environmental temperatures. Stable glow peaks suitable for dating usually occur at 300°C or higher. However, anomalous (i.e. unexpected) fading of high-temperature glow peaks at room temperature has been observed in some feldspars. This is explained as a quantum mechanical tunnelling effect (Wintle, 1973). Templer (1985) described a model which allows charge recombination to occur by transitions through an excited state common to a trap and luminescence centre pair. Anomalous fading can introduce severe discrepancies in dating if not taken into account (e.g. Mejdahl, 1990).

Strickertsson (1985) investigated the TL stability of potassium feldspars by determining trapping parameters by initial rise measurements, using the fractional glow technique. The mean lifetimes were calculated, assuming first-order kinetics, and it was concluded that only the high-temperature peaks at 299, 384 and 470°C were stable and suitable for dating and dosimetry.

Another source of apparent TL instability is thermal quenching. Some high-temperature peaks in quartz and feldspars are subject to thermal quenching processes, i.e. the increased probability of non-radiative recombination at higher temperatures (Wintle, 1975). If this effect is not taken into account, trap depth analysis may suggest that the peak is unsuitable for dose assessment, despite what is found in practice (Poolton *et al.*, 1995).

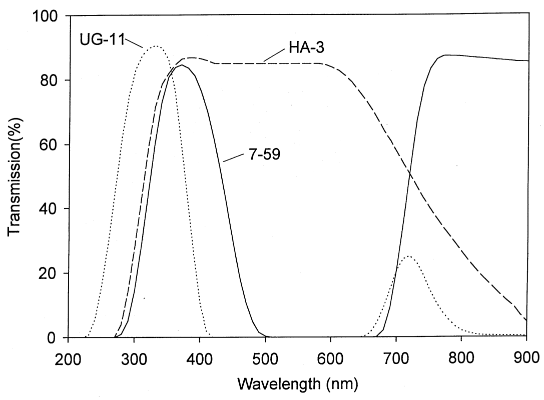

Fig. 4. Transmission characteristics of Corning 7-59, Schott UG-11 and Pilkington HA-3 filters.

4. OPTICALLY STIMULATED LUMINESCENCE

Optically stimulated luminescence (OSL) arises from the recombination of charge which has been optically released from electron traps within the crystal. These traps may be the same as those associated with the TL peaks. The population of the traps is the result of irradiation of the material, and thus the OSL intensity is related to the absorbed radiation dose. For experimental convenience OSL emitted during recombination of the detrapped charges is usually measured in a spectral region different from that of the exciting photons. During exposure to the stimulation light the OSL signal is observed to decrease to a low level as the trapped charge is depleted (decay curve). The physical principles of OSL are thus closely related to those associated with TL.

The potential of OSL in dating applications was first identified by Huntley *et al.* (1985), who used the green light from an argon laser (514 nm) to stimulate luminescence from quartz for dating sediments. Later studies characterised the OSL properties of quartz in more detail with a view to establishing the technique as a tool for dating and dosimetry (e.g. Aitken, 1990; Godfrey-Smith *et al.*, 1988; Rhodes, 1988). Hütt *et al.* (1988) discovered that infrared light (IR) could also be used for stimulation of luminescence in feldspars and subsequently Poolton and Bailiff (1989), Spooner *et al.* (1990) and Bøtter-Jensen *et al.* (1991) constructed units for stimulation based on systems of small IR light emitting diodes (LEDs). Broad-band emitters such as incandescent or arc lamps, in conjunction with selected filters, have also been used to produce both infrared and visible light stimulated luminescence from feldspar and quartz samples (e.g. Hütt and Jaek, 1989; Spooner and Questiaux, 1990; Bøtter-Jensen and Duller, 1992; Pierson *et al.*, 1994).

4.1. *Continuous wave OSL*

In the initial studies of quartz, the use of green light (514.5 nm) from an argon laser operated in continuous wave (CW) mode demonstrated that the energy of visible light is sufficient to empty the OSL electron traps directly in this material. Longer wavelength light is increasingly inefficient at stimulating OSL in quartz (e.g. Aitken, 1990; Bøtter-

Jensen *et al.*, 1994a). In contrast, luminescence can be excited in feldspars with wavelengths in the near infrared, because of one or more excitation resonances in this material. This has been explained in terms of a two-step thermo-optical process (Hütt *et al.*, 1988) where charge is promoted from the ground state of the defect to a series of metastable excited states. This difference in stimulation characteristics can be made use of in various ways, e.g. for testing the purity of quartz samples and for measurements of mixed samples (e.g. Spooner and Questiaux, 1990; Bøtter-Jensen and Duller, 1992).

Thus the two main stimulation methods currently being used in routine OSL dating are: (i) infrared stimulated luminescence (IRSL), which is useful only with feldspars, and (ii) green light stimulated luminescence (GLSL), which works with both feldspars and quartz. GLSL is also effective with ceramics (porcelain) and some synthetic materials such as Al_2O_3:C (Bøtter-Jensen and McKeever, 1996; Bøtter-Jensen *et al.*, 1997a).

In both IRSL and GLSL it is vital to avoid the excitation light source affecting the PM tube. This is achieved by a combination of suitable optical stimulation and detection filters.

4.1.1. *Continuous wave (CW) IRSL*. In CW IRSL it is comparatively easy to separate the stimulation wavelengths (typically centered around 850 nm) from the luminescence emission of feldspars (380–420 nm). As the IR light emitted from an IR light emitting diode is a narrow band (e.g. for TEMPT 484 LED: 880 ▲ 80 nm) it is only a matter of protecting the PM tube with a detection filter with high attenuation in the infrared range and a high transmission in the visible range. A widely used filter is a Schott BG-39 which is a blue-green transmission filter with excellent characteristics for IRSL measurements. A schematic of a typical IRSL configuration is shown in Fig. 5 and the BG-39 filter and IR diode characteristics are shown in Fig. 6.

4.1.2. *Continuous wave (CW) GLSL*. CW stimulation using visible light requires carefully selected filter combinations to prevent the stimulation light from interfering with the luminescence emission. It has been shown that there is an exponential relationship between both bleachability and OSL efficiency of quartz and the energy of the stimulation light, i.e. the shorter the wavelength of the excitation light the smaller the number of photons needed for stimulation (Spooner *et al.*, 1988; Spooner, 1994; Bøtter-Jensen *et al.*, 1994a). Therefore, light with a green spectrum extending into the blue is normally chosen for stimulation of quartz, and the single green line (514.5 nm) from an argon laser can be used directly. Excitation of quartz using green light emitting diodes with a peak emission at 565 nm has also been investigated (Galloway, 1993, 1994) but the maximum power delivered to a sample so far obtained is too low to allow detection of the weak OSL signals from young or insensitive samples. However, brighter green and blue LEDs have recently become commercially available and they are now being tested (see Sections 5 and 6). A sufficient excitation intensity can be achieved by using filtered wavelength bands from incandescent halogen or arc xenon lamps (Spooner and Questiaux, 1990; Bøtter-Jensen and Duller, 1992). The relative attenuation between the stimulation light band and the PM response must be of the order of 10^{-15} to suppress sufficiently the scattered light from the excitation source. This is achieved using interference filters on the excitation side, and detection filters with a selective transmission in the UV

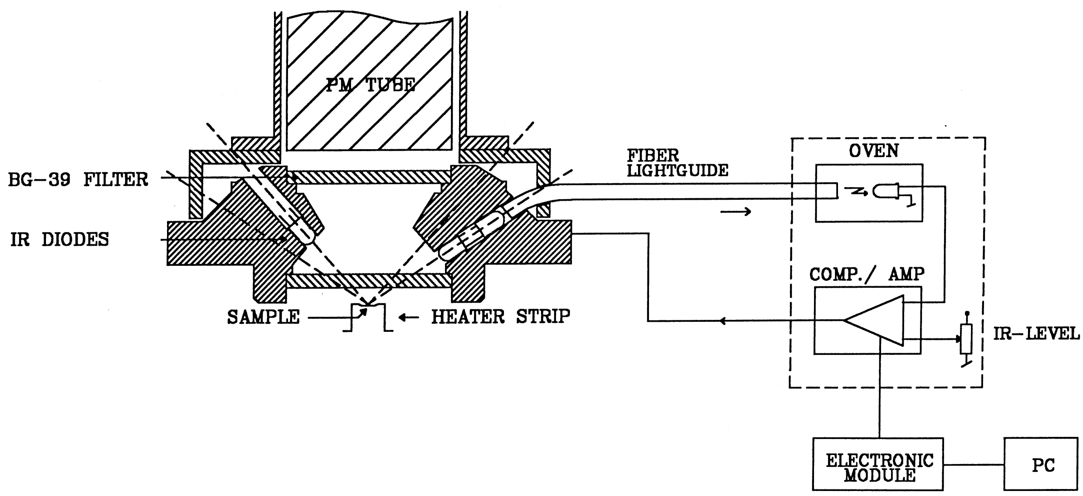

Fig. 5. Schematic diagram of an IRSL unit attachable to the automatic Risø TL apparatus. Thirty-two IR LEDs are arranged in two concentric rings focusing on the sample. A feedback system for controlling the LED current is also shown (from Bøtter-Jensen *et al.*, 1991).

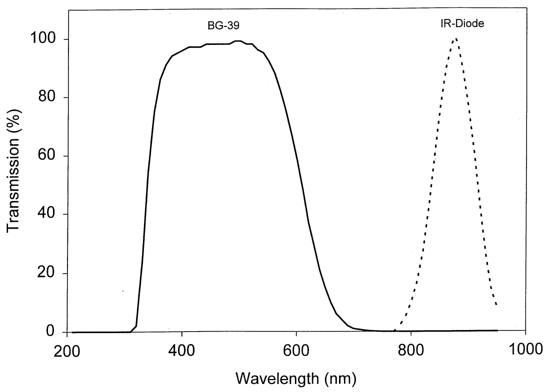

Fig. 6. Characteristics for Schott BG-39 filter and IR LED type TEMPT 484.

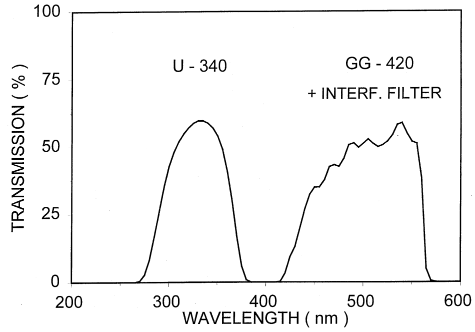

Fig. 8. Typical GLSL excitation band (420–550 nm) and detection filter characteristics. The detection filter is a 5 mm Hoya U-340. The stimulation band is generated by a 75 W halogen lamp filtered by a short-wave-pass filter (heat reflection), a short-wave-pass interference filter in combination with a 6 mm Schott GG-420 long-wave-pass filter (from Bøtter-Jensen and Duller, 1992).

range. A commonly used detection filter for GLSL using broad band excitation is a Hoya U-340 with peak transmission around 340 nm. A GLSL configuration using a halogen lamp as the excitation light source is shown schematically in Fig. 7 and typical excitation wavelength band and detection filter characteristics are shown in Fig. 8. Figure 9 shows a typical OSL decay curve obtained from a sedimentary quartz sample using a green wavelength band of 420–550 nm producing 16 mW/cm^2 at the sample.

