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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 De 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 De, 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 De measurement. A multiple aliquots regenerative protocol has been applied for TL De 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, De 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 De results consistent with the MAR-TL at temperatures of 40-50°Chigher. 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.

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°Chigher. 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.

Key words: Thermochronology; Luminescence; ITL; TL; Quartz; Protocol

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 a lower closure temperatures and suitable for an age dating range within 1 Myr (Dodson, 1973). With these features, the luminescence could be a very powerful tool to research the low temperature zone beyond the established thermochronological techniques, e.g. $^{40}Ar/^{39}Ar$, fission tracks 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 $^{40}Ar/^{39}Ar$, 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 $\frac{dD_e}{dt} = D_r - \frac{D_0 \cdot s}{e^{E/kT}}(e^{\frac{D_e}{D_0}} - 1)$ 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 second^{-1}, and k is the Boltzmann constant (Li and Li, 2012). The quotients that D_e divided by annual dose 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 um grain size range was obtained. Quartz grains were separated from bulk mineral grains by heavy liquid density separation at 2.62-2.75 $g.cm^{-3}$. They were then etched by 40% HF for 1 hour 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 $^{90}Sr/^{90}Y$ beta source was used for irradiations. The heating rate was 5 °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 °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±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 °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 °C. The natural signal of ITL at 235 °C was used for experimentation. The preheat conditions (Fig. 4A, steps 2 and 5) of the 235 °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 10s at 235 °C, a cutheat to 235 °C and a cutheat to 245 °C) were examined for SAR-ITL at 235 °C. The results are displayed in Fig. 2A, B and C. After preheat at 235°C for 10s, both the natural and regenerative signal were removed. In the case of cutheat 235 °C, the first 10s 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 °C, the natural and regenerative signals overlapped each other. The ratio between them was consistent with ITL heating time except for the first 10s. The ratio between natural and regenerative signals increases during the first 10s; thermal lag lengthen the time needed for a disk to reach thermal equilibrium. The cutheat to 235 °C resulted in a remarkable higher initial signal for the regenerative signals, while the cutheat to 245 °C gave an identical result between natural and regenerative signals. The cutheat to 10 °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 235 °C on sample FG-A (Fig. 2D). Consistent D_e

values of 153.2 ± 25.3 Gy and 147.2 ± 8.1 Gy were obtained by using test doses of 145 and 250 Gy, respectively. The test dose of 25 Gy gave a D_e value 20 percent smaller. This indicates that a test dose of 145 Gy or larger is suitable for sample measurement and a test dose approximate to that of the expected D_e should be used for the measurement. The D_e values obtained using a test dose of 250 Gy had smaller errors than that of 145 Gy. However, it is time consuming on the measurements.

Different thermal wash procedures (Fig. 4A, step 7) were applied at the end of each cycle to remove signal build-up. Three different heating temperatures, 0, 350 and 450 °C were examined to evaluate the effect of signal build-up using sample FG-A (Fig. 2E). D_e values increase as the thermal wash temperature increases, which is attributed to the lower signal build-up as a result of the higher cleaning temperature. A heating of 450 °C has cleaned the remnant signals thoroughly, according to a second TL measurement to 450 °C. This result indicated that a 450 °C cycle heat is appropriate.

Different ITL heating temperatures correspond to the thermal chronometers of different closure temperatures (except for a single TL peak covering a wide temperature range). Five ITL heating temperatures of 215, 235, 255, 275 and 295 °C were examined using sample FG-A, where the natural D_e values were estimated using SAR-ITL (Fig. 4A, steps 3 and 6). A cutheat to 10 °C higher than ITL temperature was used as a preheat. The aliquots were then heated to 500s to obtain the ITL signal.

