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Some characteristics of infrared emitting diodes relevant to luminescence dating

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Introduction

Following the report by Hütt at the 1987 Cambridge TL Seminar (Hütt et al, 1988) that a dating signal could be obtained from K-feldspar using stimulation wavelengths of 860 nm and 930 nm, we investigated the use of an array of IR emitting diodes for this purpose. This paralleled work at Durham already reported (Poolton and Bailiff, 1989), and follows the success of Godfrey-Smith et al (1988) in exciting luminescence from feldspars by such a diode. Although various sources of IR radiation have been applied - for example, Hütt et al in their original work used a xenon lamp and monochromator, and later (Hütt and Jaek, 1990) an excimer dye laser and IR diode laser - the utilization of IR LED's appears attractive for reasons of economy, safety and convenience.

Trials of a simple IR LED set based on an array of twelve 880 nm diodes (TEMT 8850) showed a substantial fall in output intensity during the minutes after switch-on, the percentage difference between the initial and stable levels increasing with current. Similar behaviour has been reported by Poolton and Bailiff (1989) for an array of 16 Telefunken TSUS 5402 diodes. This suggested the need for "photofeedback control" if a constant intensity beam was to be maintained. To this end, a 1cm "large area" silicon photodiode was incorporated into the unit (peak response of 0.5A/W at 900 nm, diminishing to 5% of peak response at 350 nm and 1150 nm). While this approach was very successful for short exposures (<1 s), it proved unable to control satisfactorily the output beam for extended exposures (>1 s). Following verification that the photodiode and associated circuitry performed correctly, it was established that the failure to control resulted from a drift in the output spectrum of these IR diodes of ~10 nm, to longer wavelengths, which when coupled to the wavelength dependent response of the photodiode meant that although the photodiode output was held constant, the LED output was not. This occasioned a comprehensive investigation of IR LED characteristics, as reported in the next section. Brief details of the unit that was subsequently developed and some aspects of its performance are given in the final sections.

IR LED characteristics

The spectral dependence of the responsivity of suitable controlling photodiodes made it appear necessary for an LED type showing virtually no wavelength drift to be found. The "initial" output spectrum (2 seconds after switch on) and that after "stabilization" by 30 minutes continuous operation were therefore measured, using a silicon photodiode at the exit slit of an Oriel monochromator (model 7240). The set was calibrated using a sodium lamp, and the spectra corrected for detector response. The percentage reduction of radiant power and the magnitude of the shift to longer wavelengths was greater at higher currents in all cases, as is to be expected since the changes are dependent on LED junction temperature (eg Optics Guide 4, Melles Griot, 1988; GE/RCA Optoelectronics Data Book, 1987). Some examples of measured diode spectra are given in figure 1.

A significant difference between the behaviour of the resin bodied diodes and that of the metal encased diodes tested (see table 1) became apparent - the latter showed more severe intensity and wavelength shifts than resin-bodied types. Also, as a metal-encased diode requires electrical isolation from its neighbours (for series connection) this type was excluded from further study.

The output of unwanted, visible wavelength emissions was also considered. Emissions from arrays of 24 diodes each of types TSUS 5402 and TEMT 484 were measured by an EMI 9635Q PMT and 2 mm thick Schott BG 39 colour glass filter (passband 370 nm to 580 nm; 50% transmission) monitoring light scattered from a ground glass disc (25 mm diameter) placed on the TL oven plate. At a diode current of 50 mA, without the 12 mm diameter light guide and associated holder positioned, the TSUS 5402 array gave a countrate of ~150 cps above dark count, and the TEMT 484 gave ~100 cps. When the light guide and holder were emplaced both countrates dropped, to ~48 cps and ~10 cps respectively. The addition of a 4 mm thick Corning 7-59 filter (passband 320 nm to 430 nm; 50% transmission) reduced the countrate from the TSUS 5402 array to ~5 cps, but left the TEMT 484 array value unchanged. Given that the ground glass disc has about 2 - 4 times the reflectivity of a typical clean stainless steel disc, these levels were considered acceptable. It is assumed that visible wavelengths were involved but incomplete blockage of IR by the BG 39 cannot be ruled out.

