

Time-resolved luminescence of low sensitivity quartz from crystalline rocks

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Abstract

Time-resolved luminescence spectra of low sensitivity natural quartz from crystalline rocks are presented. The luminescence was pulse-stimulated at $11 \mu\text{s}$ width using 470 nm blue light from quartz separated from plutonic, metamorphic, volcanic and hydrothermal samples. Measurements were made at 20 °C. All samples show evidence of a short lifetime component less than $5 \mu\text{s}$ long although in several cases too weak in intensity to be evaluated accurately. On the other hand, the value of the principal lifetime component varies considerably being about $86 \mu\text{s}$ in metamorphic quartz, $18 \mu\text{s}$ in plutonic quartz, and $100 \mu\text{s}$ in one example of hydrothermal quartz. The results illustrate a new feature of luminescence from quartz for which lifetimes less than $5 \mu\text{s}$ or greater than $75 \mu\text{s}$ have never been reported at room temperature before. It is argued that the thermal provenance of the quartz and so the annealing it will have experienced influences the size of the observed lifetime. In particular, the results are explained in terms of a model consisting of three luminescence centers with the dominant lifetime linked to preferential recombination at one center depending on the thermal history of the sample and hence the hole concentration of the center.

1. Introduction

Optically stimulated luminescence (OSL) from quartz, an ubiquitous natural dosimeter, has normally been measured using continuous optical stimulation. In this method, the luminescence is recorded simultaneously with stimulation light of essentially constant intensity. The luminescence and scattered stimulation light are discriminated by judicious use of band pass filters to transmit the luminescence and transmission filters to attenuate the intensity of the scattered stimulation light (Bøtter-Jensen, 1997 and Galloway et al., 1997). The intensity of the emitted luminescence decreases in time approximately exponentially to produce a decay curve. Typical decay curves can be deconvoluted by non-linear regression into three principal components; the fast, medium and slow component, respectively (Bailey et al., 1997, Chithambo and Galloway, 2001a and Smith and Rhodes, 1994). However, Bulur et al. (2000), using linearly ramped stimulation light, reported examples consisting of more than three components owing to the presence of multiple slow components. Further application of linearly modulated stimulation of luminescence showed that physical characteristics of each of the components, such as the photoionization cross-section, are not necessarily similar (Kuhns et al., 2000). Working along similar lines of investigation,

Chithambo and Galloway (2001a) showed that the luminescence in the slow component region in quartz is subject to both thermal enhancement and thermal quenching, the latter with an activation energy of the order of 0.64 eV. Subsequent experiments (Chithambo, 2003a) found that the activation energy of thermal assistance in the slow component differs from that in the fast and medium component regions of the OSL.

The measurements cited above (Chithambo, 2003a and Chithambo and Galloway, 2001a) were conducted using pulsed optical stimulation in which the stimulation and emission of luminescence are separated in time. The stimulation of luminescence, although still at constant intensity, is brief and the ensuing luminescence is monitored after the stimulation pulse. Pulsed optical stimulation thus produces a time-resolved luminescence spectrum consisting of two portions, the signal during the pulse and the luminescence after the pulse (Bailiff, 2000, Chithambo and Galloway, 2000a, McKeever et al., 1996 and Sanderson and Clark, 1994). Use of pulsed optical stimulation enables investigation of the time dependence of luminescence emission relative to the time of stimulation. Such investigations are possible because a time-resolved luminescence spectrum can be resolved into components each with an associated lifetime. Luminescence lifetimes as well as the form of the time-resolved luminescence spectrum provide information about the physical processes of luminescence and are thus a means of refining the use of luminescence in dating and dosimetry applications. Time-resolved optical stimulation of luminescence has been used to investigate a wide range of properties of luminescence from quartz. These include the temperature dependence of lifetimes (Bailiff, 2000; Chithambo and Galloway, 2000b, Chithambo and Galloway, 2001a and Chithambo and Galloway, 2001b), the influence of annealing on time-resolved luminescence and luminescence lifetimes (Chithambo, 2002, Chithambo, 2003a and Chithambo, 2003b; Galloway, 2002) as well as the effect of pre-measurement optical bleaching of luminescence on luminescence lifetimes (Chithambo and Galloway, 2001a and Chithambo and Galloway, 2001c; Chithambo, 2003b).

The bulk of research on time-resolved luminescence in quartz has been carried out on high sensitivity samples. For example, the quartz of Galloway and co-workers (Galloway et al., 1997 and Chithambo and Galloway, 2000a) increases in sensitivity with annealing by up to about 50 times relative to that of an unheated, daylight bleached sub-sample (Galloway, 2002).

Measurements of time-resolved luminescence by Bailiff (2000) and Bailiff and Mikhailik (2003) were also on comparatively sensitive material, namely quartz extracted from ceramics, pottery and brick each of which will have been fired at some stage during its preparation acquiring enhanced sensitivity to optical stimulation in the process. It should be noted that pulsed optical stimulation techniques have not been used for dating per se. For this reason, time-resolved luminescence in

materials encountered in OSL dating such as sedimentary or crystalline quartz has thus far remained largely unexplored.