Duller and Bøtter-Jensen (1996) showed that exposure of quartz to 514 nm light, such as is produced by an argon-ion laser, causes a similar loss of OSL signal as measured at stimulation wavelengths from 420 to 575 nm when detection is made with a Hoya U-340 filter and that over this range of stimulation wavelengths, the OSL signals produced behave in a similar way. Murray and Wintle (1997) concluded that, on the basis of their measurement of the thermal assistance energy for quartz OSL, the effective stimulating wavelength of this broad band wavelength range (420–550 nm) is 468 nm. Duller and Bøtter-Jensen's study suggests that over the range 420–575 nm, a similar set of traps and charge transport are being used to produce OSL. It also suggests that similar phenomena should be observed whether an argon-ion laser or broad band

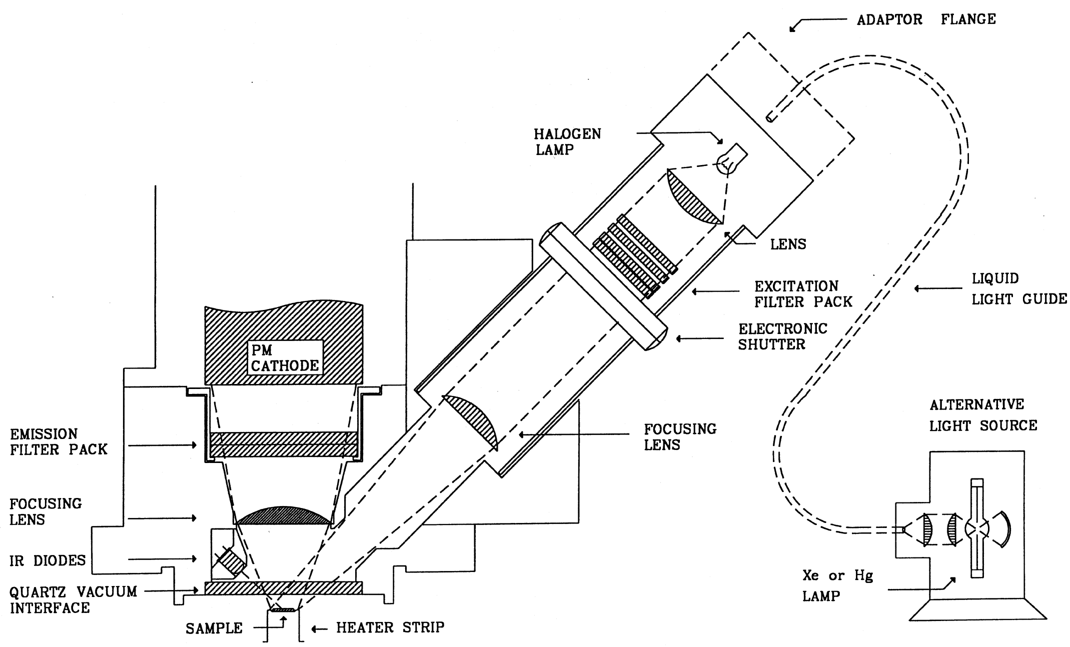

Fig. 7. Schematic diagram of a combined IRSL/GLSL unit attachable to the automatic Risø TL apparatus. Green light stimulation is produced using filtered light from a halogen lamp and IR stimulation is produced using IR LEDs (from Bøtter-Jensen and Duller, 1992).

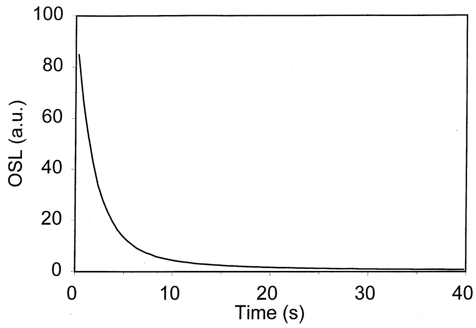

Fig. 9. Typical OSL decay curve from a sedimentary quartz sample given a beta dose of 2 Gy obtained using a green light wavelength band of 420–550 nm producing 16 mW/cm^2 at the sample position.

stimulation (420–550 nm) is used for studies of the OSL from quartz. However, Rees-Jones *et al.* (1997) recently reported differences between OSL signals from a particular quartz sample using a narrow wavelength band compared with using a wide wavelength band for stimulation.

4.2. *Pulsed OSL*

In the applications discussed so far, the light from the excitation sources – either lasers, diodes or filtered lamps – is emitted continuously and the luminescence is monitored during the period that the sample is exposed to the stimulation source. As discussed, this requires the use of filters to discriminate between the stimulation light and the emitted light, and this prevents the use of stimulation wavelengths which are the same as, or close to, those observed in the emission. More recently, a pulsed stimulation technique has been reported, in which the stimulation source is pulsed and the OSL is only monitored after the end of each pulse, i.e. only the afterglow is measured (McKeever *et al.*, 1996). Since the emission is not detected while the pulse is on, this arrangement extends the potential range of stimulation wavelength. A timing diagram for a POSL measurement is shown in Fig. 10.

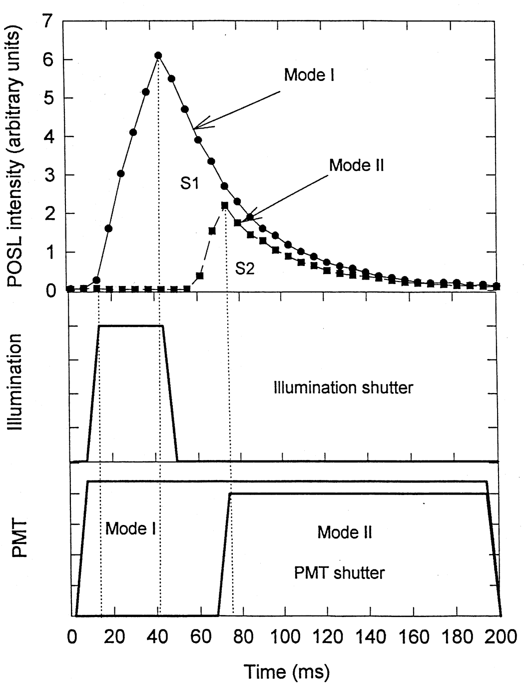

Fig. 10. Timing diagram for POSL measurements illustrating two modes of operation. In "Mode I" the POSL signal is monitored during and after the pulse illumination. To separate the stimulation light from the emission light two 420-nm interference filters are used in front of the PM tube. In "Mode II" the PM tube is closed during illumination and data acquisition is initiated 20 ms after closure of the shutter (from McKeever *et al.*, 1996).

5. THE DEVELOPMENT OF LUMINESCENCE APPARATUS

In the early 1960s manually-operated TL systems were designed mainly for basic studies of TL properties of synthetic dosimetric phosphors and natural materials such as quartz and feldspars. At a later stage automation was identified as a necessary tool to increase the capacity for routine measurement. When OSL techniques were introduced in the late 1980s, studies of OSL properties of natural materials were undertaken and many new OSL methods using different stimulation light sources were reported.

5.1. *TL apparatus*

In the 1960s, commercially available instruments (e.g. Harshaw and Eberline) could heat samples only non-linearly up to a maximum temperature of 350–400°C. TL measurements in dating routines require heating of samples to at least 500°C, and so those involved in dating had to build their own experimental readers; this early work has resulted in a variety of experimental configurations.

5.1.1. *Manually operated TL dating systems.* The main source of inspiration for the construction of TL apparatus for dating is undoubtedly the initial Oxford design for a manual TL reader (Aitken *et al.*, 1968a,b). This was later adopted as a model for the design of TL readers at several dating laboratories. The first Oxford TL system consisted of a heater strip contained in a vacuum chamber, a manually removable PM tube assembly, and electronics for converting the PM signal to glow curves on a recorder. It was discovered at an early stage that the main requirement for avoiding spurious (i.e. non-dose-dependent) signals, especially in fine grain TL measurements, included (i) evacuation of air (especially oxygen) from the sample chamber before readout, and (ii) after evacuation, filling the chamber with nitrogen before heating. The atmosphere was controlled using a vacuum gauge and manual valves for vacuum and nitrogen. The Oxford concept was later taken up and modified to meet special requirements e.g. by Unfried and Vana (1982) who built a system based on photon counting and heating samples up to 500°C in any atmosphere. Visocekas (1979) and Huntley *et al.* (1988) constructed their own manually operated experimental TL readers which were used to study TL at low and constant temperatures (isothermal decay) and TL emission spectra, respectively. Vana *et al.* (1988) developed a manual TL dating system that allowed heating up to 700°C in any atmosphere and collection of measurements on a personal computer. Brou and Valladas (1975) constructed a special high temperature TL glow-oven with cooled heater terminals which allowed for heating up to 800°C. This was used to study the high temperature peaks of volcanic materials (Valladas and Gillot, 1978). Parallel to the development work carried out in different laboratories the Daybreak and Littlemore companies introduced commercially available manually-operated TL systems based on a glow-oven for single measurements and photon counting techniques, specifically intended for dating applications.

5.1.2. *Automatic TL dating apparatus.* In the late 1960s the demand on TL dating laboratories to routinely carry out a large number of measurements accentuated the need for equipment with automatic changing of samples. An automatic TL reader, using a planchette sample changer capable of measuring 12 samples in sequence, was first developed at Risø (Bøtter-Jensen and Bechmann, 1968). With the establishment of the Nordic Laboratory for TL Dating at Risø in 1977, microprocessor and PC-controlled 24-sample automatic TL readers were developed for routine dating of a large number of samples (Bøtter-Jensen and Bundgaard, 1978; Bøtter-Jensen and Mejdahl, 1980; Bøtter-Jensen *et al.*, 1983). Bøtter-Jensen (1988) described an automatic TL system made up of a software-controlled 24-sample glow-oven/sample changer, and one or two beta irradiators, all contained in a vacuum chamber. The automated Risø TL reader (model TL-DA-8) first became commercially available in 1983 and some years later the Daybreak and Littlemore companies constructed 20-sample and 24-sample automatic TL readers, respectively, building on the concept of the initial Risø design (see Section 6). Bailiff and Younger (1988) built a 24-sample microprocessor-based semi-automatic TL apparatus, designed mainly for research, that incorporated an on-plate beta irradiator and automatic control of vacuum and nitrogen atmospheres. At a later stage Galloway (1991) produced a 40-sample system and Henzinger *et al.* (1994) reported a fully automated 60-sample automatic TL reader system developed at Atominstitut der Österreichischen Universität, Vienna. In addition to the sample changer, this system incorporated a beta irradiator position, an alpha irradiator position, a preheat position and a TL readout position. More recently Valladas *et al.* (1996) reported a simple automatic TL apparatus that can accommodate 16 samples. The turntable of this system pushes the samples in sequence onto a hotplate, and heating is performed without lifting the samples from the turntable.

5.2. *OSL apparatus*

Huntley *et al.* (1985) first showed that 514 nm laser light could be used to measure dose-dependent OSL from quartz. However, the expense of establishing such laser facilities meant that this technique would be available only in a very limited number of laboratories. As a consequence, the observation by Hütt *et al.* (1988) that OSL in feldspars could be stimulated with infrared wavelengths was of importance. This made possible the use of inexpensive and readily available IR light emitting diodes (LEDs) as the stimulation light source. As a result, IRSL rapidly became the most popular dating tool. Green LEDs give orders of magnitude less power than IR LEDs, and so the best alternative to lasers for visible light stimulation was the light spectra obtained from heavy filtered halogen or xenon lamps (e.g. Bøtter-Jensen and Duller, 1992).