The first 10-20s and last 50s were integrated as the signal and background respectively. A test dose approximate to the expected D_e was used and a 450 °C heat was applied at the end of each cycle. For dose recovery tests, aliquots were heated for 1 hour at 500 °C, and then were given artificial doses similar to the estimated natural D_e. The measured ratios (measured/given doses) which decrease with ITL heating temperatures, were 0.99 ± 0.09 for 215 °C, 0.97 ± 0.08 for 235 °C, 0.97 ± 0.06 for 255 °C, 0.89 ± 0.04 for 275 °C, and 0.88 ± 0.04 for 295 °C. The results in Fig. 3 demonstrate that signal intensity increases with temperature, whilst the dose recovery ratio decreases. At 215 °C, although the dose recovery result was excellent, the natural signal was too weak to be measured. For 275 and 295 °C, the ITL signals were bright but the dose recovery tests showed D_e underestimations. Therefore, we used 235 and 255 °C as SAR-ITL measurement temperatures. The recuperation for ITL at 235 and 255 °Cwas 3.8% and 2.3% respectively and the recycling ratios were 1.10 ± 0.11 and 1.04 ± 0.05 respectively. This indicates that the 235 and 255 °C are appropriate as the SAR-ITL measurement temperature.

The measurement conditions of the SAR-ITL protocol were summarized in Fig. 4. Each cycle was composed of the seven steps. A thermal wash (Fig. 4A, step 7) is introduced to ensure no signal build-up after each measurement cycle (Tribolo and Mercier, 2012). The first 10-20s and last 50s are integrated as signal and background, respectively. Unless specified, all the SAR-ITL experiments used six aliquots of 5 mm diameter for each sample in the measurement. SAR-ITL results of three samples were shown in Table 1. For both 235 and 255 °C, the FG-B had the largest D_e (200.6 and 350.8 Gy), and FG-A had the second largest D_e (153.2 and 258.4 Gy) and FG-C had smallest D_e (48.9 and 200.7 Gy). The results demonstrated the differences of the thermal histories between samples. Neither positive nor negative correlation can be identified between the D_e values and elevations.

4. MAR-TL

The established SAR-ITL protocol is not perfect, because it is not appropriate for measuring temperature over 255°C due to poor recovery dose test. Samples with a large D_e would require long measurement periods using the SAR-ITL protocol. To obtain multiple signals at different temperatures and to optimize the measurement time, the MAR-TL protocol was explored. Sample FG-A was used for the study of experimental conditions.

An important issue of MAR-TL is whether the regenerative growth curve can represent the natural growth curve or not. To verify this, the growth curves of sample FG-A were built up using both additive dose and regenerative dose methods. For the additive dose method, six groups of six aliquots were given 0, 70, 140, 210, 280 and 350 Gy respectively. For the regenerative dose method, ten groups of six aliquots were given 70, 140, 210, 280, 350, 420, 490, 560, 630 and 700 Gy respectively, after an initial resetting at 450 °C. Signals were normalized by the 175 Gy regenerative second glows TL. The integrations of TL counts of 5 °C were plotted as a function of given doses in Fig. 5. The regenerative dose growth curve was translated by 250 Gy, such that it matched the observed additive dose growth curve (Prescott et al., 1993). The good similarity in growth curve shape, between additive and regenerated growth curves suggest that the thermal resetting did not induce a change in sensitivity in the regenerated growth (Mejdahl and Jensen, 1994; Tribolo and Mercier, 2012).

The natural TL signal of sample FG-A can be distinguished from backgrounds at temperatures higher than 235 °C. Three different cutheat temperatures (Fig. 4B, steps 1, 4 and 7), 215, 235 and 255°C were examined in the MAR-TL sequence. The D_e values are plotted against temperature using an integration of every 5 °C (D_e(T) plot) (Fig.6). For each D_e (T) plot, the D_e values at temperature lower than preheat temperature are not meaningful. The natural signals were partially or completely removed by preheating since these are at lower temperatures. Hence, the regenerated doses would dominate these temperature regions. The results in Fig. 6 indicate that D_e values are higher at TL temperatures 255-300 °C after a cutheat to 255 °C in comparison with cutheats of 215 and 235 °C. After cutheats of 215 and 235 °C, the D_e showed the same values with signals from TL temperatures greater than 235 °C. For the 300-350 °C, these three curves under different preheat conditions overlapped and formed one single curve, indicating that the different preheat conditions only has a limited effect on D_e values. Therefore a cutheat to 235 °C was used as the preheat condition. Still, D_e values of TL below 250 °C can be influenced by preheating. Blackbody radiation became significant above 330 °C. The

D_e values of TL from 250 to 330 °C were used in thermochronological study. The validity of the second glow normalization is evaluated by R-squared value of growth fitting (Fig. 4B, step 6). After the normalization, the adjusted R-squared value of fitting was 0.9833 in comparing the value of 0.3552 before normalized. Uncertainties due to the normalization procedure were included in the total equivalent dose uncertainties.