The aim of this work is to study properties of time-resolved luminescence in quartz from crystalline rocks in order to assess the effect of geological provenance on luminescence lifetimes. The materials studied are known to be poorly sensitive to optical stimulation and to suffer changes in the sensitivity that cannot be properly corrected by the standard single-aliquot regenerative dose protocol (Preusser et al., 2006).

2. Geological details

Samples used in this study were collected as boulders from various river-beds all over Westland, the western foreland of the New Zealand Alps. The samples are either plutonic rocks, e.g., granite or orthogneiss (samples MOR, LR, MR1) or metamorphic rocks, e.g., augengneiss (samples TAR1, FJ2R, FJ2V) which formed during the alpine orogeny of the New Zealand Alps (Table 1; Preusser et al., 2006 and Suggate et al., 1978). In addition to these plutonic and metamorphic samples investigations were also carried out on quartz phenocrysts in a volcanic quartz-rhyolite from Nevada, USA (sample B9060) and a hydrothermal quartz crystal from the Bedretto valley, Switzerland (sample B7913). Plutonic and volcanic samples are typical for crystallization in a silicate melt as low- or high-quartz modification, respectively, and hydrothermal quartz is typical for crystallization in an aqueous solution at elevated temperature. In contrast, the metamorphic samples contain quartz which dynamically recrystallized under subgrain rotation and/or grain boundary migration conditions. The selected suite of samples thus represents the large variety of growth conditions of quartz found in nature. Geological details of samples studied are listed in Table 1.

Table 1.

Geological classification of samples studied

Sample	Origin	Occurrence	Type of rock	Modification	Approx. T_{\max} (°C)
B7913	Pizzo Vespero, Bedretto	Vein,	Crystal	Low-quartz	400
	Switzerland	Hydrothermal			
TAR1	Otira Gorge, Bridge to Brunner	River Cobble,	Quartz segregation	Low-quartz	450–550
	New Zealand	Metamorphic			
FJ2R	Franz Josef glacial forefield	River Cobble,	Augengneiss	Low-quartz	450–550
	New Zealand	Metamorphic			
FJ2V	Franz Josef glacial forefield	River Cobble,	Quartz segregation	Low-quartz	450–550
	New Zealand	Metamorphic	in FJ2		
MOR	Woodstock Gold Mine	River Cobble,	Granite	Low-/High-quartz	550–700
	New Zealand	Plutonic			
LR	Lake Rotorua	River Cobble,	Orthogneiss	Low-/High-quartz	550–700
	New Zealand	Plutonic			
MR1	Marnia river	River Cobble,	Granite	Low-/High-quartz	550–700
	New Zealand	Plutonic			
B9060	Bandelier National Monument	Host rock,	Quartz-Rhyolite	High-quartz	800
	Nevada, USA	Volcanic			

3. Experimental details

3.1. Sample preparation

All samples were ground then etched in fluorosilicic acid (H_2SiF_6) for 14 days followed by a further 40 min in 40% hydrogen fluoride (HF) to remove feldspar inclusions. Samples were rinsed

in distilled water following each chemical treatment. Luminescence measurements were made on samples deposited onto 9 mm diameter aluminium discs.

3.2. Pulsed optical stimulation of luminescence

Luminescence was measured using an LED-based pulsing instrument consisting of a pulse-stimulation system and a signal acquisition and processing assembly. The pulses, which are used to drive a set of LEDs, are generated from an integrated circuit multivibrator, a 2N74221, at pulse widths defined by its external RC timing components. The ensuing pulsed luminescence is detected and processed by a combination of an EMI 9635QA photomultiplier and a multichannel scaler (EG & G ORTEC MCS-plusTM). The design and operation of the instrument has been described in detail by [Chithambo and Galloway \(2000a\)](#) and some aspects of its performance by [Chithambo \(2005\)](#).

In this work, the multichannel scaler, set at a dwell time of $2\ \mu\text{s}$ and under software control, was used to simultaneously trigger a set of light-emitting diodes as well as to initiate a data-recording scan. Once a scan is started, the MCS begins to acquire photon counts, sequentially recording data in its memory channel-by-channel. The time-resolved luminescence spectrum is built up as a distribution of cumulative photon counts against time for the dynamic range selected. In this work, measurements were made over a dynamic range of $300\ \mu\text{s}$. Time-resolved luminescence spectra were measured over 4×10^5 – 5×10^6 scans to achieve good signal statistics. The number of sweeps used in this study is thus significantly greater than the 100 000 typically used in previous experiments (e.g. [Chithambo, 2003a](#) and [Chithambo, 2003b](#)) owing to the poor sensitivity to optical stimulation of the present quartz samples.

