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Application of pulsed OSL to the separation of the luminescence components from a mixed quartz/feldspar sample

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Abstract

It is known that the pulsed optically stimulated luminescence (OSL) characteristics of quartz and feldspars are very different. These differences can be used to preferentially discriminate against the feldspar signal in mixed quartz\feldspar mineral assemblages, or in separated quartz contaminated with a feldspar signal. We have developed instrumentation for the study of high-speed pulse stimulated OSL. Our system uses the standard blue/IR LED stimulation unit of a Risø reader (allowing stimulation pulses down to 1–2 μs duration) and can thus be applied to the routine analysis of samples. Using this stimulation source, and high-speed photon timing, the OSL yield can be monitored throughout the pulsing cycle and subsequent OSL decay. It is found that the total photon yield per unit stimulation power in pulsed mode is, for quartz, twice and, for feldspar, nearly four times, that in continuous wave mode. Observation of this OSL signal, between stimulation pulses, is seen to be characteristic of the mineral being examined, and has been used to preferentially discriminate against feldspar contamination in a mixed quartz/feldspar sample. Simple implementation of this technique by gating the counting period, so that counts are only accumulated during a windowed period, reduces the feldspar signal to 1.6% of its original value relative to that of the quartz.

Keywords: Pulsed; OSL; Luminescence; Lifetime; Time-resolved

1. Introduction

In the measurement of quartz OSL using blue or green stimulation and UV detection filters, contamination by a feldspar signal is often observed. This can be detected by infrared stimulation, which will give a signal from the feldspar but not from the quartz. Unfortunately, however, even prolonged IR stimulation does not remove all the blue stimulated signal from feldspar (Duller, 1997) and several other authors have developed measurement strategies to minimise any unwanted feldspar contamination (e.g. Banerjee et al., 2001; Wallinga et al., 2002). A further approach is presented here, namely the use of differences in luminescence lifetime to discriminate between quartz and feldspar signals under pulsed stimulation.

Pulsed luminescence techniques have long been used for the measurement of the optical properties of materials, for example, assessing the impurity and defects in semiconductors (e.g. Yano et al., 1998), and for better characterising electro-optical devices. Typically in these cases, the luminescence to be studied occurs at a lower energy than that of the breakthrough of stimulation light through the detection filter and so a pulsed technique is adopted to allow the unwanted, dominating fluorescence components to be removed by analysing their time dependence after the stimulation pulse, or to allow the photon detector to recover from the leakage of stimulation light through the optical filter. For such experiments, high-power pulsed lasers have been the obvious choice, offering exceptionally high-power density during pulses and being easily modulated to give ultra fast, reproducible pulses. In OSL (where the observed luminescence occurs at a *higher* energy than that of the stimulation), lasers were again the obvious choice for their high-stimulation intensity and easily focused beam

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(Huntley et al., 1985). However, as fluorescence occurs at a longer wavelength than the stimulation beam there was not the same need to pulse the stimulation laser. More recently with the advent of increasingly powerful and inexpensive light emitting diodes (LEDs) continuous wave OSL (CW–OSL) operation has become the standard mode of operation.

Several groups have, however, adopted pulsed techniques for OSL measurements: McKeever et al. (1996) uses high-power pulsed green laser stimulation to reduce the influence of background signal when measuring low doses with aluminium oxide. Sanderson (1991) uses a pulsed LED system to achieve a similar effect for detecting evidence of food irradiation. Clark et al. (1997), Sanderson and Clark (1994), and Bailiff (2000), have used pulsed laser techniques to investigate the luminescence characteristics of both quartz and feldspar, and have shown that quartz and feldspar have significantly different luminescence lifetimes (the characteristic time taken between stimulation and emission of luminescence); they then used this to identify quartz/feldspar rich sites on a solid sample section using a 2D scanning system (Bailiff and Mikhailik, 2003). Finally, Chithambo (2003) and Chithambo and Galloway (2000a,b) have carried out extensive experiments on the behaviour of quartz under pulsed OSL stimulation, measuring the effect of sample heating on the life time. These measurements were done using pulsed green LED stimulation.