The MAR-TL protocol is summarized in Fig. 4B and the MAR-TL results are shown in Table 1. D_e values from 250 to 330 °C at increments of 10 °C are listed. Each D_e is calculated from 10°C integration on the lowest 5 and highest 5 degrees. Similar to the results of SAR-ITL, no systematic dependence of De is found on elevation. For the 250-330 °C, the FG-B had the largest D_e (76.9-530.3 Gy), and the FG-C had second largest D_e (63.3-494.8 Gy) and the FG-A had smallest D_e (49.2-475.0 Gy).

5. Discussions

5.1 Advantages vs. disadvantages

The SAR-ITL protocol has following advantages. 1) Only a small amount of materials is needed for a D_e measurement. 2) An inter aliquot normalization is not required. 3) The sensitivity changes which occur during repeated regeneration and measurement cycles can be corrected using the ITL induced by a test dose. 4) The ITL heating temperature is lower than conventional TL. The influence of the atmosphere of the TL ovenat high temperature is not critical for ITL (e.g. nitrogen; Aitken, 1985). 5) The blackbody radiation is lower during an ITL measurement compared to conventional TL. 6) The thermal stability and components of ITL signal can be assessed and separated by fitting the decay curves with multi-exponential components (Aitken, 1985; Huot et al., 2006; Tribolo and Mercier, 2012). There are however disadvantages when using the SAR-ITL protocol. 1) The ITL signal may artificially build up over measurement cycles if the residual signals are not properly reset. 2) A single aliquot protocol requires far longer amounts of measurement time compared to multiple aliquots. 3) Quartz from some rocks such as limestone and dolomite might have very weak ITL signals.

The MAR-TL has following advantages. 1) Multiple D_e values of different TL peaks can be measured in one run, with each TL peak represents a geothermometer. 2) Different TL peaks, representing thermal chronometers of different closure temperatures, could provide multiple stage information of cooling processes. 3) The kinetics parameters of TL signals have been well studied (Chen and Mckeever, 1997; Aitken, 1985; Hornyak et al., 1992). 4) A multiple aliquot TL method requires shorter machine time. 5) The thermal stability of TL signals can be identified from the temperature of TL glow curve (Aitken, 1985). There are however disadvantages. 1) The normalization between aliquots typically increase the dispersion in the growth curve. 2) A D_e value may be difficult to interpret if extracted from a temperature region that contains one or more overlapping TL peaks. 3) The influences of the atmosphere of the TL oven at high temperature need to be accounted for (e.g.

nitrogen; Aitken, 1985). 4) The blackbody radiation might influence the TL signal at high temperature (>400 °C).

5.2 Sensitivity change of SAR-ITL

The sensitivity change during the first heating of the SAR-ITL protocol has been reported to be a problem in the dating of sediment (Buylaert et al., 2006; Huot et al., 2006). Tribolo and Mercier (2012) identified two groups of burnt quartzites which behaved entirely differently in terms of sensitivity change; they used samples with little or no sensitivity change to determine their equivalent doses. In this study, all samples were evaluated by comparing the additive and regenerative dose TL growth curve (Prescott et al., 1993; Mejdahl and Jensen, 1994). Where growth curves overlapped after sliding, this indicated that sensitivity change after the first heating was not an issue for the samples (Fig. 5). For ITL, because the heating temperatures were relatively low, the first heat sensitivity change is less problematic compared to a TL measurement. It is therefore concluded that the SAR-ITL and MAR-TL protocols used in this study have negligible effects of sensitivity changes (Tribolo and Mercier, 2012).

5.3 Comparison between SAR-ITL and MAR-TL

The results from the SAR-ITL and MAR-TL protocols should yield the same D_e(T) plots, which correspond to TL peaks as well as a thermal chronometer. The ITL signal of a certain temperature originates from the traps which correspond to TL peaks at higher temperatures. Murray and Wintle (2000b) have reported the 330 °C ITL signal came from 375 °C TL peak. The D_e values of SAR-ITL appear to be shifted compared with the D_e(T) plots of MAR-TL. A 40-50 °C translation is needed to overlap the D_e values obtained from SAR-ITL and MAR-TL. This is consistent with the fact that an ITL measurement probe trapped elections held at a deeper TL temperature (Murray and Wintle, 2000). Though, it doesn't apply to all D_e values. The FG-C SAR-ITL D_e of 48.9 ± 6.9 Gy appears to be lower than the value expected from MAR-TL after 40 °C shift. They overlapped well with a 25 °C shift. This temperature differences might be sample dependent and dictated by unique character of these quartz samples. Since the traps depth energy (E) increases with the measuring temperature, D_e values determined under different temperatures are different. Here, no D_e(T) plateau could be obtained (Fig.6). A plateau would be present only if the TL glow curve was dominated by a single TL peak, within a temperature range.