Luminescence was stimulated at a pulse width of $11\ \mu\text{s}$ per scan using a set of four 470 nm blue LEDs (Nichia NSPB-500), each pulsed at 70 mA. Luminescence was detected in the spectral region 340–380 nm (FWHM) by the photomultiplier through filters Schott BG39 (transmission band 340–620 nm FWHM) and UG11 (transmission band 270–380 nm FWHM). Each LED had a 3 mm thick GG420 long pass filter in front to attenuate scattered stimulation light. The signal during stimulation consists of scattered stimulation light from the LEDs and a monotonically increasing luminescence component. After the pulse, the luminescence is measured over photomultiplier noise only.

It should be noted that during stimulation, the time-resolved spectrum is influenced both by the shape of the LED stimulation pulse (fall time 25 ns) as well as by the lifetime of the luminescence of the material under study. However, after the pulse, the only factor affecting the shape of the

time-resolved spectrum is the luminescence lifetime ([Chithambo and Galloway, 2000a](#)). In the present study only the portion of spectra after the pulse was used hence the resultant lifetimes, all of the order of μs , are from the quartz samples to be discussed.

Time-resolved luminescence spectra were recorded at ambient temperature, typically $20\text{ }^{\circ}\text{C}$. Unless otherwise stated, samples were beta irradiated to 42.9 Gy before stimulation. The luminescence was measured from samples immediately after irradiation without any preheating. The preheating could be omitted because the phosphorescence prevalent after irradiation is not correlated in time with pulsed optical stimulation and so does not affect luminescence lifetimes ([Galloway, 2002](#)). Lifetimes were evaluated from portions of the time-resolved luminescence spectra after the pulse by fitting exponential functions of the form

$$f(t) = \sum_i A_i \exp\left(-\frac{t}{\tau_i}\right) + B, \quad (1)$$

where τ_i is the lifetime of the i th component, A_i is a scaling factor, t is time, and B a constant added for background. The curve-fitting was based on the Marquardt–Levenberg minimization method and was done using the commercial software SigmaPlot (SPSS Inc[®], USA).

The basis for use of exponential functions is that for a short rectangular pulse of width t_1 , in a material for which λ is the probability per unit time that a stimulated electron will produce luminescence during the excitation pulse, the number of stimulated electrons is $N(t_1)$. After the luminescence stimulation pulse, the luminescence $L(t)$ as the number of stimulated electrons decreases in time exponentially as

$$L(t) = \lambda N(t_1) \exp(-(t-t_1)/\tau), \quad (2)$$

where τ is the lifetime of the decay ([Chithambo and Galloway, 2000a](#)).

4. Results

4.1. Time-resolved luminescence of hydrothermal quartz

[Fig. 1](#) shows, in (a), a time-resolved spectrum measured over 5×10^6 scans from sample B7913 and a corresponding background signal and in (b), the portion of the time-resolved spectrum after the stimulation pulse. [Fig. 1](#) (a) shows that both during and after stimulation, the luminescence adds only a small component to the total measured signal. [Fig. 1](#)(a) further shows qualitative evidence of a short lifetime component in the region near $10\ \mu\text{s}$, too rapid to be resolved properly at the $2\ \mu\text{s}$ dwell time used to record the time-resolved luminescence.

