



# Low temperature thermochronology using thermoluminescence signals from quartz



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## HIGHLIGHTS

- The luminescence signals from quartz were studied for low temperature thermochronology.
- Thermoluminescence (TL) and isothermal thermoluminescence (ITL) signals/protocols were investigated.
- SAR-ITL and MAR-TL protocols were proposed for thermochronological study using rock samples.

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## ABSTRACT

Isothermal thermoluminescence (ITL) and thermoluminescence (TL) signals from quartz were studied. A single aliquot regenerative dose protocol has been applied for ITL  $D_e$  determination (SAR-ITL). In the SAR-ITL protocol, the preheat condition was a cutheat to 10 °C higher than measurement temperature. The test dose was approximate to the expected  $D_e$ , and a 450 °C heat was given at end of each cycle to minimize signal build-up. Based on signals strength and dose recovery test, temperatures of 235 and 255 °C were selected for the ITL  $D_e$  measurement. A multiple aliquots regenerative protocol has been applied for TL  $D_e$  determination (MAR-TL). The preheat procedure was a cutheat of 235 °C and a second glow TL of 175 Gy was used for normalization. The sensitivity change of first heating to 450 °C was negligible, supported by comparison between additive and regenerative dose growth curves. Based on the natural TL signal and preheat condition studies,  $D_e$  values at temperatures of 250–330 °C were used for thermochronological study. These two protocols were applied to rock samples collected at different elevations from Nujiang River (also called Salween River) valley slope. The SAR-ITL gave  $D_e$  results consistent with the MAR-TL at temperatures of 40–50 °C higher. The results clearly demonstrate the differences in the thermal histories between the analyzed samples. The SAR-ITL and MAR-TL protocols were both found to be suitable for application in thermochronology.

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## 1. Introduction

Luminescence dating has been developed as a useful archaeological and geological dating tool since the 1960s (Aitken et al., 1964). For burnt materials like pottery, burnt flint, burnt stone and volcanic lava, thermoluminescence (TL) dating is typically applied. For sedimentary materials like loess, beach dunes, colluvial deposits, fluvial and lacustrine sands, optically stimulated luminescence (OSL) dating is applied. For these materials, the luminescence clock is set to zero before being buried or preserved. Many dating protocols have been established, such as multiple aliquot

additive dose thermoluminescence (MAA-TL) protocol (Aitken, 1985), multiple aliquot regenerative dose thermoluminescence (MAR-TL) protocol (Aitken, 1985), single aliquot regenerative dose optically stimulated luminescence (SAR-OSL) protocol (Murray and Wintle, 2000a, 2003; Li et al., 2002; Li and Li, 2011) and single aliquot regenerative dose isothermal thermoluminescence (SAR-ITL) protocol (Jain et al., 2005; Tribolo and Mercier, 2012).

The thermoluminescence signal is temperature sensitive by definition (Johnson, 1966). It has been used as a low temperature thermochronological method for non-burnt rock samples that have experienced cooling processes of exhumation. The fundamental principles, theoretical formulas and numerical simulation have been studied over last 20 years (e.g. Prokein and Wagner, 1994; Herman et al., 2010; Li and Li, 2012). The luminescence dating has lower closure temperatures and suitable for an age dating range

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within 1 Myr (Dodson, 1973; Li and Li, 2012). With these features, the luminescence could be a very powerful tool to research the low temperature zone beyond the established thermochronological techniques, e.g.  $^{40}\text{Ar}/^{39}\text{Ar}$ , fission track and (U–Th)/He methods. It can therefore be used to determine the instantaneous reaction response to abrupt and rapid crust uplift, such as river incision, glacial denudation and normal faulting. In contrast, most of the established thermochronological techniques, such as  $^{40}\text{Ar}/^{39}\text{Ar}$ , fission track and (U–Th)/He dating can only measure the average rate of regional exhumation in the order of tens of Myr, which is much slower than the true uplift rate of the crust.

Numerous previous studies of rock samples have indicated that luminescence signals are dependent on thermal history (Nambu et al., 1996; Han et al., 1997; Tsuchiya et al., 2000; Herman et al., 2010; Li and Li, 2012). Different from ambient temperature condition, the equivalent dose of a cooling system can be expressed as  $(dD_e/dt) = D_r - (D_0 \cdot s / e^{E/kT})(e^{D_e/D_0} - 1)$  where  $D_e$  is the equivalent dose (Gy),  $D_0$  (Gy) is the characteristic dose of saturation,  $D_r$  (Gy/ka) is the dose rate of radiation, temperature  $T$  (K) is a function of time  $t$  (ka),  $E$  (eV) is the activation energy of the traps of interest,  $s$  is the frequency factor described the attempt to escape frequency in  $\text{s}^{-1}$ , and  $k$  is the Boltzmann constant (Li and Li, 2012). The quotients that  $D_e$  divided by dose rate were considered as apparent ages, because  $D_e$  is a function of  $T$ ,  $t$  and  $D_r$ . For a luminescence signal to be used to investigate rock thermal history, the following work has to be carried out. 1) Identifying a bright enough luminescence signal. 2) Finding an appropriate protocol for  $D_e$  measurement. 3) Studying rock microdosimetry for the annual dose estimation. 4) Obtaining the  $D_e$  values and apparent ages that correspond to the closure temperatures. 5) Deriving a cooling rate based on the determined trap parameters ( $E$ ,  $s$  and kinetic orders).

In this paper, we aim to investigate suitable protocols through fundamental and systematical experimental study. SAR-ITL and MAR-TL protocols were studied for thermochronology of rocks.