In OSL measurements, preheating of samples is normally required to remove charge from shallow traps prior to light stimulation (e.g. Huntley *et al.*, 1996). This can either be done in an oven kept at a selected temperature or for short duration preheat, as part of the measurement cycle in the reader. The rate of decay of OSL, and the degree of bleaching, have also been shown to depend on the sample temperature at which the OSL measurement is carried out. For instance Wintle and Murray (1997) recommend OSL of quartz at 125°C to remove interaction with the 110°C TL peak. Therefore, it is important that OSL apparatus be equipped with a heating facility for both preheating and readout at elevated temperature. Also, since erasure of the OSL signal still leaves most of the TL signal unaffected, it is possible to measure first OSL and then TL on the same sample as suggested by Godfrey-Smith *et al.* (1988) and demonstrated by Bøtter-Jensen and Duller (1992).

5.2.1. *IRSL apparatus.* Poolton and Bailiff (1989), Spooner *et al.* (1990) and Bøtter-Jensen *et al.* (1991) described the use of IR LEDs for IR stimulation of feldspars and obtained very promising results. Bøtter-Jensen *et al.* (1991) constructed an IRSL add-on unit to be mounted directly between the PM tube assembly and the glow-oven of the automated Risø TL apparatus (see Fig. 5). Thirty-two IR LEDs were arranged in two concentric rings. IRSL emitted vertically through the ring of diodes was then measured with the same PM as used for the

TL measurements. A BG-39 detection filter rejected the scattered IR light. The total power delivered to the sample using GaA1/As IR LEDs (TEMPT 484, 880 ▲ 80 nm) was measured as 40 mW/cm^2 at a diode current of 50 mA. A feedback servo system served to stabilise the current through the LEDs (see Fig. 5).

Spooner and Questiaux (1990) used an infrared light spectrum filtered from a xenon lamp for optical stimulation of feldspar samples. The use of an excimer dye laser and an IR diode laser for IRSL dating was described by Hütt and Jaek (1989, 1990).

5.2.2. *GLSL apparatus*. The demand for OSL dating of quartz and an alternative to laser stimulation led to the development of OSL systems based on green light LEDs or green light wavelength bands filtered from incandescent broad band lamps. Galloway (1993, 1994) described initial investigations into the use of green light LEDs for stimulation of quartz and feldspars. The system was based on a ring of 16 green LEDs, type TLMP 7513 with peak emission at 565 nm, illuminating the sample. The relatively small power that could be delivered to the sample and the heavy filtering of the photomultiplier cathode necessary to avoid stray light from the LED emission band resulted in slowly decaying OSL curves that required readout times in the order of 2000 s to give useful signals for dose assessment. However, these initial investigations into green LEDs for OSL dosimetry provided a good basis for investigations of new more powerful green LEDs being continuously developed (see Section 6).

Bøtter-Jensen and Duller (1992) developed a compact green light OSL (GLSL) system based on the light emitted from a simple low-power halogen lamp. This lamp provides a broad band light source from which a suitable stimulation spectrum can be selected using optical filters. The stimulation unit also incorporated a ring of IR LEDs at a short distance from the sample. The GLSL/IRSL unit was designed to be mounted onto the automated Risø TL apparatus, thus providing flexible combined IRSL/GLSL/TL features. A low-power (75 W) tungsten halogen lamp filtered to produce a stimulation wavelength band from 420–550 nm delivered a power of 16 mW/cm^2 to the sample. The OSL signals obtained from quartz were observed to decay at the same rate as that observed using an argon laser (514 nm) delivering 50 mW/cm^2 at the sample, presumably because of the higher energies present in the broad band from the filtered halogen lamp. The principle of the GLSL unit is shown in Fig. 7.

5.3. *Commercially available TL/OSL systems*

Three main distributers of TL/OSL dating equipment are: Daybreak Nuclear and Medical Systems, USA, ELSEC-Littlemore Scientific Engineering Company, UK, and Risø National Laboratory, Denmark.

The Daybreak instrument programme includes a standard 20-sample automatic TL reader (model 1100) using an on-board computer and serial interface to a host computer. The samples are moved by a sweep arm from the sample turntable to the heating/reading position and back. An upgraded model 1150 TL reader is available with a capacity of 57 samples achieved by vertically stacking three 20-sample platters. Various OSL attachments are available based on xenon and halogen lamps. A compact fibre optic illuminator attachment was recently reported by Bortolot (1997) (see Section 6), and a new OSL reader design (without TL facilities) based on 60-sample capacity is under development.

The Littlemore Company has two standard automated luminescence dating instruments available. One is a 24-sample automated TL reader (without OSL attachments) and the other is a 64-sample optical dating system (without TL facilities) which is available with either IR LED stimulation or visible light stimulation using a filtered lamp module. An attachable beta irradiator is provided for the automated TL reader.

Risø National Laboratory provides an automatic combined TL/IRSL/GLSL dating system that can accommodate different sample turntables containing 24, 36 or 48 samples, respectively. The most recent model of OSL accessory is a unit containing IR LEDs in close proximity to the sample, and green light stimulation from long-life (2000 h) high-power (150 W) halogen and xenon lamps and a liquid lightguide to provide high transmission. A close sample-to-detector spacing has resulted in a significantly enhanced OSL sensitivity (see Section 6). A software-controlled beta irradiator attachment for in situ irradiations of samples is also provided. A new sequence software has also significantly extended the flexibility and measurement capabilities.

5.4. *Development of specialised OSL equipment*

5.4.1. *OSL equipment for sediment dating and retrospective accident dosimetry*. Intensive dating of thick sediment deposits can be very time-consuming, and often provides little information that could not be obtained from a few carefully selected samples. Changes in the stratigraphy relating to, for instance, breaks in the deposition history will show up as discontinuities in the apparent radiation dose in the sediment either as a result of different age or different bleaching. As a consequence, it is desirable to be able to rapidly assess the luminescence properties of the sediment at regular intervals down a section, preferably in the field. Poolton *et al.* (1994) described a compact portable computer-controlled OSL apparatus that allows the measurement of infrared OSL of sediments in the field, whether in the form of loose grains or compressed pellets. The unit uses IR LEDs for excitation with bleaching and IRSL regeneration provided by cold gas discharge lamps.

When several tens of metres of sediment core are available for study, it is often difficult to decide exactly where to select material for detailed analysis and age determination. Poolton *et al.* (1996a) described an automatic system for measuring the age-related OSL of split sediment cores. The basis for the design is a core logger system with a conveyer belt allowing optical sensors to be moved along the length of split sediment cores up to a length of 1.7 m. A stepper motor drive ensures constant scan rates and an accuracy in positioning of better than 0.1 mm. The optical sensor consists of a photoexcitation and detection module together with lamps for bleaching and regenerating the OSL. The OSL core scanner can also be used to measure depth dose profiles on small cores drilled out of bricks for retrospective dose determination after nuclear accidents (Bøtter-Jensen *et al.*, 1995). The scanner system uses both IR and green light stimulation and is shown schematically in Fig. 11.

5.4.2. *Detection of irradiated food.* Sanderson *et al.* (1989), Autio and Pinnioja (1990) and Schreiber *et al.* (1993) used TL methods on dust and pebble contaminants in foodstuffs for detection of irradiated food. More recently Sanderson *et al.* (1994, 1995) developed and used what he calls photostimulated luminescence methods (the same as IRSL) to identify irradiated food. A new instrument for rapid screening of irradiated food was developed at Scottish Universities Research Centre (SURRC) based on pulsed infrared stimulation, which is designed to allow direct measurements of OSL signals from mineral contaminants in herbs and spices for screening purposes, without the need for sample preparation or re-irradiation. Samples are introduced directly in petri dishes and the instrument produces a qualitative screening measurement over 15 s. The principle of the technique is to pulse stimulate a sample using IR diodes. The pulsing allows higher current and thus larger illumination power at the sample than is possible using continuous wave (see Section 4.1). The background is measured without illumination between the pulses, while the diodes cool, and is subtracted automatically (Sanderson *et al.*, 1996).

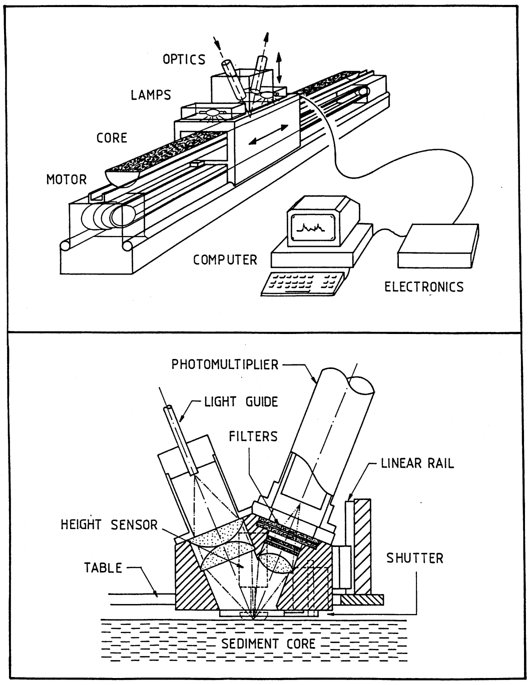

Fig. 11. Schematic of the Risø OSL split core scanner system and detail of the luminescence excitation/detection head. Sediment cores up to 1.7 m in length can be analysed in the system (from Poolton *et al.*, 1996a).

6. OPTIMISATION OF LUMINESCENCE DETECTION

A single luminescent grain emits light in all directions, i.e. in 4π geometry. If the sample is heated or illuminated on a metal support, the maximum light signal is then reduced by at least 50% (to 2π geometry), unless the support for the sample is polished and the sample transparent, etc. Sample-to-PM tube distance is thus very important, since only a small increase will lead to loss of light collected. If greater sample-to-PM tube distance is needed, suitable optics are required to retain the sensitivity of the design. Markey *et al.* (1996) designed and tested OSL attachments to the automated Risø system based on reflecting the luminescence from ellipsoidal mirrors; these provide the greatest flexibility for the incorporation of different excitation sources. By lifting the samples into the focal point of the ellipsoidal mirror, whether thermally or optically stimulated, a gain in sensitivity of 3 to 4 was achieved compared to the standard Risø OSL system. Readout systems based on metallic mirrors are dependent on a stable reflectivity and thus the choice of a pure metal surface such as nickel electroplated with rhodium is of great importance. In the full-reflector system reported by Markey *et al.* (1997) excitation illuminaton is introduced by up to four optional lightguides. A schematic of the full reflector system is shown in Fig. 12.