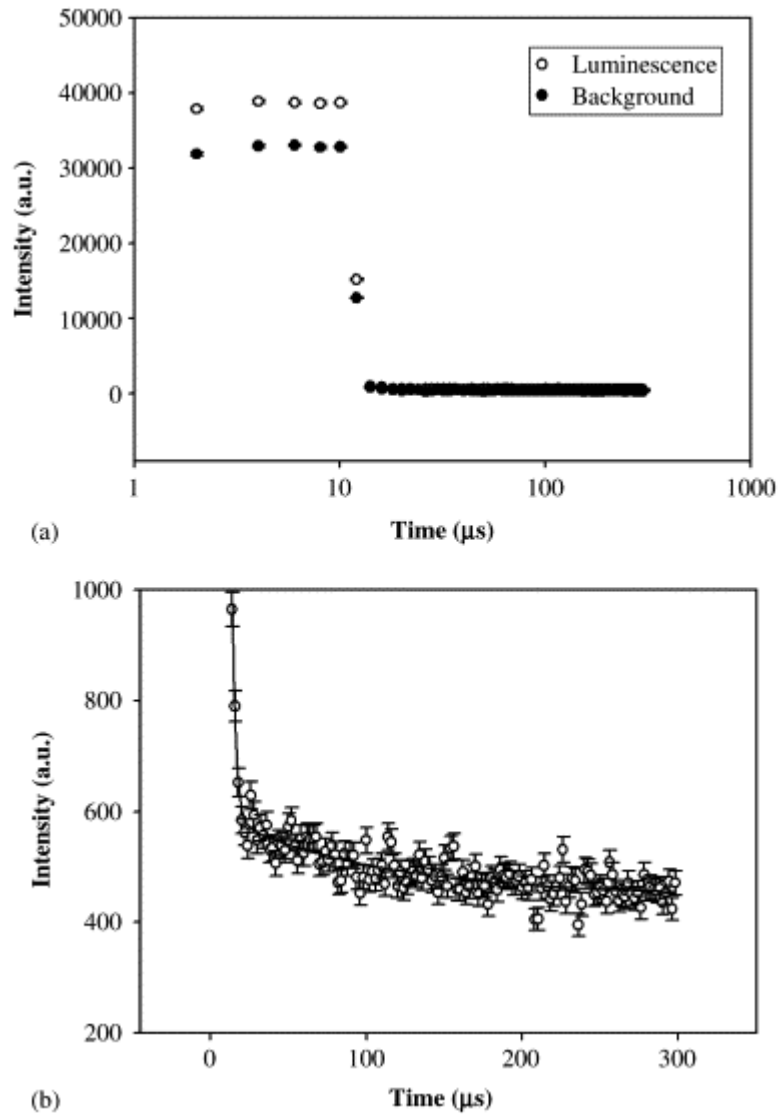


Fig. 1. Time-resolved luminescence spectrum measured over 5×10^6 scans from sample B7913 and the corresponding background signal (a) the luminescence after the stimulation pulse (b).

The time-resolved luminescence after the pulse (Fig. 1(b)) shows qualitatively that there are two possible lifetime components in the luminescence from sample B7913, the short lifetime mentioned above and a longer lifetime component decreasing in intensity up to $300 \mu\text{s}$ limit used. The solid line through the data points is the best fit of a sum of two exponential functions in Eq. (1). The best fit gave lifetime values of about $3 \mu\text{s}$, and $100 \pm 23 \mu\text{s}$. The precise calculated value of the shorter lifetime is a guide only since it has an order of magnitude similar to the dwell time used ($2 \mu\text{s}$) and cannot thus be expected to have been resolved accurately. Lifetimes of the order of either 3 or $100 \mu\text{s}$ as found in sample B7913 have never been reported in quartz and thus present a possible new feature of luminescence processes in quartz.

4.2. Time-resolved luminescence of volcanic quartz

Fig. 2 shows the time-resolved spectrum of luminescence measured from a sample of volcanic quartz, sample B9060. The form of the signal during stimulation, i.e. up to about $10\ \mu\text{s}$, is similar to that in Fig. 1, resembles that of a background, and hence shows the luminescence to be a minor component of the signal during stimulation. The spectrum further shows, as for B7913 in Fig. 1, the presence of a possible fast lifetime component that could not be reliably resolved at $2\ \mu\text{s}$ dwell time. However, the log-linear plot shows qualitative evidence of a longer lifetime component although with large statistical scatter in data points due to poor intensity of the luminescence from the sample. The luminescence after the pulse could be reliably fitted by a single exponential function (inset to Fig. 2) with a lifetime of $36.7 \pm 7.2\ \mu\text{s}$.

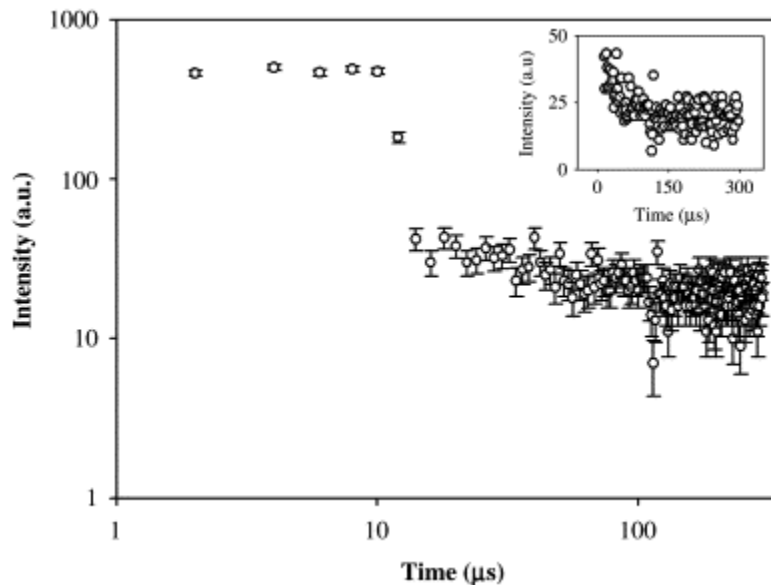


Fig. 2. The time-resolved luminescence spectrum of sample B9060. The inset shows the signal after the stimulation pulse fitted by a single exponential function.

4.3. Time-resolved luminescence of metamorphic quartz

Three samples of metamorphic quartz all from Westland, New Zealand, were investigated. These are labelled TAR1, FJ2V, and FJ2R.

Fig. 3 shows time-resolved spectra from samples TAR1 in (a) and FJ2V in (b). Fig. 3(a) measured over 1×10^6 scans, shows evidence of luminescence both during and after the stimulation pulse. The form of the spectra after the pulse suggests that the luminescence is dominated by a single long-lived component. The inset shows the signal after the pulse well fitted by a single exponential function with a best estimate for the lifetime of $86.4 \pm 4.3\ \mu\text{s}$. A similar value, $86.2 \pm 15.6\ \mu\text{s}$

was obtained for luminescence from sample FJ2V (accumulated over 4×10^6 scans) as shown in Fig. 3 (b). The size of the uncertainty in the lifetime corresponds to the amount of statistical scatter in the associated time-resolved luminescence spectrum.