## 2. Samples and equipment

Three rock samples were collected from a “V” shape valley slope of the Nujiang River (Salween River), and named from top to bottom as FG-A, FG-B and FG-C. They are mylonite (FG-A), schist (FG-B) and gneiss (FG-C) and contain abundant quartz, and have experienced rapid cooling in recent geological history due to the river incision.

The raw samples were sawed using rock cutting machinery and crushed by hand hammer gently to maintain the original minerals size. After dry sieving, 150–180  $\mu\text{m}$  grain size range was obtained. Quartz grains were separated from bulk mineral grains by heavy liquid density separation at 2.62–2.75  $\text{g cm}^{-3}$ . They were then etched by 40% HF for 1 h to remove the outside layer compromised by alpha particles and remaining feldspar grains. All preparations were performed under fluorescent lamp or dim red light.

$D_e$  measurements were conducted with a TL/OSL DA15 Risø reader. It is equipped with an EMI Q9235 photomultiplier tube with three 2.5 mm Hoya -U340 filters attached in front for detection in the UV wavelength range (around 340 nm). A  $^{90}\text{Sr}/^{90}\text{Y}$  beta source was used for irradiations. The heating rate was 5  $^\circ\text{C/s}$  for all experiments. The purity of quartz grains was tested by monitoring the presence of feldspar through measuring the infrared stimulated luminescence (IRSL) (Duller, 2003) and 110  $^\circ\text{C}$  thermoluminescence (TL) peak (Li et al., 2002). Unless specified, all the ITL and TL experiments used six aliquots of 5 mm diameter for each data point in the measurement.

Three different signals, OSL, ITL and TL, were examined to identify sufficiently bright signals. The OSL signal under blue light stimulation (six clusters of LEDs,  $470 \pm 20$  nm) was not detected for

some rocks. The ITL and TL signals are strong enough in all of our rocks samples (Fig. 1).

## 3. SAR-ITL

The SAR-ITL signal at 310–330  $^\circ\text{C}$  has been used for the dating of sediments in previous studies (Choi et al., 2006; Huot et al., 2006). However, in this study lower heating temperatures were chosen because they are temperature sensitive and correspond to lower thermal stability. These would record the most recent cooling processes before the equilibrium state or signal saturation was reached. Sample FG-A was used in these experiments.

The natural TL signal of sample FG-A was detected at temperatures starting from 235  $^\circ\text{C}$ . The natural signal of ITL at 235  $^\circ\text{C}$  was used for experimentation. The preheat conditions (Fig. 4A, steps 2 and 5) of the 235  $^\circ\text{C}$  ITL signal were varied to identify a bright enough signal and study the effect on  $D_e$  value. Three preheating conditions (a preheat for 10 s at 235  $^\circ\text{C}$ , a cutheat to 235  $^\circ\text{C}$  and a cutheat to 245  $^\circ\text{C}$ ) were examined for SAR-ITL at 235  $^\circ\text{C}$ . The results are displayed in Fig. 2A–C. After preheat at 235  $^\circ\text{C}$  for 10 s, both the natural and regenerative signal were removed. In the case of cutheat 235  $^\circ\text{C}$ , the first 10 s signal of the regenerative dose was much higher than the natural signal, indicating that the existence of a less stable signal in the regenerative signals. In the case of a cutheat to 245  $^\circ\text{C}$ , the natural and regenerative signals overlapped each other. The ratio between them was consistent with ITL heating time except for the first 10 s. The ratio between natural and regenerative signals increases during the first 10 s; thermal lag lengthen the time needed for a disk to reach thermal equilibrium. The cutheat to 235  $^\circ\text{C}$  resulted in a remarkable higher initial signal for the regenerative signals, while the cutheat to 245  $^\circ\text{C}$  gave an identical result between natural and regenerative signals. The cutheat to 10  $^\circ\text{C}$  higher was used as the preheating in this study.

Different test doses (Fig. 4A, step 4) were studied to evaluate the effect on ITL  $D_e$  and to identify an appropriate test dose value. Three different test doses, 25, 145 and 250 Gy were tested for SAR-ITL at

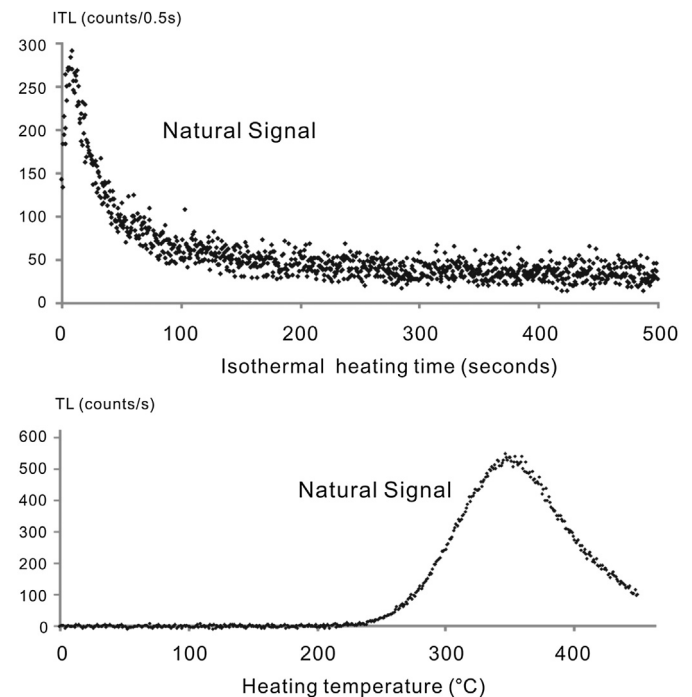


Fig. 1. Typical ITL and TL signal (from sample FG-A).

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