As a cheaper alternative to the ellipsoidal mirror system a new compact combined IRSL/GLSL unit with a much improved sample-to-PM tube distance has been developed. A significantly enhanced GLSL sensitivity is achieved by using an 8-mm diameter liquid lightguide system with high transmission (98% over 380–550 nm) for illumination of the sample. Filtered wavelength bands are provided using either a 150 W tungsten halogen lamp (life-

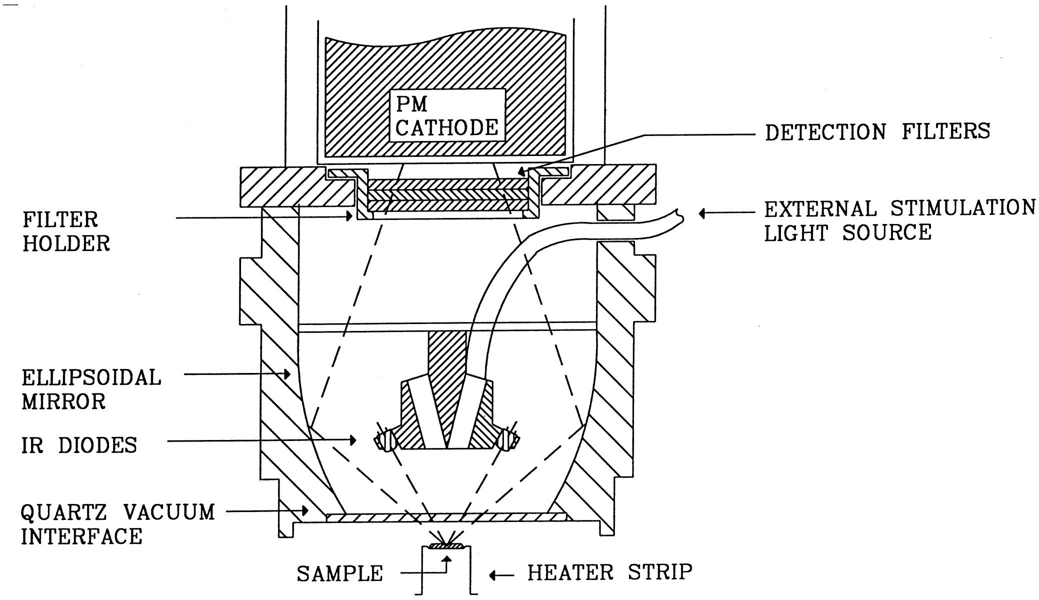

Fig. 12. Schematic of the Risø full reflector OSL system (from Markey *et al*., 1996).

time 2000 h) or a 150 W xenon lamp mounted in a remote lamphouse equipped with electronic shutter and exchangeable excitation filter pack. The new liquid lightguide OSL unit uses quartz lenses for defocusing the stimulation light to ensure that it falls uniformly on the sample. The signal-to-noise ratio was further improved by using multi-layer metal oxide coated (ZrO_2/SiO_2) Hoya U-340 detection filters, specially made by DELTA Light and Optics, Denmark, which attenuate the stray light from the transmission window found in the red region of a normal U-340 filter. IRSL is performed using IR LEDs close to the sample. The unit focusses the emitted luminescence onto the photocathode using a quartz lens with short focal length. A schematic diagram of the combined IRSL/GLSL unit is shown in Fig. 13.

Bortolot (1997) introduced a compact OSL unit based on multiple bundle fibre optics (see Fig. 14). An improved sample-to-PM tube distance is obtained by splitting the fibre bundle into two ends with opposed rectangular light bars close to the sample. The unit also incorporates two IR LED bars and can be mounted between the top lid and

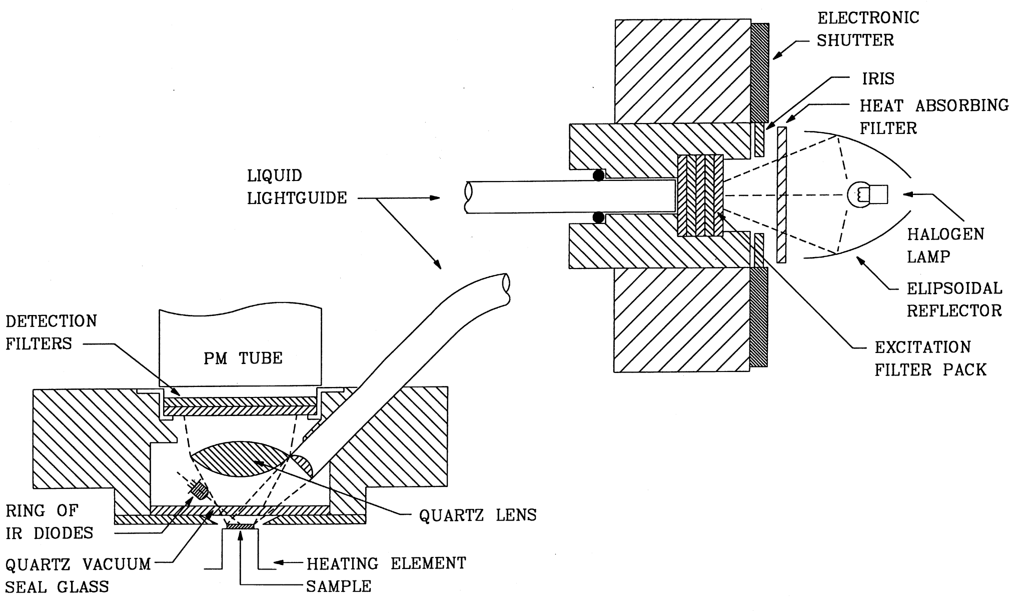

Fig. 13. Schematic of the new compact Risø liquid lightguide-based combined IRSL/GLSL stimulation unit attachable to the automatic Risø TL reader.

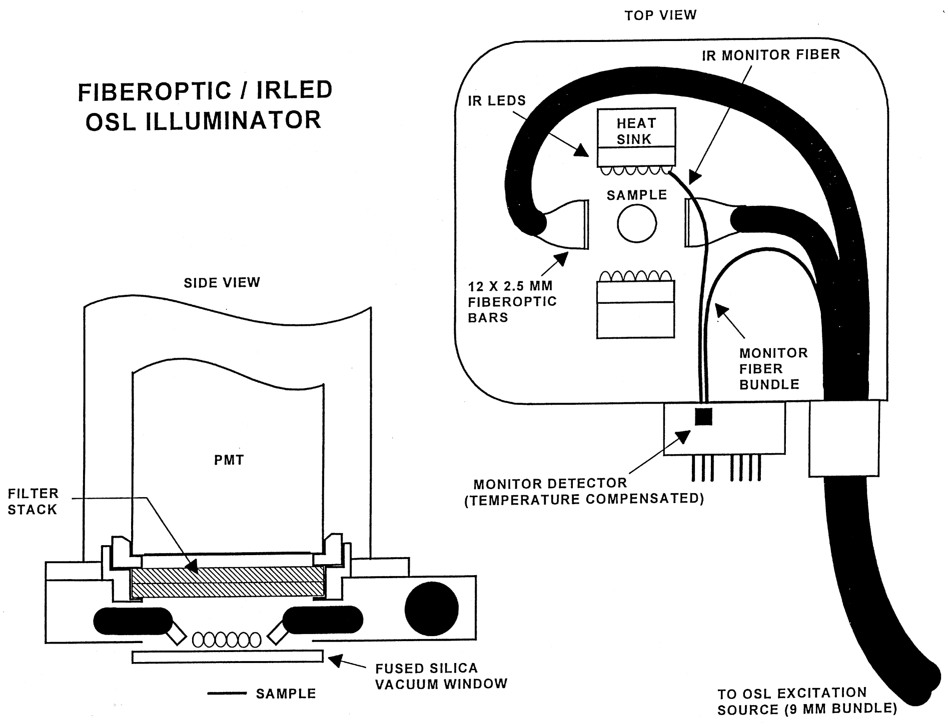

Fig. 14. Schematic of the Daybreak combined fibre optic/IRLED OSL illuminator (from Bortolot, 1997).

PMT housing of the Daybreak 1100 system. Galloway *et al.* (1997) reported the testing of a new type of green LED with enhanced brightness. They further investigated the use of detection filters consisting only of Schott UG-11 filters that were coated with metal oxide on each side (Schott DUG-11). These have the same advantage as described for the coated U-340 filters in the previous section, namely the attenuation of the light from the transmission windows found in the red region of a normal UG-11 filter (see Fig. 4). The enhanced illumination power achieved in combination with the DUG-11 detection filters improved the overall sensitivity by a factor of 1000 compared with their previous green LED system. However, the excitation power achieved using green LEDs is still much below that obtained with filtered lamps and lasers.

Recently, new bright blue LEDs have been tested at Risø for OSL illumination of quartz and porcelain samples (Bøtter-Jensen *et al.*, 1997b). Using a metal oxide coated U-340 detection filter, the emission from the blue LEDs needs to be filtered by a Schott GG-420 cut-off filter in order to avoid the highest energy part of the LED emission wavelength stimulation band interfering with the detection filter window. An increase of OSL efficiency per unit power at the sample of a factor of 5 has been observed using blue LEDs on a variety of quartz and porcelain samples compared to that obtained using green light stimulation. Studies so far have shown that OSL signals from quartz behave similarly, whether stimulation is by blue LEDs or broad band green light. In a comparison of 34 heated and unheated quartz samples, the ratio of the ED from blue stimulation to that from broad band green light was 0.98 ± 0.02 (Bøtter-Jensen *et al.*, 1997b). A prototype of a blue LED OSL attachment to the automated Risø reader is shown in Fig. 15 and decay curves from a sedimentary quartz sample illuminated with both blue LEDs and green light from a filtered halogen lamp are shown in Fig. 16.

There is increasing interest in determining the natural dose in materials using only single aliquots or even single grains of a sample. Single-aliquot procedures in luminescence dating were introduced by Duller (1991) and developed further by Mejdahl and Bøtter-Jensen (1994, 1997), Galloway (1996) and Murray *et al.* (1997). In a true single-aliquot procedure, the dose is measured using only one aliquot; this aliquot is repeatedly irradiated, heated and optically stimulated in an automatic process. It is then important that the sample is not disturbed i.e. it must be kept in the same orientation and not agitated during the entire measurement sequence. Change of the sample geometry during a measurement cycle may, especially in OSL, lead to poor reproducibility because of variations in self-shielding and geometry from one optical stimulation cycle to another (Singhvi, 1996). Therefore, when designing automatic luminescence measurement instruments, attention should be paid to maintaining a constant sample geometry during a full

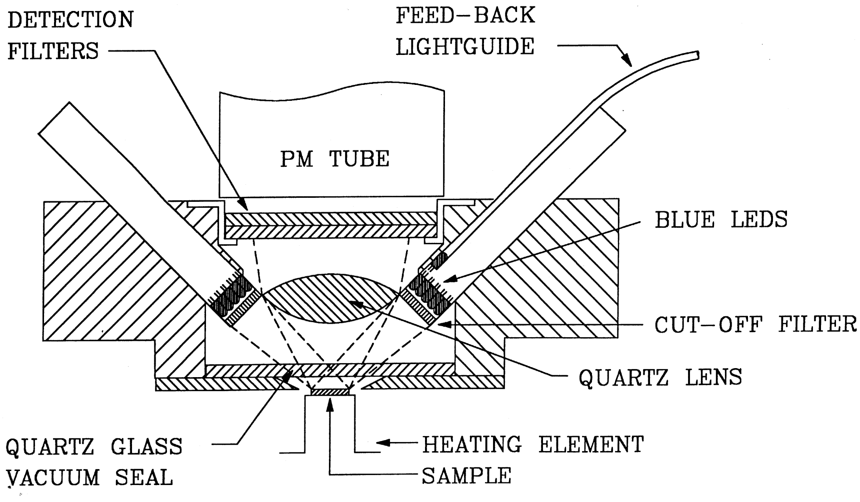

Fig. 15. Schematic of the Risø prototype blue LED OSL attachment.

measurement cycle, e.g. no rotation of the samples as a result of sample changing.