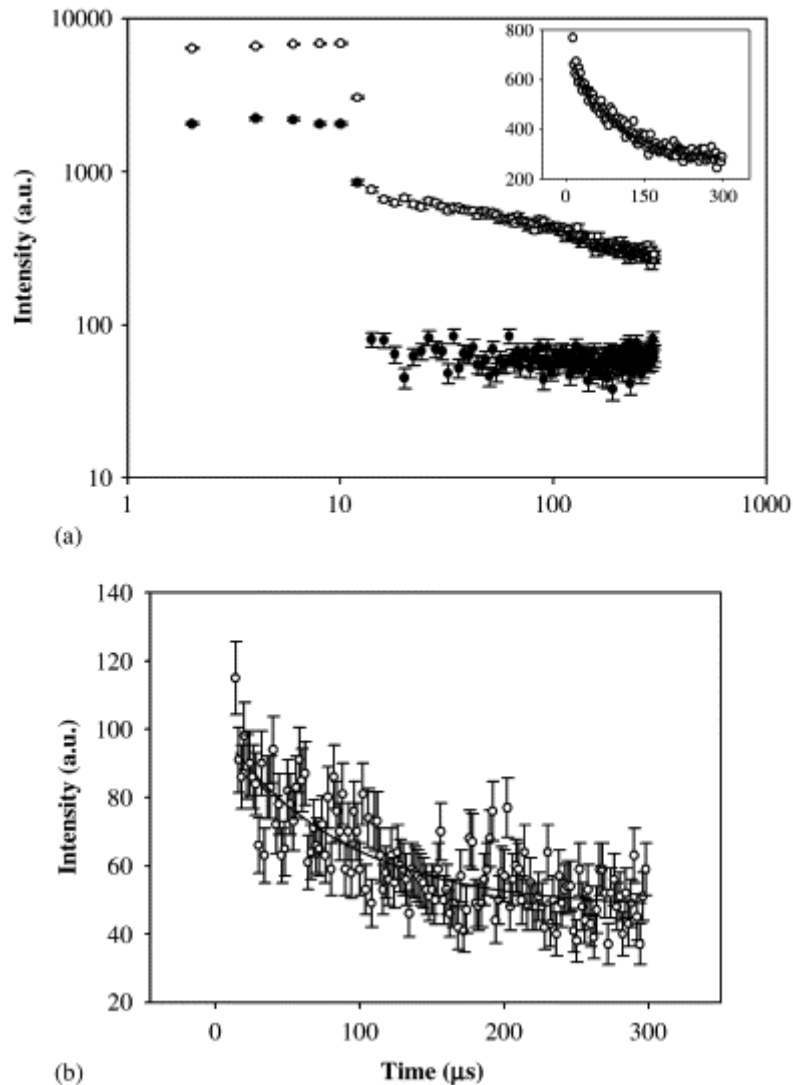


Fig. 3. Time-resolved luminescence from sample TAR1 (a) and FJ2V (b).

The time-resolved luminescence after the stimulation pulse from sample FJ2R, the third metamorphic quartz is shown in Fig. 4. The spectrum shows a clearly discernible change in form within the $300 \mu\text{s}$ dynamic range with clear evidence of a long lifetime component. The data could be fitted with a single-exponential function (solid line through data points) and adequately so as reflected by the residuals plot in the inset. The resultant lifetime was $75.9 \pm 4.7 \mu\text{s}$. Lifetimes in the range of $75\text{--}86 \mu\text{s}$ are longer than typical lifetimes in quartz (Bailiff, 2000, Chithambo and Galloway, 2000a and Chithambo and Galloway, 2001a).

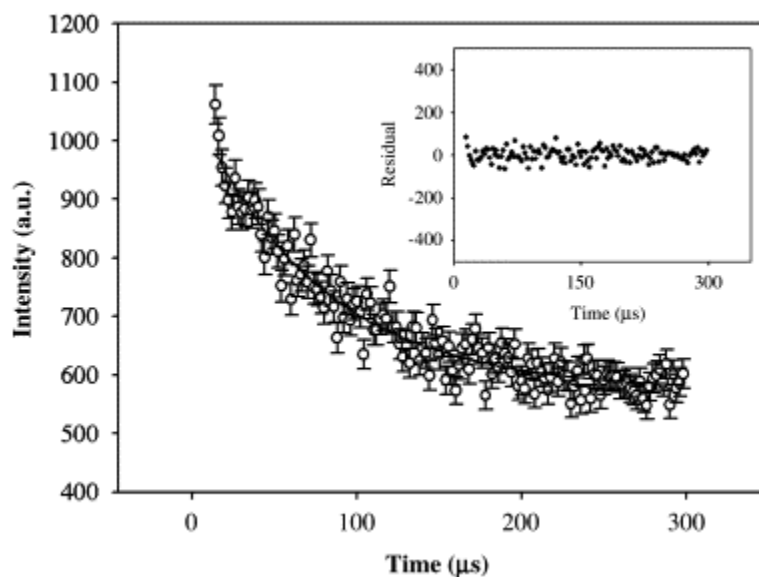


Fig. 4. Time-resolved luminescence from sample FJ2R. The solid line through data points is the best fit of a single exponential function. The residuals (inset) reflect the adequacy of the simple exponential fit.