The LED array

By operating the diodes at a low enough current for warm-up effects Poolton and Bailiff (1989) obtained satisfactory operation without feedback control. In our module we use higher currents but allow a warm-up time (typically 30 min) before measurements are commenced; sample illumination is actuated by means of a fast electronic shutter. A passive photodiode monitors the IR beam and permits manual control. An advantage of this arrangement is that, with shutter closed, the output current of the photodiode, being dependent on the detection of IR photons scattered from the shutter blades, avoids any effects due to variations in sample-to-sample reflectivity.

The LED chosen was TEMT 484 (for spectrum, see fig. 2). The narrow beam cone permits the array to be focused onto the sample from a sufficient distance to allow the shutter to be interposed, without losing power at the sample by beam spreading. The array is shown in figure 3, and further details may be found in Spooner et al (1990).

Performance

The unit is inserted in a standard TL set between the PMT and glow-oven, so making the TL oven available as a servo- controlled isothermal plate to maintain the sample temperature to within ~ ±1 °C; this is necessary because the initial sample luminescence increases by ~ 1 % / °C.

The run-to-run variation of beam intensity, as set manually from the monitoring photodiode reading and measured at the sample position by an on-plate photodiode arrangement is < 0.5% at 50 mA. Even without manual control long term stability is good - over 10 hours the beam intensity remained within ~ ±1 % of the intensity 1 hour after switch on. This was with the laboratory temperature held constant to ~ ±3 °C using an air conditioner.

The uniformity of sample illumination was checked by observing the luminescence stimulated by giving short IR exposures to a small chip of irradiated microcline feldspar positioned at various points on a sample disc (following Poolton and Bailiff, 1989). The intensity profile across a 9.7 mm disc was found to be circularly symmetric, with peak power at the central position dropping to 95 % at 3 mm distance and ~ 80% at the rim. Rotation of the module through 360° indicated a variation in total power delivered to the sample position of <0.5 %, as monitored by the on-plate photodiode (see fig. 4). The IR power received by the sample was 10.7 mW cm^{-2} at 50 mA; measured using the on-plate photodiode calibrated against a known power beam of 514.5 nm Ar-ion laser light, with due allowance being made for wavelength difference. In addition, qualitative observations of the IR beam were made using IR convertor cards, while a convenient check of the overall system performance is given by short-exposure monitoring of standardized portions of a bright, old sample, such as loess of several hundred thousand years age.

A single 2 mm Schott BG 39 is used as the basic optical filter required to shield the PMT from scattered IR photons (the unshielded EMI 9635Q tube detects ~105 cps from the above set, operated at 50 mA). Additional filters, such as Corning 7-59 or Schott GG-455 colour glass filters, may be added to this to select more specific detection wavebands. Leakage of IR onto the sample through the closed shutter was measured as $<10^{-6}$ of the beam power received by the sample with shutter open.

Summary

All types of LED tested showed output beam wavelength shifts and intensity decreases during warm-up; the wavelength shift precludes the use of a straightforward feedback control system based on a photodiode. Hence for our array we have chosen a type (TEMT 484) having a narrow beam which allows the diodes to be sufficiently far from the sample for an electronic shutter to be interposed; the diodes are kept on continuously and so deliver a stable output beam.

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Table 1. Characteristics of various IR LEDs.