In OSL it is well known that not all grains of a sample emit the same amount of luminescence (Li, 1994; Lamothe *et al.*, 1994; Rhodes and Pownall, 1994; Murray *et al.*, 1995; Murray and Roberts, 1997). Imaging systems (e.g. Duller *et al.* (1997), see Section 8) have shown a large variety of luminescence brightness of the individual grains across a typical sample. This creates interest in the possibility of measuring single grains of samples of which the mineralogy and OSL properties are well known. Templer and Walton (1983) first showed how to map the luminescence from the surface of slices of material and very recently, Murray and Roberts (1997) reported a single-grain optical dating technique that provided an accurate date on a sediment with very heterogeneous composition.

Single-grain dosimetry obviously requires high sensitivity in measurements and it is thus important to design TL/OSL equipment with optimal signal-to-noise ratio (S/N). The S/N is highly dependent on (i) suppression of the dark noise of the PM tube, for example by means of cooling, (ii) the light collection efficiency which is improved either by minimising the sample-to-detector distance or by incorporating suitable optics, (iii) suppression of the black-body radiation in TL and (iv) suppression of stray light from the stimulation light in OSL. The latter points are achieved by using properly selected optical filters.

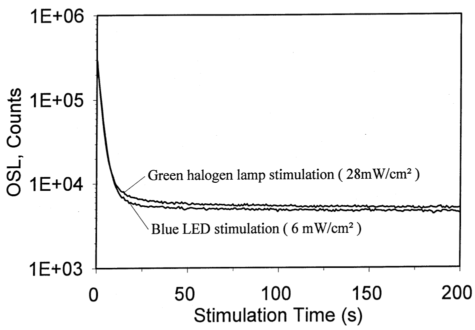

Fig. 16. Decay curves obtained from a sedimentary quartz using stimulation light from an array of blue LEDs and a filtered wavelength band (420–550 nm) from a halogen lamp, respectively.

7. LUMINESCENCE SPECTROMETRY

Ideally, in both TL and OSL applications, the spectral emission and stimulation characteristics of, for example, quartz and feldspar materials prepared for dosimetric evaluation would be routinely measured. As well as giving valuable information about the physical processes involved, it would also allow the possibility of routinely choosing the most suitable emission and stimulation energy windows in which to carry out the measurements.

7.1. *Emission spectrometry*

A simple TL glow curve (TL versus temperature) does not always yield unambiguous information, for instance, when the emission spectrum changes with temperature during a TL measurement. This may be due to the radiative recombination of the released charge occurring at more than one defect site within the crystal. For this reason it is important to be able to obtain 3-D glow curves, i.e. emission spectra in which the intensity is displayed as a function of both temperature and wavelength. 3-D glow curves thus give information both about the trap distribution (TL versus temperature) and the charge recombination centres (TL versus wavelength).

Several instruments based on different optical principles have been developed and described in the literature. Dispersive rapid scanning systems based on diffraction gratings were described in the early 1970s by Harris and Jackson (1970) and Mattern *et al.* (1971). Methods using optical filters have also been employed: Bailiff *et al.* (1977) reported a rapid scanning TL spectrometer based on successive narrow band interference filters of 20 nm bandwidth fixed on a common turntable; Bøtter-Jensen *et al.* (1994b) developed a compact scanning monochromator based on a moveable variable interference filter. Huntley *et al.* (1988) built a spectrometer based on a custom-made concave holographic grating in connection with a microchannel plate PM tube and image converter to obtain wavelength-resolved spectra of a variety of mineral samples. A sensitive spectrometer based on Fourier transform spectroscopy which offers high aperture for light collection and continuous detection at all wavelengths in the range 350–600 nm was developed by Prescott *et al.* (1988). Luff and Townsend (1993) reported a highly sensitive TL spectrometer for producing 3-D isometric plots of TL intensity against wavelength and temperature. This spectrometer, which is shown schematically in Fig. 17, uses two multi-channel detectors that can measure spectra in the wavelength range 200–800 nm. Also Martini *et al.* (1996) developed a high-sensitivity spectrometer for 3-D TL analysis based on wide angle mirror optics, a flat-field holographic grating and a two-stage micro-channel plate detector followed by a 512 photodiode array. Recent developments in charge coupled device (CCD) camera techniques led to the development of emission spectrometers with high resolution. Rieser *et al.* (1994) reported a high sensitivity TL/OSL spectromenter based on a liquid nitrogen cooled CCD camera, with simultaneous detection over the range 200–800 nm. In this instrument thermal stimulation can be performed up to 700°C and optical stimulation from UV to IR with monochromatic light from a 200 W mercury lamp. Krause *et al.* (1997) studied the OSL emission spectra from feldspars obtained by the CCD-based spectrometer and found four wavelength maxima at 280, 330, 410 and 560 nm, respectively.

7.2. *Stimulation spectrometry*

Hütt *et al.* (1988) demonstrated the importance of analysing the optical stimulation spectra (i.e. OSL versus stimulation wavelength) of feldspars and Poolton *et al.* (1996b) showed that stimulation spectra of natural samples provided some information about the mineralogy. As the OSL signal decays under constant illumination, consideration of procedures for correcting the stimulation spectra produced must be considered. Bailiff (1993) and Bailiff and Barnett (1994) used a titanium–sapphire laser, tuneable between 700 and 1000 nm, to analyse the time-decaying OSL stimulation spectra from feldspars, both at room temperature and at low temperatures. A typical stimulation spectrum obtained from Orthoclase feldspar samples using the tuneable laser is shown in Fig. 18. Ditlevsen and Huntley (1994) used argon krypton, He–Ne, and argon-pumped dye lasers operated in CW mode to study optical excitation characteristics of

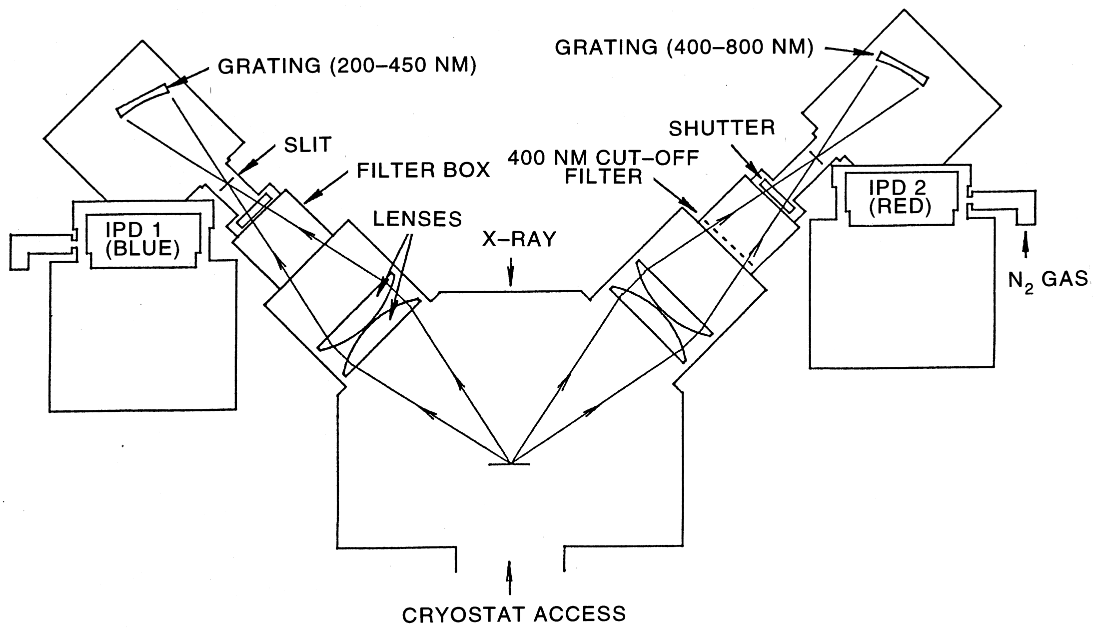

Fig. 17. Schematic of the spectrometer developed at the University of Sussex, showing the sample chamber and the arrangements for the collection optics, spectrometers and detectors (from Luff and Townsend, 1993).

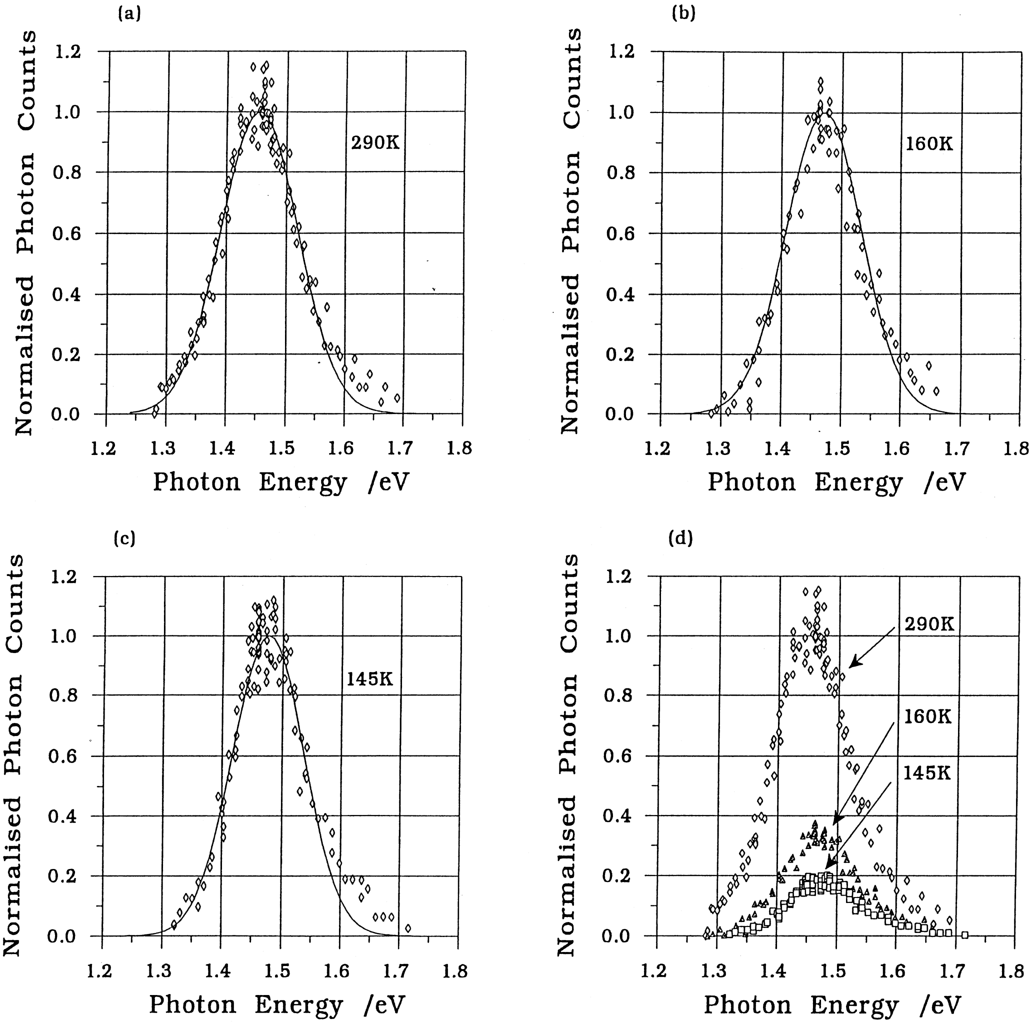

Fig. 18. Optical stimulation spectra from Orthoclase feldspar samples obtained at the University of Durham using a tuneable sapphire laser (from Bailiff and Barnett, 1994).