4.4. Time-resolved luminescence of plutonic quartz

Three examples of plutonic quartz were studied, samples MR1 and MOR from granite and sample LR from an orthogneiss host rock.

Fig. 5 shows a time-resolved spectrum from sample MR1 accumulated over 1×10^6 scans plotted together with the corresponding background. The signal after the stimulation pulse shows some indication of a short lifetime component as well as evidence of a longer low-intensity component. Although the luminescence after the pulse could be fitted by a sum of two exponentials (inset), the resultant standard errors of the lifetimes were too large for the calculated lifetime values to be deemed reliable.

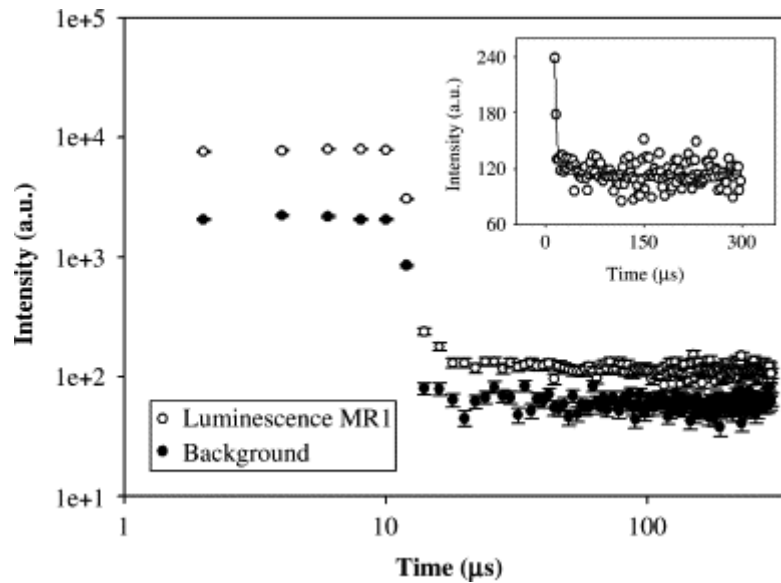


Fig. 5. Time-resolved luminescence from sample MR1. The inset shows the data after the stimulation pulse fitted by a sum of two exponential functions. The implications of the fit are discussed in the text.

Sample MOR is the second plutonic quartz investigated in these studies. In order to counter the particularly poor sensitivity to stimulation of this sample, a somewhat larger dose of 75 Gy was used. [Fig. 6](#) shows the time-resolved luminescence spectrum after the pulse. The inset to [Fig. 6](#) shows the full spectrum. The best fit to the time-resolved luminescence after the pulse needed only a single exponential term and gave a lifetime of $17.9 \pm 1.6 \mu\text{s}$.

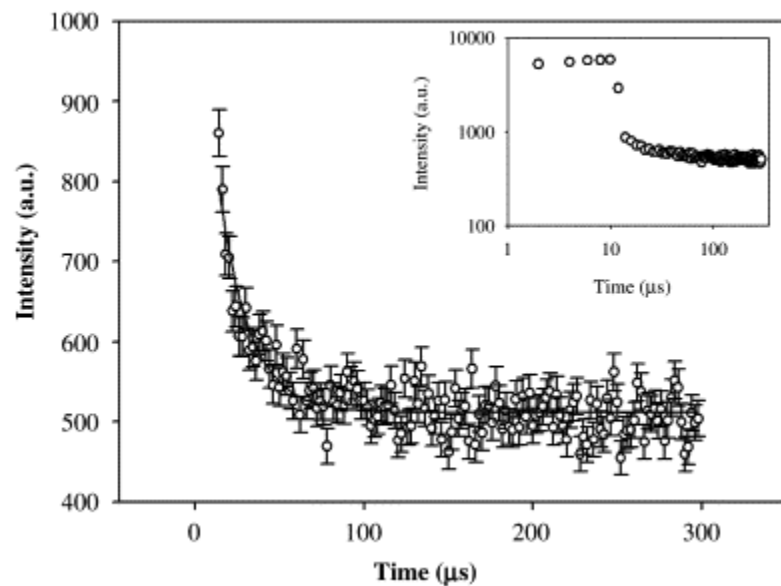


Fig. 6. Time-resolved luminescence from sample MOR. The solid line through data points is the best fit of a single exponential function. The inset shows the complete luminescence spectrum.