Diode	Manufacturer	Power	Cone 1/2 angle	Peak λ (nm)	Measured FWHM (nm)	Spectral shift (nm)	Spectral shift (nm)	Int'y change Power	Temp. coeff's λ	Temp. coeff's	Comments
Note		*1*	*1*	*1*		*2*	*3*	*4*			
TEMT 8850	Three-Five Systems Ltd.	50 mW/SR at 100mA	15°	880	68	7	12	10.9	-	-	metal jacket
TEMT 484	Three-Five Systems Ltd.	100mW/SR at 100mA	8°	880	72	7	7	6	-0.53% per °C	-	resin
SFH 481-1	Siemens	10-20 mW/SR at 100mA	15°	880	86	8	14	14.3	-0.50% per °C	0.25 nm/°C	metal jacket
LED 55C	GE/RCA	5.4 mW at 100mA	15°	940	40	6	-	19.4	-	0.28 nm/°C	metal jacket
LED 55CF	GE/RCA	5.4 mW at 100mA	38°	940	40	7	-	15.5	-	0.28 nm/°C	metal jacket
CQX 19	Telefunken	20 mW at 250mA	40°	950	40	5	-	12	-	-	metal jacket (bulky)
TSUS 5402	Telefunken	15 mW at 100mA	25°	950	59	7	8	7.2	-0.80% per °C from 25°C	-	resin
IN 6266	GE/RCA	0.25 mW	20° at 100mA	940	40	5	-	11.6	-	0.28 nm/°C	metal jacket

Notes
1.Manufacturer's data
2.Shift in peak wavelength at 50 mA between 2 seconds and 30 minutes after switch on
3.Shift in peak wavelength at 100 mA between 2 seconds and 30 minutes after switch on
4.Intensity change at 50 mA between 2 seconds and 30 minutes

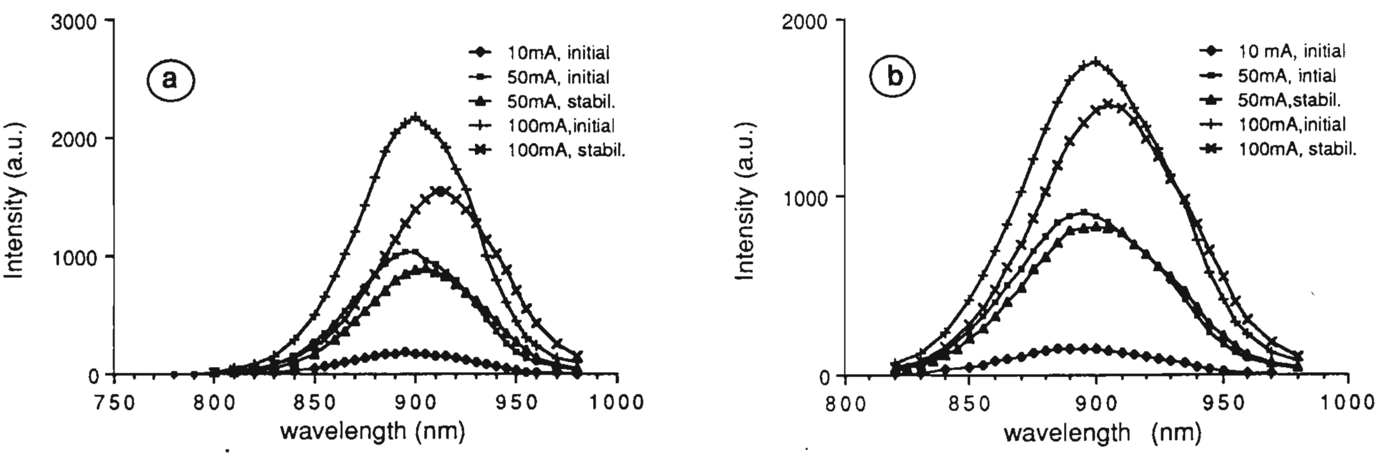

Figure 1.
The initial and stabilized output spectra of single (a) TEMT 8850 diode and (b) TEMT 484 diode, at 50 mA and 100 mA. Data taken following 30 minutes operation at 10 mA are also shown, and were indistinguishable from the initial output spectra at this current, within experimental error. Uncertainty in wavelength is within the data points. ("Initial" refers to measurements made 2 seconds after the diodes were switched on, and "stabilized" indicates 30 minutes continuous operation before measurement).