6. Conclusion

The SAR-ITL and MAR-TL protocols were studied and applied to rock samples collected from the Nujiang River valley slope. For SAR-ITL, the preheat procedure had a 10 °C higher cutheat than measuring temperature. The test dose was approximate to the anticipated D_e, and a 450 °C thermal wash was applied at the end of each measurement cycle. SAR-ITL measured at 235 and 255 °C have been shown to give reliable results. For MAR-TL, we did not observed any sensitivity change after the initial thermal resetting. Results from both SAR-ITL and MAR-TL demonstrated differences between the thermal histories of the samples. The D_e values from SAR-ITL and MAR-TL overlapped after a 40-50 °C. SAR-ITL and MAR-TL are appropriate for thermochronological studies.

7. Acknowledgement

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Figure caption:

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

Fig. 2: A, B and C: ITL signals at 235 °C with different preheat conditions. The primary y-axis shows the ITL counts and the secondary y-axis shows the ratio between natural and regenerative signals. D: D_e plot of SAR-ITL at 235 °C against test dose. The dashed line shows the mean of D_e values by using test doses of 145 and 250 Gy, and the yellow shaded region shows the standard deviation.E: De plot of SAR-ITL at 235 °C against cycle clear-up temperatures.

Fig. 3: SAR-ITL natural signals at heating temperatures of 215, 235, 255, 275, 295 °C.

Fig. 4: SAR-ITL and MAR-TL protocol.

Fig. 5: A comparison between additive and regenerative dose growth curve of TL signal. The TL counts were normalized by 140 Gy second glow TL signals.

Fig. 6: D_e(T) plot of MAR-TL results by 215, 235, 255 °C cutheat.The D_e values were calculated by interpolating the mean natural TL signals of six aliquots onto a regenerated growth curve, from TL signals of six aliquots.