In this paper, we outline the design and performance of a relatively inexpensive pulsed diode stimulation unit and fast photon counter, suitable for routine investigation. These facilities allow us to investigate the differences in time resolved characteristics of feldspar and quartz, and use these differences to quantitatively separate the quartz signal in a quartz/feldspar mixture, on a routine basis.

2. Instrument description

2.1. The diode pulsing unit

The pulsing system is a self-contained unit, based around an 8-bit microcontroller, with 8 k-byte of flash memory and control software. The power level, on- and off-time settings are user set via front panel thumb wheels. These settings are used to initialise the programmable timer and preset the voltage to be applied to each cluster of blue or IR diodes. The system is connected between the Risø Minisys OSL output and the LED stimulation head of a standard Risø automated reader (Bøtter-Jensen et al., 1999, 2000). The microcontroller detects the switch-on and switch-off signals from the Minisys for each of the blue and IR diode groups.

When a switch-on signal is received, the programmable timer begins to pulse the preset voltage connected to the selected LEDs, through a solid state switch. A delay of 800 ns is inserted between the programmable timer and this switch to allow for external/internal synchronisation, for example, with the high-speed counter, or counting window (discussed later).

Once pulsing of the LEDs has begun the stimulation intensity is monitored using a photo-diode built into the stimulation head. This feedback signal is digitised using a 14-bit analogue to digital converter so it can be read by the microcontroller. This then compensates for any drift in stimulation intensity by adjusting the voltage applied to the LEDs accordingly. For on pulses of $< 100\,\mu s$ this signal is monitored on a pulse by pulse basis; for longer periods, however, the pulses are monitored at regular intervals *within* the on-period itself.

So that the LED pulser unit may be used directly with the standard Minysis system, a photon-count gating circuit has been included. This provides a counting window within the *off-period* of each pulse cycle. Incoming TTL photo multiplier (PM) pulses are gated *off* while the LEDs are switched on, but allowed to pass, (to be counted) during the LED off-period. The exact starting time of this window relative to the diode pulse is preset using the internal synchronisation mentioned above; the start time can be adjusted from ~ 500 ns prior to the LEDs being switched off, to $\sim 25\mu s$ after. The counting window ends ~ 500 ns prior to the next LED switch-on pulse.

2.2. Fast photon counter

A fast counter has also been developed, to allow detailed studies of the luminescence behaviour during and after a stimulation pulse. The purpose of this counter is to label each photon count with the time of detection relative to the beginning of each diode pulse. The core of the counter is a high-speed first in first out (FIFO) buffer with asynchronous read/write times of 14 ns, and with $16\,k \times 9$ bits of data storage. The input end of this device is directly connected to an 8-bit clock (256 channels), which is driven by a standard crystal oscillator module allowing the dwell time (time resolution per channel) to be set from 18 ns to 1 μs (with a further divide down counter allowing up to 256 μs per channel, with an inevitable loss of precise synchronisation with the diode pulse). The output of this buffer connects to a 1 Mb/s universal serial bus (USB) transceiver with the data loading controlled by high-speed synchronous logic. A microcontroller has also been incorporated to allow the inclusion of data flags to signal the start and end of an OSL sequence in the data stream. The system takes its timing signals directly from the diode pulsing system described above, and the photon pulses from the PM tube preamplifier output of the Minisys control box.

A signal is sent to this counting system at the beginning of an OSL measurement (from the Minisys via the pulsing system); this then allows any subsequent photon pulses to be registered, and this in turn writes the current clock time to the high-speed buffer. The rising edge of the synchronising LED signal from the pulsing unit is used to reset this clock, write a zero to the high-speed buffer, and then increment the clock by one. This ensures the start of a stimulation pulse is written to the data stream in advance, and so any subsequent photons will be time stamped with respect to this. Thus the main limitation imposed by this approach is that the LEDs must be pulsed within $256 \times$ the channel resolution time. (Otherwise the clock will automatically reset to zero on the 257th crystal pulse). The asynchronous FIFO nature of the buffer allows data to be written independently of the output data stream and so the