Figure 2
(a) The stabilized output spectrum of a single TEMT 484 diode operating at 50 mA.
(b) The optical response spectrum of alkali feldspar according to Hütt and Jaek (1989).

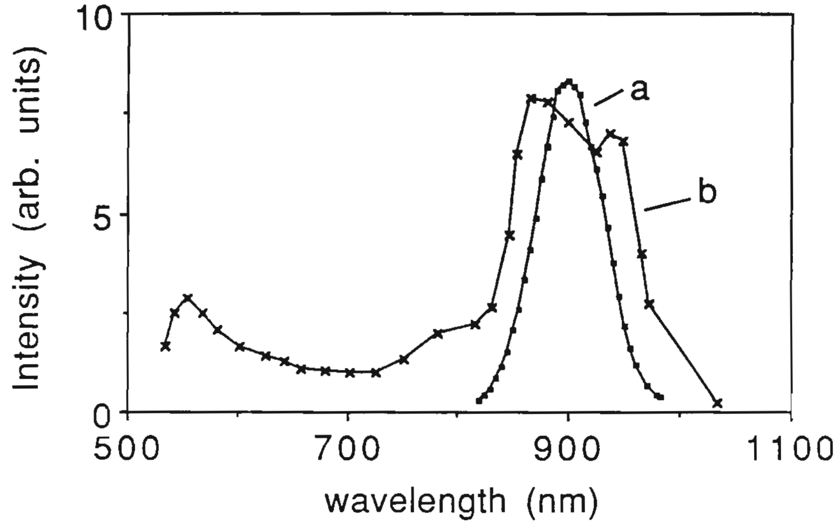

Figure 3
Schematic diagram showing the IR LED unit.
Components are: 1) sample, 2) 25.4 mm aperture Ilex electronic shutter, 3 ms full open or close time, Melles Griot, 3) diode array, 4) 12 mm diameter quartz light guide, 5) monitoring photodiode (Siemens IR-sensitive PIN photodiode, #BPW 34, temperature sensitivity ~0.05 %/°C), 6) monitoring thermo-couple, 7) Peltier effect heat pump, and 8) aluminium body.

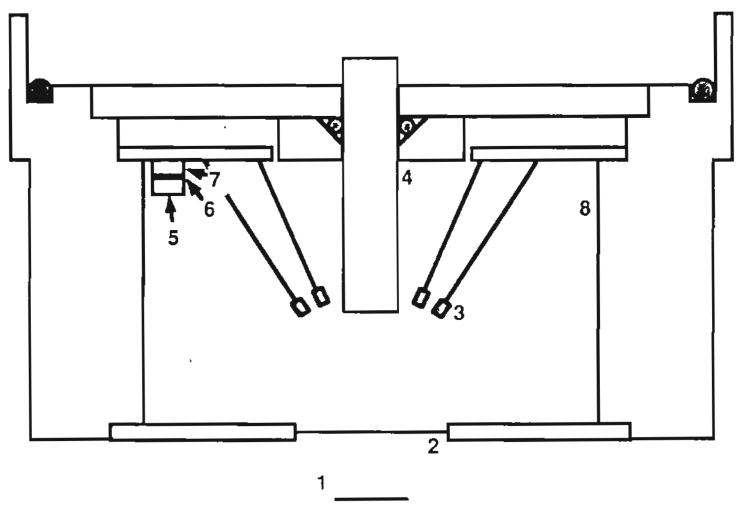

Figure 4
Schematic diagram showing the photodiode and filter assembly used for on-plate beam power calibrations and monitoring. The 1 cm^2 active area silicon photodiode presents a target area equivalent to that for a sample disc; flux reduction to levels giving linear photodiode response with power is achieved using 2 x 2.0 ND neutral density filters (Kodak and Schott).

1) LED array, 2) TL oven 3) oven plate, 4) photodiode, 5) filters, 6) X-Y spacer, 7) Z spacer.