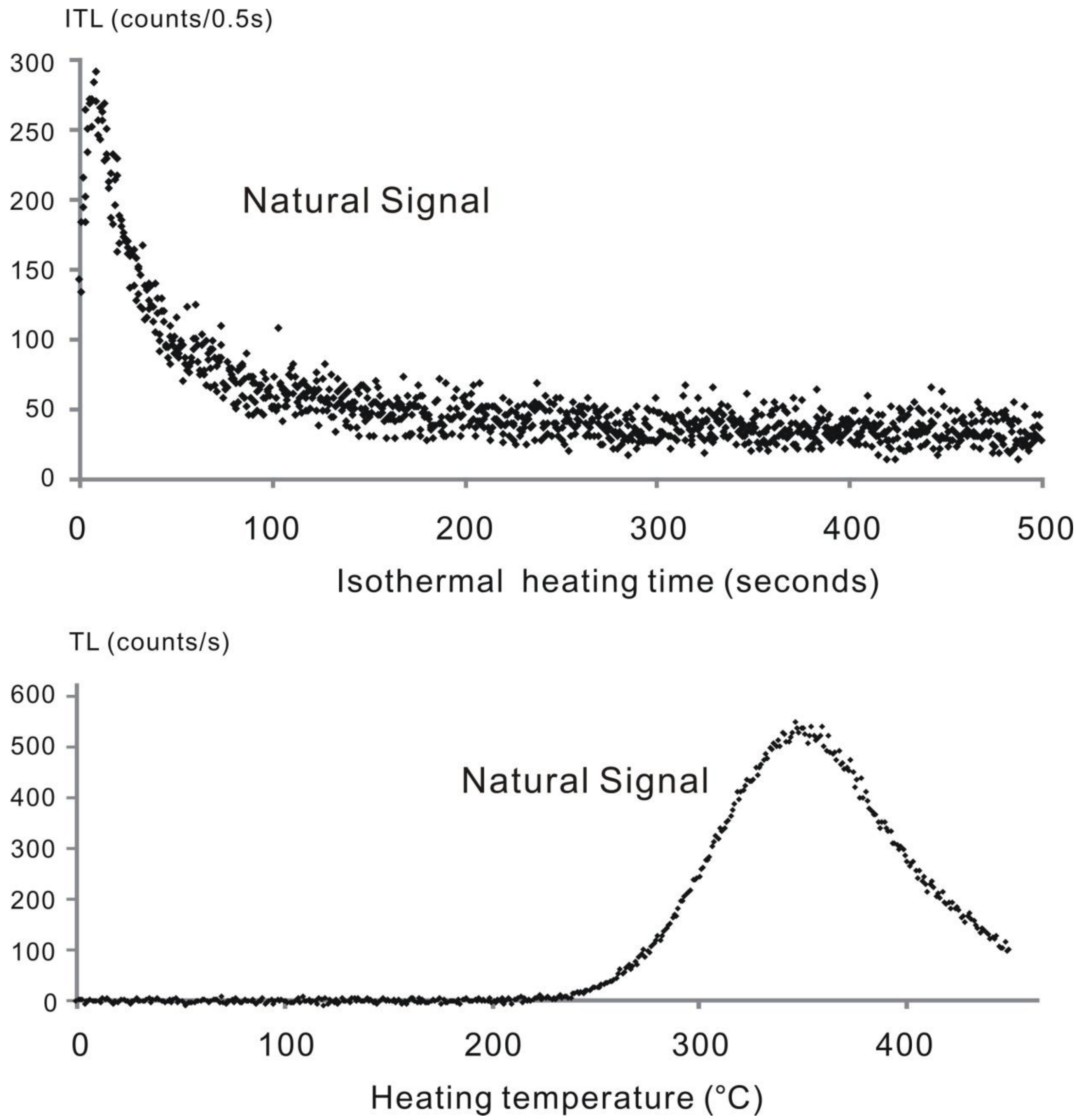
ITL (counts/0.5s)
300
250
200
150
100
50
0
Natural Signal
0
100
200
300
400
500
Isothermal heating time (seconds)
TL (counts/s)
600
500
400
300
200
100
0
Natural Signal
0
100
200
300
400
Heating temperature (°C)

Fig. 1

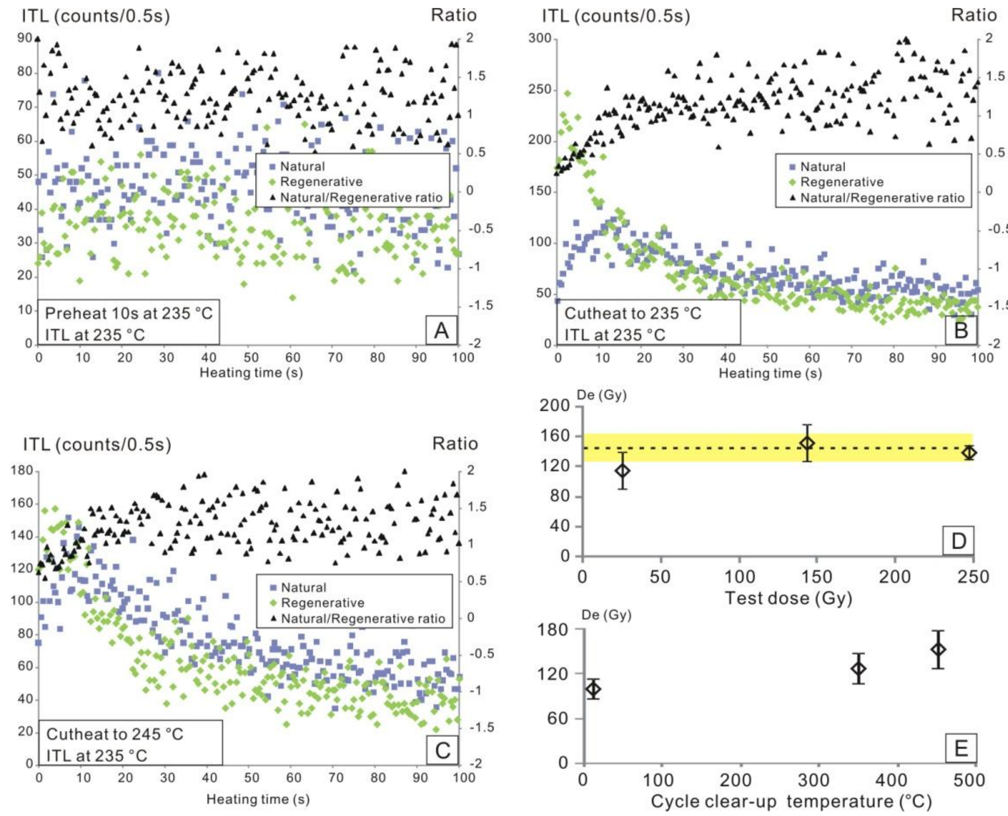
ITL (counts/0.5s)
Ratio
Natural
Regenerative
Natural/Regenerative ratio
Preheat 10s at 235 °C
ITL at 235 °C
A
Heating time (s)
Cutheat to 235 °C
ITL at 235 °C
B
Cutheat to 245 °C
ITL at 235 °C
C
De (Gy)
Test dose (Gy)
D
Cycle clear-up temperature (°C)
E