Fig. 7 shows a time-resolved luminescence spectrum from sample LR, a plutonic quartz extracted from an orthogneiss source rock. The spectrum shows qualitative evidence of two possible lifetime components, one near $10\ \mu\text{s}$ and a longer one thereafter too weak in intensity to be evaluated accurately. Non-linear regression analysis, requiring only one exponential term, confirmed the possible presence of a short lifetime with value $4.0 \pm 0.5\ \mu\text{s}$.

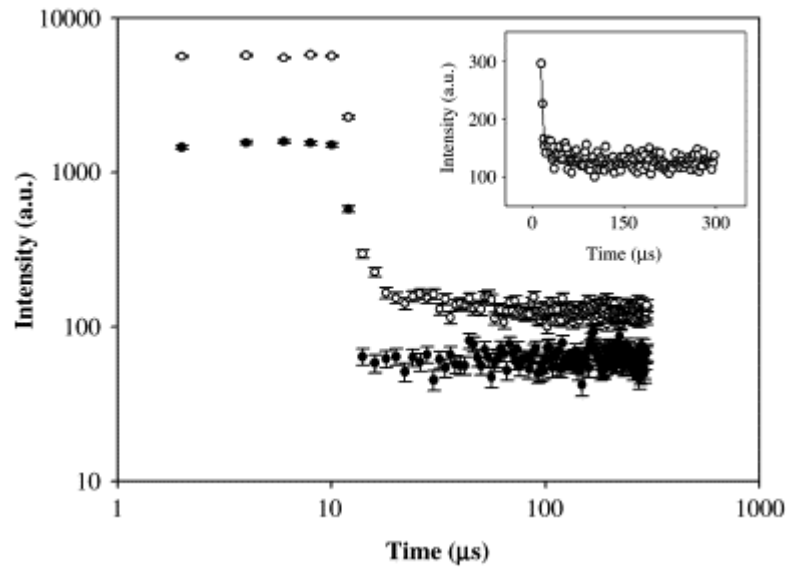


Fig. 7. Time-resolved luminescence from sample LR. The inset shows the luminescence after stimulation fitted by a single exponential function.

It should be noted that lifetimes of either 4 or $18\ \mu\text{s}$ as found in samples LR and MOR, respectively, have also been reported in some feldspars (Clark and Bailiff, 1998). Therefore, the possibility that the luminescence from samples LR and MOR was in each case from feldspar was tested using infrared stimulation (950 nm TSUS5402 LEDs) which should easily produce luminescence from feldspar but not from quartz at room temperature (Hütt et al., 1988 and Questiaux, 1991). It was found that the luminescence counting-rate from either of irradiated (42.9 Gy) samples MOR or LR was similar to the background showing that the luminescence in Figs. 6 and 7 should be from quartz rather than feldspar.

5. Discussion

Luminescence lifetimes in quartz are known to be affected by the thermal history of the quartz (Chithambo, 2002 and Chithambo, 2003b; Galloway, 2002) as is the sensitivity of OSL from quartz (Bøtter-Jensen et al., 1993, Bøtter-Jensen et al., 1995 and Galloway, 2002). It should therefore be expected that the OSL properties of quartz from rocks including luminescence lifetimes should also be affected by the thermal provenance of the source rocks. Although both the

age and denudation of the rocks may be relevant, characteristics of luminescence lifetimes of quartz from rocks should be explained by the same models that account for the dependence of lifetimes in quartz on high-temperature annealing such as that suggested by [Galloway \(2002\)](#).

The energy band diagram of [Galloway \(2002\)](#) with a slight modification is shown in [Fig. 8](#). The energy levels shown represent shallow traps (ST), optically sensitive traps (OST), and deep traps (DT) which cannot be optically stimulated. The additional level labelled STE represents self-trapped excitons formed during irradiation ([Song et al., 2000](#)) but stable at room temperature during measurement. The STE level also represents other trapping processes which like the DT tend to reduce the amount of electrons available for luminescence. In this model, built on that of [Bøtter-Jensen et al. \(1995\)](#) and [Zimmerman \(1971\)](#), the influence of annealing on lifetimes can be explained by the transfer of holes during annealing from a non-luminescent center R to luminescence centers L_a and L_b as well as from L_a to L_b . Values of luminescence lifetimes will then depend on whether luminescent recombination occurs at L_a or L_b . For example, luminescence lifetimes in sedimentary quartz (BDH Ltd, UK) which are independent of annealing below 500 °C, decrease from about 40 down to 31 μs when the annealing temperature is increased from 500 to 800 °C, that is, from τ_a to τ_b ([Galloway, 2002](#)). In general, previous work has shown that lifetimes measured at room temperature in quartz have one dominant lifetime whose precise value depends mainly on irradiation dose, annealing temperature, preheating temperature, and temperature during stimulation ([Bailiff, 2000](#) and [Bailiff and Mikhailik, 2003](#); [Chithambo, 2002](#), [Chithambo, 2003a](#) and [Chithambo, 2003b](#); [Galloway, 2002](#)).