quartz and feldspars. One problem in using high power tuneable lasers is that the OSL obtained at each wavelength has to be normalised and corrected for the beam power and instrument response. Bailiff and Barnett (1994) observed that the infrared resonance peak position of different feldspars shifted to higher photon energies at lower temperatures and the full-width half-maximum of the peak reduced with decreasing temperature. Clark and Sanderson (1994) performed OSL excitation spectroscopy using filtered light from a 300 W xenon lamp coupled to a computer-controlled, stepper motor driven f 3.4 monochromator. Bøtter-Jensen *et al.* (1994b) designed a compact scanning monochromator based on variable interference filters covering the wavelength band 380–1020 nm. When mounted onto the automatic Risø TL/OSL reader, this enables very rapid scanning of a variety of feldspar and quartz samples (Bøtter-Jensen *et al.*, 1994a). The excitation light source is a low-power (75 W) tungsten–halogen lamp. An optical stimulation spectrum obtained in the wavelength band 420–650 nm (1.9–2.9 eV) from a sedimentary quartz sample using the Risø monochromator is shown in Fig. 19. It may be an advantage in optical stimulation spectrometry to use low-power stimulation light sources in order to lose as little charge as poss-

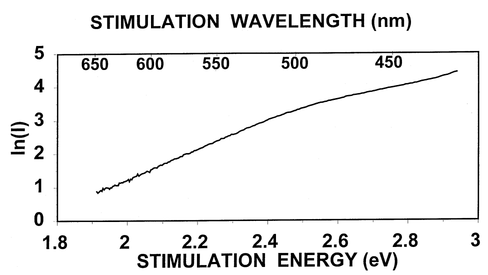

Fig. 19. Optical stimulation spectrum, ln(*I*) versus stimulation energy, for a sedimentary quartz obtained with the Risø IR monochromator attachment (from Bøtter-Jensen *et al.*, 1994a).

ible during OSL readout. Then corrections are needed only for the intensity spectrum of the exciting lamp since the trapped charge evicted during a rapid scan can be reduced to typically 10%.

8. LUMINESCENCE IMAGING

The majority of luminescence measurements are made using PM tubes with bialkali photocathodes. These devices offer high sensitivity in the blue and near ultra-violet. However, the PM tube used for such measurements integrates the luminescence signal from the entire sample and gives no indication of any spatial variation in luminescence intensity within a sample. Duller (1991) initiated the development of a technique for measuring the dose in a single aliquot. The study of luminescence signals even from individual grains is likely to become important, especially for the understanding of sources of scatter from one aliquot to another, to separate mineral-specific luminescence signals from polymineralic samples, and in the development of methods for single grain dosimetry (e.g. Murray and Roberts, 1997). Single grain dosimetry, however, would be far more practical if many grains mounted on the same aliquot could be irradiated, preheated and measured simultaneously and then using an imaging system to separate the luminescence signals from the individual grains. Hashimoto *et al.* (1986) developed techniques for imaging TL signals from sliced rock samples and quartz from beach sands using extremely high-sensitivity colour films. At a later stage Hashimoto *et al.* (1989) and Kawamura and Hashimoto (1995) converted the TL colour images (TLCI) from photographic form into a computer process that made it possible to obtain quantitative information and to distinguish for example between blue and red coloured grains. Hashimoto *et al.* (1995) obtained OSL images of some X- and gamma-irradiated granite slices using photon detection through a 570 nm bandpass filter with diode-laser excitation of 910 nm. Several other laboratories have attempted to develop systems capable of imaging the luminescence signal from a sample. Recently three groups have used imaging photon detectors (IPDs), two at University of Oxford (Smith *et al.*, 1991; McFee and Tite, 1994) and another at University of Utah, Salt Lake City (Berggraaf and Haskell, 1994). These instruments retain the high sensitivity of a PM tube, but are rather expensive and difficult to operate. The development of solid state imaging systems based on charge coupled device (CCD) technology offers an alternative. Duller *et al.* (1997) constructed a CCD camera based imaging system that could be directly attached to the automated Risø TL/OSL reader. The CCD has a similar sensitivity to that of a PM tube, although the spectral responses are very differ-

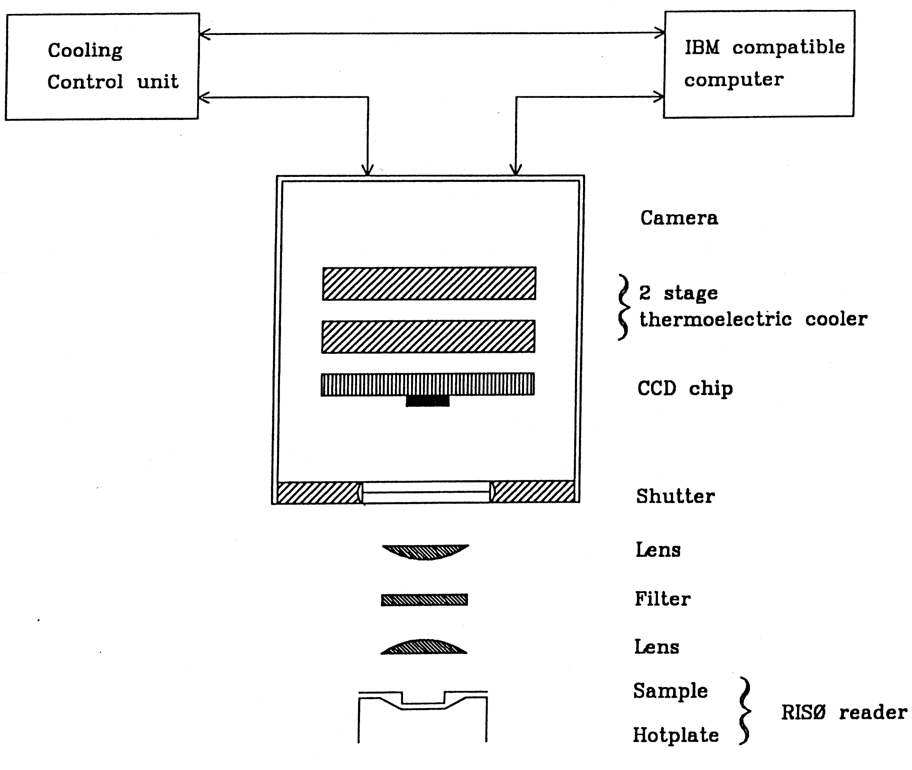

Fig. 20. Schematic diagram showing the components of the CCD system mounted on the Risø automated luminescence reader (from Duller *et al.*, 1997).

ent. This CCD system is capable of detecting natural luminescence signals with a spatial resolution of as high as 17 μm. Temperature-resolved TL signals and time-resolved OSL curves can be obtained using software and the luminescence signals generated within single grains in the bulk sample can be separately analysed. A schematic diagram of the CCD camera is shown in Fig. 20 and Fig. 21 plots IRSL decay curves derived from a CCD image of a feldspar sample.

9. CONCLUSION

Techniques and methods applied in luminescence dating and dosimetry at many laboratories around the world have been reviewed and an attempt has been made to describe the state of the art in instrument and method development.

There is one problem which remains to be addressed in the development of combined TL/OSL instrumentation using different stimulation light spectra. This is concerned with the design of a flexible optical detection filter changing system to allow for rapid (automatic) selection of the optimal detection window whether using infrared or visible light stimulation. Changing of excitation or detection filters may, if not properly protected either by hardware or software, cause serious damage to the PM tube because of insufficient suppression of stray light from the stimulation light source.

The growing industrial interest in ultra bright LEDs as light indicators (e.g. from automobile manufacturers) may soon make visible LEDs commercially available with substantially higher emission power than is available today. These LEDs should provide sufficient power to be considered a real alternative to laser and incandescent lamp stimulation light sources in OSL. The immediate advantages of using LEDs over filtered broad band lamps are: (i) reduced heat dissipation, with less effect on the stimulation optics and (ii) no need for mechanical shutters to control stimulation exposure.

In the future, a major effort will no doubt be put into the development of sensitive systems capable of measuring luminescence from small aliquots, even down to single grains. The immediate advantages of this are that the accrued dose can be determined from only one aliquot and that variations in dose from grain to grain can be studied in detail. The latter feature will be especially valuable in studies of young, incompletely bleached materials and in the identification of sediment disturbance in natural deposits. Such improvements will continue to require increases in detection sensitivity.

Further developments and investigations of luminescence imaging systems for obtaining spatially resolved TL and OSL signals from multi-mineral samples are also foreseen. These systems give rapid and valuable information about the mineralogy of the sample and enable individual analysis of luminescence signals from single grains of a sample. This has the potential to avoid the cumbersome

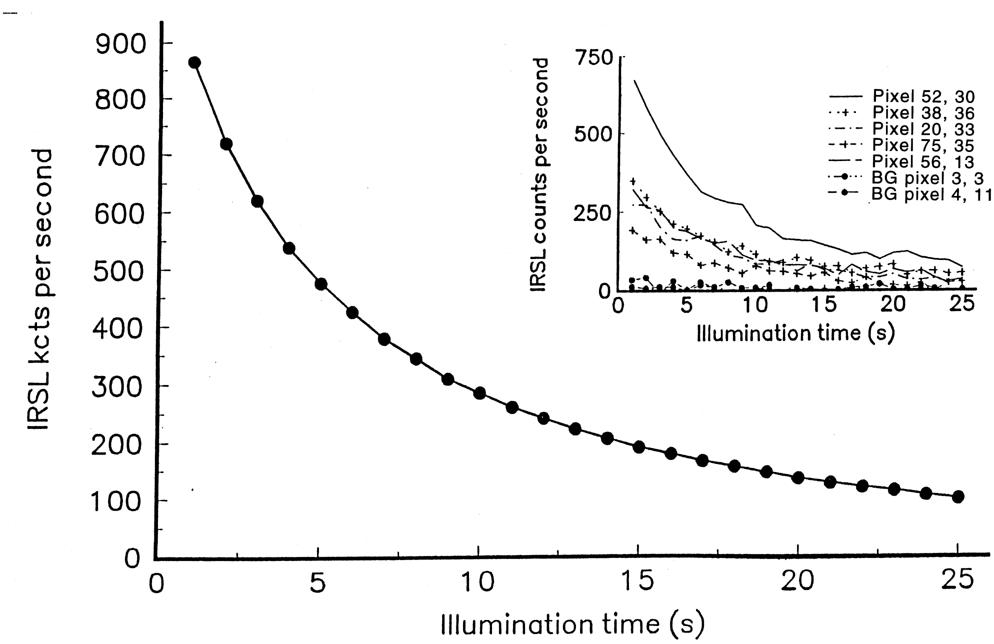

Fig. 21. IR-stimulated luminescence decay curves obtained from a feldspar sample using the the CCD camera. CCD images were integrated for 1 s and 10×10 pixel binning was used, giving spatial resolution of 170×170 μm. The main curve is the signal from the entire CCD, while the insert shows the signal from single pixels (from Duller *et al.*, 1997).

mechanical and chemical separation processes presently required.