Fig. 2

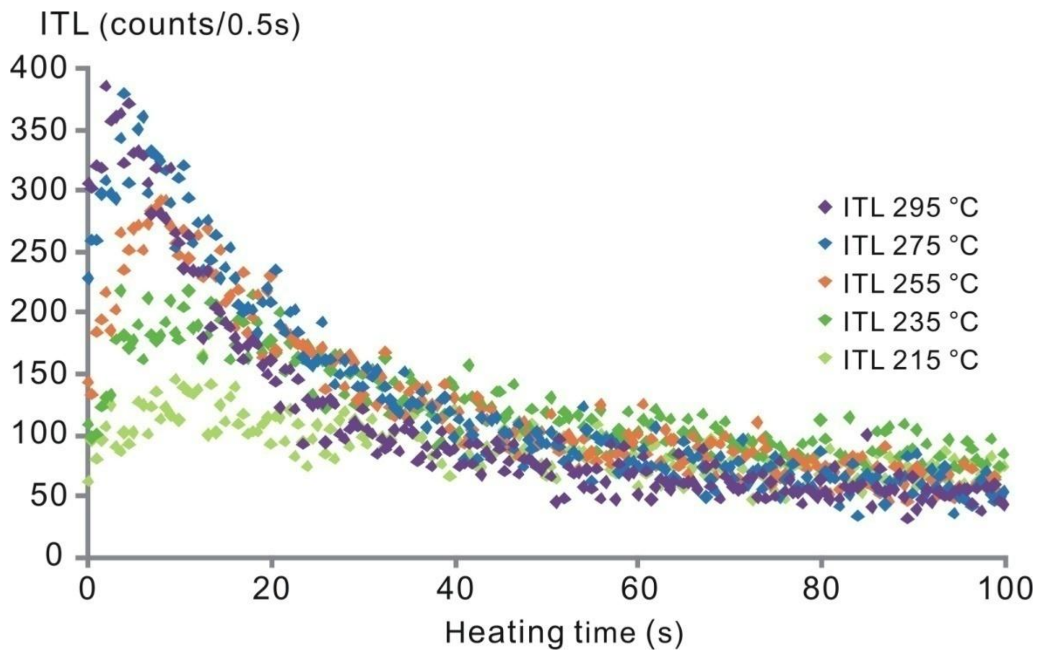
ITL (counts/0.5s)
400
350
300
250
200
150
100
50
0
0
20
40
60
80
100
Heating time (s)
ITL 295 °C
ITL 275 °C
ITL 255 °C
ITL 235 °C
ITL 215 °C