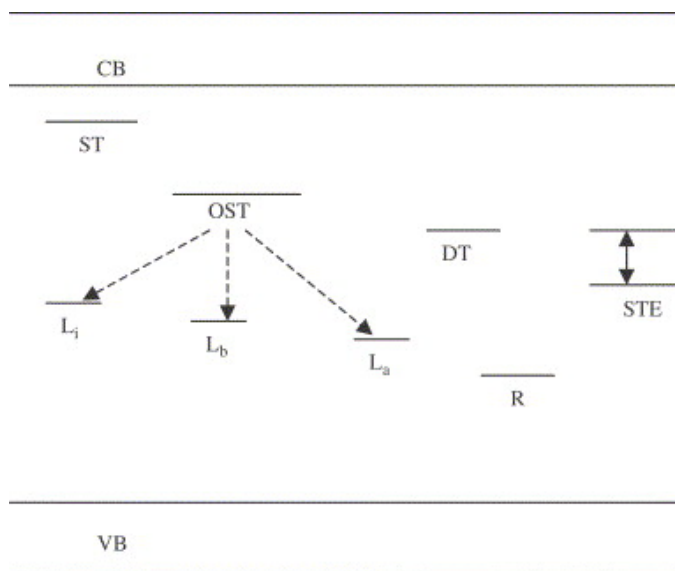


Fig. 8. The model of [Galloway \(2002\)](#) applied to explain luminescence lifetimes in quartz. ST represents shallow electron traps; OST, optically sensitive traps and DT denote deep traps which

are not optically stimulated. The deep traps reduce the electron concentration available for luminescence as does the self-trapped excitons (STE) additional in this case to the model of Galloway (2002). Levels R , L_a , L_b and L_i are luminescence centers.

The luminescence lifetimes of quartz studied in this work are summarized in Table 2 and classified as long (τ_a), intermediate (τ_i) and short lifetime (τ_b) components, respectively. In terms of the model of Fig. 8, the long lifetime component, τ_a , is associated with the L_a center and the short lifetime component, τ_b , with the L_b center.

Table 2.

Summary of luminescence lifetimes of quartz

Sample	Occurrence	τ_a	τ_i	τ_b
		(μs)	(μs)	(μs)
B7913	Vein, Hydrothermal	100 \pm 23		2.6 \pm 0.4
TAR1	River Cobble, Metamorphic	86.4 \pm 4.3		
FJ2R	River Cobble, Metamorphic	75.9 \pm 4.7		
FJ2V	River Cobble, Metamorphic	86.2 \pm 15.6		
MOR	River Cobble, Plutonic		17.9 \pm 1.6	
LR	River Cobble, Plutonic			4.0 \pm 0.5
MR1	River Cobble, Plutonic			
B9060	Host rock, Volcanic		36.7 \pm 7.2	

Lifetimes comparable to the $2 \mu\text{s}$ dwell time are too rapid to have been resolved accurately and should be regarded with caution.

Table 2 shows that the principle lifetime component in metamorphic as well as hydrothermal quartz is τ_a . Otherwise, the value of lifetime does not systematically depend on type of quartz since the short lifetime component is found in hydrothermal quartz, and plutonic quartz (sample LR) which also has an intermediate lifetime component τ_i (sample MOR) as has the volcanic quartz. Qualitative evidence suggest that the short lifetime is also present in metamorphic quartz but the associated data was too sparse to allow reliable quantitative fitting. Since in practice, luminescence will be emitted from any or all luminescence centers, the presence of a dominant lifetime indicates

preferential luminescent recombination at one center depending on the thermal history of the sample and so the hole concentration of the center.

Finally, incidences of an intermediate lifetime, in this case of value between 20 and 30 μs as in samples MOR and B9060 are less easy to explain. However, previous measurements (Galloway, 2002) found that for certain combinations of annealing temperature and temperature during stimulation there was a third lifetime of value about 18 μs which could be attributed to the presence of a third luminescence center. It seems plausible to assume that if the intermediate lifetime components seen in this work are not of type τ_a , then they originate from luminescent recombination at the third center shown as L_i in Fig. 8.

6. Summary

Time-resolved luminescence of low sensitivity natural quartz from crystalline rocks has been studied. Measurements of luminescence were made using pulsed optical stimulation using 470 nm blue light on samples of plutonic, metamorphic, volcanic and hydrothermal quartz. All samples show evidence of a lifetime component shorter than 5 μs although in several cases too low in intensity to allow accurate quantitative fitting. On the other hand, the value of the principal lifetime component varies considerably being about 86 μs in metamorphic quartz, 18 μs in plutonic quartz, 36 μs in volcanic quartz, and 100 μs in hydrothermal quartz. It is expected that the thermal provenance of the source rock has a direct bearing on precise value of the dominant lifetime measured. In particular, the presence of a dominant lifetime is explained in terms of preferential luminescent recombination at one luminescence center.

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