Acknowledgements—The author has drawn heavily on publications kindly supplied by a number of authors who have contributed to the development of a wide variety of luminescence techniques and methods. However, having been in the field of developing luminescence instruments and methods for many years, the author of the present paper is inevitably biased towards the work carried out at Risø and I apologise to those who might feel that their work has not been given adequate attention.The author is grateful to Andrew Murray and Vagn Mejdahl for going through the manuscript and for helpful comments and discussions.The development of the new Risø OSL unit described was partly funded by the EU project "Dose Reconstruction".

REFERENCES

Aitken, M. J., Tite, M. S. and Reid, J. (1964) Thermoluminescent dating of ancient ceramics. *Nature* **202,** 1032–1033.

Aitken, M. J., Zimmerman, D. W. and Fleming, S. J. (1968a) Thermoluminescent dating of ancient pottery. *Nature* **219,** 442–444.

Aitken, M. J., Alldred, J. C. and Thompson, J. (1968b) A photon-ratemeter system for low-level thermoluminescence measurements. In: *Proc. 2nd Int. Conf. on Luminescence Dosimetry*, Gatlinburg, CONF-680920, pp. 281–290. U.S. National Bureau of Standards, Washington D.C.

Aitken, M. J. (1985) *Thermoluminescence Dating*. Academic Press, London.

Aitken, M. J. and Smith, B. W. (1988) Optical dating: recuperation after heating. *Quat. Sci. Rev.* **7,** 387–393.

Aitken, M. J. (1990) Optical dating of sediments: Initial results from Oxford. *Archaeometry* **32,** 19–31.

Autio, T. and Pinnioja, S. (1990) Identification of irradiated foods by thermoluminescence of mineral contamination. *Z. Lebens. Unters. Forsch.* **191,** 177–180.

Bailiff, I. K., Morris, D. A. and Aitken, M. J. (1977) A rapid interference spectrometer: Application to low level thermoluminescence emission. *J. Phys. E: Sci. Instrum.* **10,** 1156–1160.

Bailiff, I. K. and Younger, E. J. (1988) Computer-controlled TL apparatus. *Nucl. Tracks Radiat. Meas.* **14,** 171–176.

Bailiff, I. K. (1993) Measurement of the stimulation spectrum (1.2–1.7 eV) for a specimen of potassium feldspar using a solid state laser. *Radiat. Prot. Dosim.* **47,** 649–653.

Bailiff, I. K. and Barnett, S. M. (1994) Characteristics of infrared-stimulated luminescence from a feldspar at low temperatures. *Radiat. Meas.* **23,** 541–545.

Bailiff, I. K. (1995) The use of ceramics for retrospective dosimetry in the Chernobyl exclusion zone. *Radiat. Meas.* **24,** 507–512.

Berggraaf, D. and Haskell, E. H. (1994) A software package for TL/OSL spectrometry and extraction of glow curves from individual grains. *Radiat. Meas.* **23,** 537.

Bortolot, V. J. (1997) Improved OSL excitation with fiberoptics and focused lamps. *Radiat. Meas.* **27,** 101–106.

Bøtter-Jensen, L. and Bechmann, P. (1968) A versatile automatic sample changer for reading of thermoluminescence dosimeters and phosphors. In: *Proc. 2nd Int. Conf. on Luminescence Dosimetry*, Gatlinburg, CONF-680920, pp. 640–649. U.S. National Bureau of Standards, Washington D.C.

Bøtter-Jensen, L. (1978) A single, hot N_2-gas TL reader incorporating a post-irradiation annealing facility. *Nucl. Instrum. Meth.* **153,** 413–418.

Bøtter-Jensen, L. and Bundgaard, J. (1978) An automatic reader for TL dating. *PACT* **2,** 48–56.

Bøtter-Jensen, L. and Mejdahl, V. (1980) Determination of archaeological doses for TL dating using an automated TL apparatus. *Nucl. Instrum. Meth.* **175,** 213–215.

Bøtter-Jensen, L., Bundgaard, J. and Mejdahl, V. (1983) An HP-85 microcomputer-controlled automated reader system for TL dating. *PACT* **9,** 343–349.

Bøtter-Jensen, L. (1988) The automated Risø TL dating reader system. *Nucl. Tracks Radiat. Meas.* **14,** 177–180.

Bøtter-Jensen, L., Ditlevsen, C. and Mejdahl, V. (1991) Combined OSL (infrared) and TL studies of feldspars. *Nucl. Tracks Radiat. Meas.* **18,** 257–263.

Bøtter-Jensen, L. and Duller, G. A. T. (1992) A new system for measuring OSL from quartz samples. *Nucl. Tracks. Radiat. Meas.* **20,** 549–553.

Bøtter-Jensen, L., Duller, G. A. T. and Poolton, N. R. J. (1994a) Excitation and emission spectrometry of stimulated luminescence from quartz and feldspars. *Radiat. Meas.* **23,** 613–616.

Bøtter-Jensen, L., Poolton, N. R. J., Willumsen, F. and Christiansen, H. (1994b) A compact design for monochromatic OSL measurements in the wavelength range 380–1020 nm. *Radiat. Meas.* **23,** 519–522.

Bøtter-Jensen, L., Jungner, H. and Poolton, N. R. J. (1995) A continuous OSL scanning method for analysis of radiation depth-dose profiles in bricks. *Radiat. Meas.* **24,** 525–529.

Bøtter-Jensen, L., Markey, B. G., Poolton, N. R. J. and Jungner, H. (1996) Luminescence properties of porcelain ceramics relevant to retrospective radiation dosimetry. *Radiat. Prot. Dosim.* **65**(1–4), 369–372.

Bøtter-Jensen, L. and McKeever, S. W. S. (1996) Optically stimulated luminescence dosimetry using natural and synthetic materials. *Radiat. Prot. Dosim.* **65**(1–4), 273–280.

Bøtter-Jensen, L., Agersnap Larsen, N., Markey, B. G. and McKeever, S. W. S. (1997a) Al_2O_3:C as a sensitive OSL dosemeter for rapid assessment of environmental photon dose rates. *Radiat. Meas.* **27,** 295–298.

Bøtter-Jensen, L., Mejdahl, V. and Murray, A. S. (1997b) New light on OSL. Submitted to *Quat. Sci. Rev.* (*Quat. Geochron*).

Bräunlich, P., Gasiot, J., Fillard, J. P. and Castagné, M. (1981) Laser heating of thermoluminescent dielectric layers. *Appl. Phys. Lett.* **39**(9), 769–771.

Brou, R. and Valladas, G. (1975) Appareil pour la mesure de la thermoluminescence des petits échantillons. *Nucl. Instrum. Methods* **127,** 109–113.

Clark, R. J. and Sanderson, D. C. W. (1994) Photostimulated luminescence excitation spectroscopy of feldspars and micas. *Radiat. Meas.* **23,** 641–646.

Daniels, F., Boyd, C. A. and Saunders, D. F. (1953) Thermoluminescence as a research tool. *Science* **117,** 343–349.

Ditlevsen, C. and Huntley, D. J. (1994) Optical excitation of trapped charges in quartz, potassium feldspars and mixed silicates: the dependence on photon energy. *Radiat. Meas.* **23,** 675–682.

Duller, G. A. T. (1991) Equivalent dose determination using single aliquots. *Nucl. Tracks. Radiat. Meas.* **18,** 371–378.

Duller, G. A. T. and Bøtter-Jensen, L. (1996) Comparison of optically stimulated luminescence signals from quartz using different stimulation wavelengths. *Radiat. Meas.* **26,** 603–609.

Duller, G. A. T., Bøtter-Jensen, L. and Markey, B. G. (1997) A luminescence imaging system based on a charge coupled device (CCD) camera. *Radiat. Meas.* **27,** 91–99.

Galloway, R. B. (1991) A versatile 40-sample system for TL and OSL investigations. *Nucl. Tracks Radiat. Meas.* **18,** 265–271.

Galloway, R. B. (1993) Stimulation of luminescence using green light emitting diodes. *Radiat. Prot. Dosim.* **47,** 679–682.

Galloway, R. B. (1994) On the stimulation of luminescence with green light emitting diodes. *Radiat. Meas.* **23**(2/3), 547–550.

Galloway, R. B. (1996) Equivalent dose determination using only one sample: alternative analysis of data obtained from infrared stimulation of feldspars. *Radiat. Meas.* **26,** 103–106.

Galloway, R. B., Hong, D. G. and Napier, H. J. (1997) A substantially improved green light emitting diode system for luminescence stimulation. *Meas. Sci. Technol.* **8,** 267–271.

Godfrey-Smith, D. I., Huntley, D. J. and Chen, W.-H. (1988) Optical dating studies of quartz and feldspar sediment extracts. *Quat. Sci. Rev.* **7,** 373–380.

Godfrey-Smith, D. I. and Haskell, E. H. (1993) Application of optically stimulated luminescence to the dosimetry of recent radiation events monitoring low total absorbed dose. *Health Phys.* **65,** 396–404.

Harris, A. M. and Jackson, J. H. (1970) A rapid scanning spectrometer for the region 200–850 nm: application of thermoluminescent emission spectra. *J. Phys.* **E3,** 374.

Hashimoto, T., Hayashi, Y., Koyanagi, A., Yokosaka, K. and Kimura, K. (1986) Red and blue coloration of thermoluminescence from natural quartz sands. *Nucl. Tracks Radiat. Meas.* **11,** 229–235.

Hashimoto, T., Yokasaka, K., Habaku, H. and Hayashi, Y. (1989) Provenance search of dune sands using thermoluminescence colour images (TLCIs) from quartz grains. *Nucl. Tracks Radiat. Meas.* **16,** 3–10.

Hashimoto, T., Notoya, S., Ojima, T. and Hoteida, M. (1995) Optically stimulated luminescence (OSL) and some other luminescence images from granite slices exposed with radiations. *Radiat. Meas.* **24,** 227–237.

Henzinger, R., Kubelik, M. and Vana, N. (1994) Die entwicklung eines vollautomatischen TL- auswertegerätes (HVK) unter besonderer berücksichtigung der ziegeldatierung. In: *Proc. Jahrestagung der Deutschen Mineralogischen Gesellschaft und der Gesellschaft Deutscher Chemiker-Arbeitkreis Archäometrie*, Oldenburg, März 1994.

Huntley, D. J., Godfrey-Smith, D. I. and Thewalt, M. L. W. (1985) Optical dating of sediments. *Nature* **313,** 105–107.

Huntley, D. J., Godfrey-Smith, D. I., Thewalt, M. L. W. and Berger, G. W. (1988) Thermoluminescence spectra of some mineral samples relevant to thermoluminescence dating. *J. Lumin.* **39,** 123–136.