Fig. 3

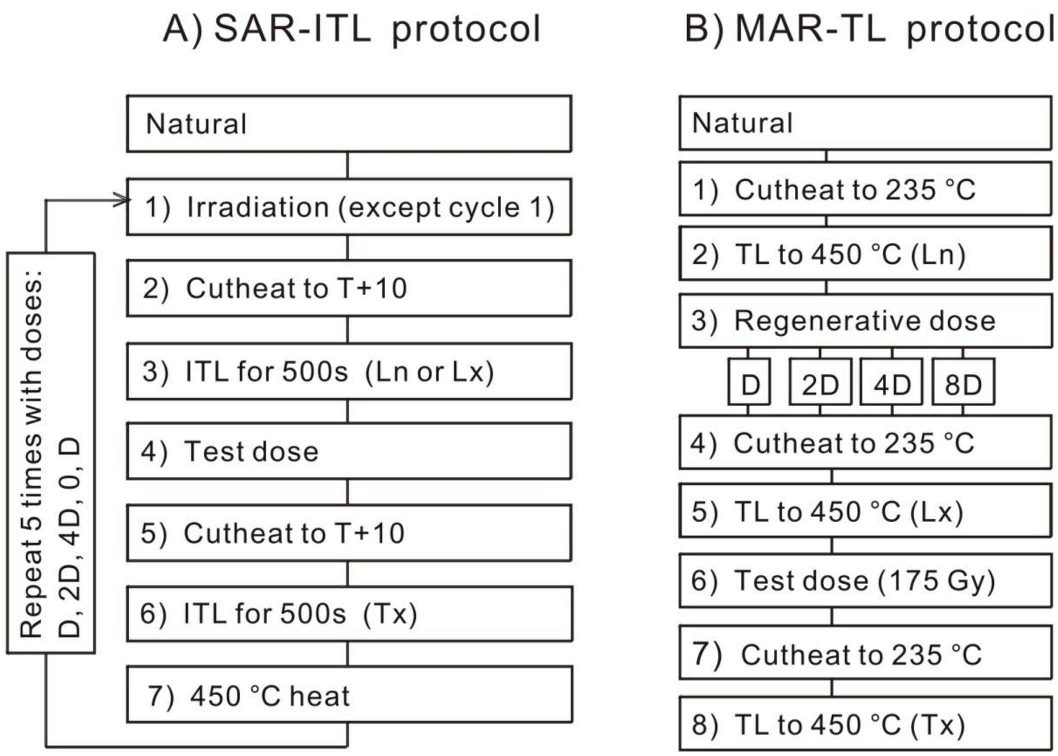
A) SAR-ITL protocol
Natural
1) Irradiation (except cycle 1)
2) Cutheat to T+10
3) ITL for 500s (Ln or Lx)
4) Test dose
5) Cutheat to T+10
6) ITL for 500s (Tx)
7) 450 °C heat
Repeat 5 times with doses:
D, 2D, 4D, 0, D
B) MAR-TL protocol
Natural
1) Cutheat to 235 °C
2) TL to 450 °C (Ln)
3) Regenerative dose
D
2D
4D
8D
4) Cutheat to 235 °C
5) TL to 450 °C (Lx)
6) Test dose (175 Gy)
7) Cutheat to 235 °C
8) TL to 450 °C (Tx)

Fig. 4

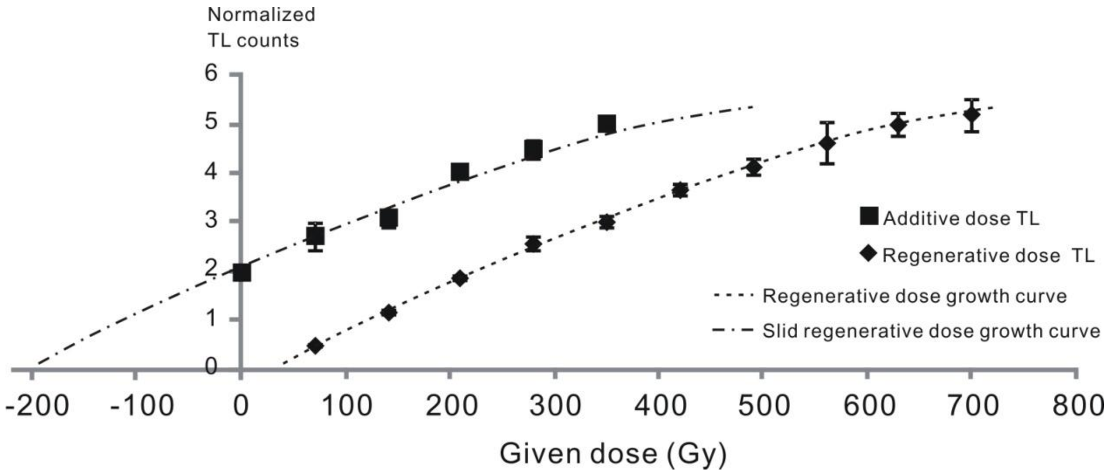
Normalized
TL counts
6
5
4
3
2
1
0
-200
-100
0
100
200
300
400
500
600
700
800
Given dose (Gy)
Additive dose TL
Regenerative dose TL
Regenerative dose growth curve
Slid regenerative dose growth curve

Fig. 5

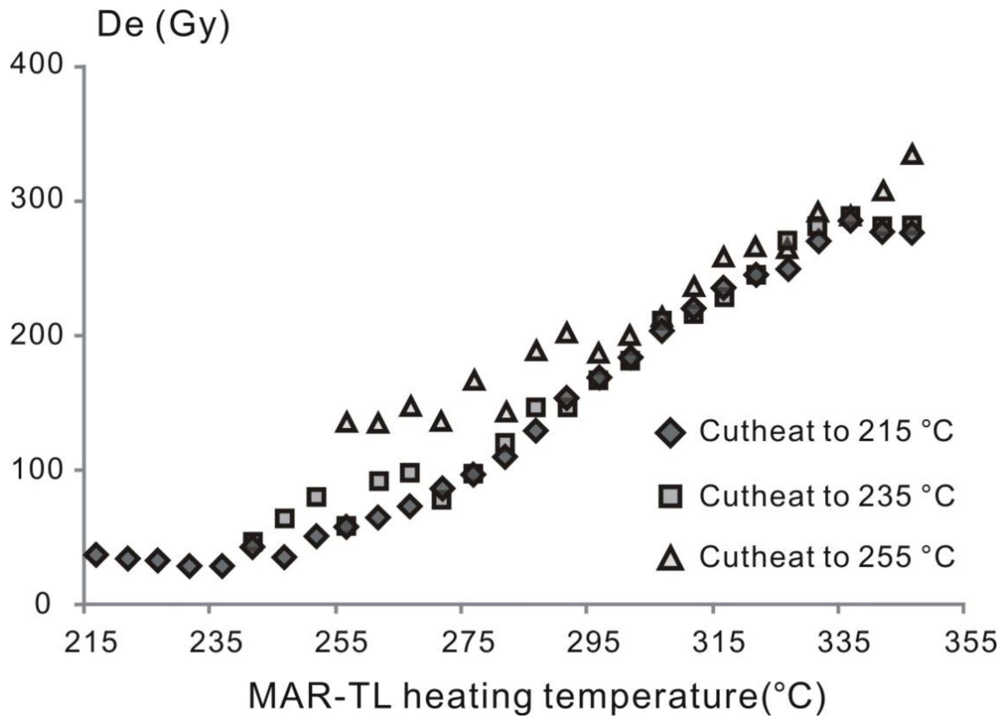
De (Gy)
400
300
200
100
0
215
235
255
275
295
315
335
355
MAR-TL heating temperature(°C)
Cutheat to 215 °C
Cutheat to 235 °C
Cutheat to 255 °C
Fig. 6

Table 1 The results of SAR-ITL and MAR-TL

Sample	De of SAR-ITL (Gy)		De of MAR-TL (Gy)								
	At 235 °C	At 255 °C	At 250 °C	At 260 °C	At 270 °C	At 280 °C	At 290 °C	At 300 °C	At 310 °C	At 320 °C	At 330 °C
FG-A	153.2 ±25.3	258.4 ±19.6	49.2 ±11.4	66.4 ±13.7	93.0 ±12.0	127.4 ±14.7	166.6 ±14.1	220.5 ±14.6	273.7 ±14.7	338.7 ±23.3	387.7 ±39.1
FG-B	200.6 ±19.2	350.8 ±30.1	76.9 ±18.0	102.6 ±20.1	146.5 ±27.2	204.1 ±32.9	271.1 ±41.0	337.9 ±47.5	394.5 ±51.0	451.3 ±60.7	476.1 ±68.0
FG-C	48.9 ±6.9	200.7 ±28.2	63.3 ±19.5	79.4 ±21.5	104.1 ±27.6	124.6 ±34.5	176.8 ±52.5	230.1 ±69.1	305.2 ±84.5	355.4 ±104.5	413.7 ±116.3