Huntley, D. J., Short, M. A. and Dunphy, K. (1996) Deep traps in quartz and their use for optical dating. *Can. J. Phys.* **74,** 81–91.

Hütt, G., Jaek, I. and Tchonka, J. (1988) Optical dating: K-feldspars optical response stimulation spectra. *Quat. Sci. Rev.* **7,** 381–386.

Hütt, G. and Jaek, I. (1989) Infrared stimulated photoluminescence dating of sediments. *Ancient TL* **7,** 48–51.

Hütt, G. and Jaek, I. (1990) Photoluminescence dating on alkali feldspars: Physical ground, equipment and some results. *Radiat. Prot. Dosim.* **34,** 73–74.

Kawamura, K. and Hashimoto, T. (1995) Construction of automatic photographic system for after-glow colour images (AGCI). *Radioisotopes* **44,** 379–388.

Krause, W. E., Krbetschek, M. R. and Stolz, W. (1997) Dating of Quaternary Lake sediments from the Schirmacher Oasis (East Antarctica) by infra-red stimulated luminescence (IRSL)detected at the wavelength of 560 nm. *Quat. Sci. Rev. (Quat. Geochron.)* **16,** 387–392.

Lamothe, M., Balescu, S. and Auclair, M. (1994) Natural IRSL intensities and apparent luminescence ages of single feldspar grains extracted from partially bleached sediments. *Radiat. Meas.* **23,** 555–562.

Li, S.-H. (1994) Optical dating: insufficiently bleached sediments. *Radiat. Meas.* **23,** 563–567.

Luff, B. J. and Townsend, P. D. (1993) High sensitivity thermoluminescence spectrometer. *Meas. Sci. Technol.* **4,** 65–71.

Markey, B. G., Bøtter-Jensen, L., Poolton, N. R. J., Christiansen, H. E. and Willumsen, F. (1996) A new sensitive system for measurement of thermally and optically stimulated luminescence. *Radiat. Prot. Dosim.* **66**(1/4), 413–418.

Markey, B. G., Bøtter-Jensen, L. and Duller, G. A. T. (1997) A new flexible system for measuring thermally and optically stimulated luminescence. *Radiat. Meas.* **27,** 83–89.

Martini, M., Paravisi, S. and Liguori, C. (1996) A new high sensitive spectrometer for 3-D thermoluminescence analysis. *Radiat. Prot. Dosim.* **66,** 447–450.

Mattern, P. L., Lengweiler, K. and Levy, P. W. (1971) Apparatus for the simultaneous determination of thermoluminescent intensity and spectral distribution. *Mod. Geol.* **2,** 293–294.

McFee, C. J. and Tite, M. S. (1994) Investigations into the thermoluminescence properties of single quartz grains using an imaging photon detector. *Radiat. Meas.* **23,** 355–360.

McKeever, S. W. S., Markey, B. G. and Akselrod, M. S. (1996) Pulsed optically-stimulated luminescence dosimetry using α-Al_2O_3:C. *Radiat. Prot. Dosim.* **65,** 267–272.

Mejdahl, V. (1969) Thermoluminescence dating of ancient Danish ceramics. *Archaeometry* **11,** 99–104.

Mejdahl, V. (1990) Thermoluminescence dating. *Norw. Arch. Rev.* **23,** 21–29.

Mejdahl, V. and Bøtter-Jensen, L. (1994) Luminescence dating of archaeological materials using a new technique based on single aliquot measurements. *Quat. Sci. Rev. (Quat. Geochron.)* **13,** 551–554.

Mejdahl, V. and Bøtter-Jensen, L. (1997) Experience with the SARA OSL method. *Radiat. Meas.* **27,** 291–294.

Murray, A. S., Olley, J. M. and Caitcheon, G. C. (1995) Measurement of equivalent doses in quartz from contemporary water-lain sediments using optically stimulated luminescence. *Quat. Sci. Rev. (Quat. Geochron.)* **14,** 365–371.

Murray, A. S., Roberts, R. G. and Wintle, A. G. (1997) Equivalent dose measurement using a single aliquot of quartz. *Radiat. Meas.* **27,** 171–184.

Murray, A. S. and Roberts, R. G. (1997) Determining the burial time of single grains of quartz using optically stimulated luminescence. Earth Planet. Sci. Lett. (in press).

Murray, A. S. and Wintle, A. G. (1997) Factors controlling the shape of the OSL decay curve in quartz. *Radiat. Meas.*, in press.

Pierson, J., Forman, S. L., Lepper, K. and Conley, G. (1994) A variable narrow bandpass optically stimulated luminescence system for Quaternary geochronology. *Radiat. Meas.* **23,** 533–535.

Poolton, N. R. J. and Bailiff, I. K. (1989) The use of LEDs as an excitation source for photoluminescence dating of sediments. *Ancient TL* **7,** 18–20.

Poolton, N. R. J., Bøtter-Jensen, L., Wintle, A. G., Jakobsen, J., Jørgensen, F. and Knudsen, K.

L. (1994) A portable system for the measurement of sediment OSL in the field. *Radiat. Meas.* **23,** 529–532.

Poolton, N. R. J., Bøtter-Jensen, L. and Duller, G. A. T. (1995) Thermal quenching of luminescence processes in feldspars. *Radiat. Meas.* **24,** 57–66.

Poolton, N. R. J., Bøtter-Jensen, L., Wintle, A. G., Ypma, P. J., Knudsen, K. L., Mejdahl, V., Mauz, B., Christiansen, H. E., Jakobsen, J., Jørgensen, F. and Willumsen, F. (1996a) A scanning system for measuring the age-related luminescence of split sediment cores. *Boreas* **25,** 195–207.

Poolton, N. R. J., Bøtter-Jensen, L. and Johnsen, O. (1996b) On the relationship between luminescence excitation spectra and feldspar mineralogy. *Radiat. Meas.* **26,** 93–101.

Prescott, J. R., Fox, P. J., Akber, R. A. and Jensen, H. E. (1988) Thermoluminescence emission spectrometer. *Appl. Phys.* **27**(16), 3496–3502.

Rees-Jones, J., Hall, S. J. B. and Rink, W. J. (1997) A laboratory inter-comparison of quartz optically stimulated luminescence (OSL) results. *Quat. Sci. Rev. (Quat. Geochron.)* **16,** 275–280.

Rhodes, E. J. (1988) Methodological considerations in the optical dating of quartz. *Quat. Sci. Rev.* **7,** 359–400.

Rhodes, E. J. and Pownall, L. (1994) Zeroing of the OSL signal in quartz from young glaciofluvial sediments. *Radiat. Meas.* **23,** 581–585.

Rieser, U., Krebetcheck, M. R. and Stolz, W. (1994) CCD-camera based high sensitivity TL/OSL-spectrometer. *Radiat. Meas.* **23,** 523–528.

Sanderson, D. C. W., Slater, C. and Cairns, K. J. (1989) Detection of irradiated food. *Nature* **340,** 23–24.

Sanderson, D. C. W., Carmichael, L. A., Ni Rian, S., Naylor, J. D. and Spencer, J. Q. (1994) Luminescence studies to identify irradiated food. *Food Sci. Technol. Today* **8**(2), 93–96.

Sanderson, D. C. W., Carmichael, L. A. and Naylor, J. D. (1995) Photostimulated luminescence and thermoluminescence techniques for the detection of irradiated food. *Food Sci. Technol. Today* **9**(3), 150–154 (B).

Sanderson, D. C. W., Carmichael, L. A. and Naylor, J. D. (1996) Recent advances in thermoluminescence and photostimulated luminescence detection methods for irradiated foods. In: *Detection Methods for Irradiated Food: Current Status* (eds C. H. McMurray *et al.*), pp. 124–138. Royal Society of Chemistry, Cambridge.

Schreiber, G. A., Helle, N. and Bögl, K. W. (1993) Detection of irradiated food – methods and routine application (a review). *Int. J. Radiat. Biol.* **63,** 105–130.

Shapiro, E. G. (1970) A wide-dynamic current-to-frequency converter. IEEE Trans. Nucl. Sci. NS-17, no. 1, 335.

Singhvi, A. K. (1996) Personal communication.

Smith, B. W., Wheeler, G. C. W. S., Rhodes, E. J. and Spooner, N. A. (1991) Luminescence dating of zircon using an imaging photon detector. *Nucl. Tracks Radiat. Meas.* **18,** 273–278.

Spooner, N. A., Prescott, J. R. and Hutton, J. T. (1988) The effect of illumination wavelength on the bleaching of thermoluminescence (TL) of quartz. *Quat. Sci. Rev.* **7,** 325–330.

Spooner, N. A., Aitken, M. J., Smith, B. W., Franks, M. and McElroy, C. (1990) Archaeological dating by infrared stimulated luminescence using a diode array. *Radiat. Prot. Dosim.* **34,** 83–86.

Spooner, N. A. and Questiaux, D. G. (1990) Optical dating – Achenheim beyond the Eemian using green/infrared stimulation. In: *Synopsis from a Workshop on Long and Short Range Limits in Luminescence Dating*, April 1989. RLAHA Occasional Publication No. 9, pp. 97–103, Oxford.

Spooner, N. A. (1994) On the optical dating signal from quartz. *Radiat. Meas.* **23**(2/3), 593–600.

Strickertsson, K. (1985) The thermoluminescence of potassium feldspars – glow curve characteristics and initial rise measurements. *Nucl. Tracks Radiat. Meas.* **10,** 613–617.

Templer, R. and Walton, A. (1983) Image intensifier studies of TL in zircons. *PACT* **9,** 299–308.

Templer, R. H. (1985) The localised transition model of anomalous fading. *Radiat. Prot. Dosim.* **17,** 493–497.

Unfried, E. and Vana, N. (1982) Archaeological dating at the Atominstitute Vienna. *PACT* **6,** 438–445.

Valladas, G. and Gillot, P. Y. (1978) Dating of Olby lava flow using heated quartz pebbles: some problems. *PACT* **2,** 141–150.

Valladas, G., Mercier, N. and Létuvé, R. (1996) A simple automatic TL apparatus of new design. Ancient TL, in press.

Vana, N., Erlach, R., Fugger, M., Gratzl, W. and Reichhalter, I. (1988) A computerized TL read-out system for dating and phototransfer measurements. *Nucl. Tracks Radiat. Meas.* **14,** 181–184.

Visocekas, R. (1979) De la luminescence da la calcite aprés irradiation cathodique: thermoluminescence et luminescence par effet tunnel. Thése de Doctorat d'Etat, Université Pierre et Marie Curie.

Visocekas, R. (1993) Tunnelling radiative recombination in K-feldspar sanidine. *Nucl. Tracks Radiat. Meas.* **21,** 175–178.

Wintle, A. G. (1973) Anomalous fading of thermoluminescence in mineral samples. *Nature* **245,** 143–144.

Wintle, A. G. (1975) Thermal quenching of thermoluminescence in quartz. *Geophys. J. R. Astr. Soc.* **41,** 107–113.

Wintle, A. G. and Huntley, D. J. (1980) Thermoluminescence dating of ocean sediments. *Can. J. Earth Sci.* **17,** 348–360.

Wintle, A. G. and Murray, A. S. (1997) The relationship between quartz thermoluminescence, photo-transferred thermoluminescence, and optically stimulated luminescence. *Radiat. Meas.*, **27,** 611–624.