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1 Further studies on the relationship between IRSL and BLSL at

2 relatively high temperatures for potassium-feldspar from sediments

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11 Abstract:

In optical dating of potassium-feldspar, the luminescence signals can be stimulated 12 by both infrared (IR) light and blue light (BL). To develop reliable dating methods 13 using different stimulation light sources for feldspars, it is important to understand the 14 sources of the traps associated with the infrared stimulated luminescence (IRSL) and 15 blue light stimulated luminescence (BLSL) and their relationship. In this study, we 16 explored the luminescence characteristics of IRSL and BLSL at different stimulation 17 temperatures (from 60 °C to 200 °C) and their relationship based on five sets of 18 experiments, i.e. post-IR BLSL, post-BL IRSL experiments, pulse annealing test, dose 19 response test and laboratory fading rate test. Our results suggest that the luminescence 20 characteristics of IRSL and BLSL and their relationship are dependent on stimulation 21 temperature. For IR stimulation at a relatively high temperature of 200 °C, at least two 22 components of IRSL signals are involved in the process. One component of IRSL 23 signals can be easily bleached by BL stimulation at 60 °C, while the other is relatively 24 25 hard to be bleached by BL stimulation at 60 °C. The two components have different luminescence properties, such as thermal stability, dose response and laboratory fading 26 27 rate.

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31 1. Introduction

Both quartz and potassium-rich feldspar (K-feldspar) have been widely used as 33 natural dosimeters for optically stimulated luminescence (OSL) dating (Aitken, 1998). 34 Compared with quartz OSL, the infrared stimulated luminescence (IRSL) signal from 35 K-feldspar (Hütt et al., 1988) has advantages of much brighter luminescence signals 36 and much higher dose saturation level, making feldspar as an attractive candidate for 37 luminescene dating of the natural sedimentary samples. However, the usage of 38 39 K-feldspar for dating has long been hindered by the anomalous fading of the trapped charges related to the IRSL signals (e.g. Spooner, 1994; Huntley and Lamonthe, 2001; 40 Li and Li, 2008). 41

More recently, progress in understanding anomalous fading in feldspar has raised 42 the prospect of isolating a non-fading component from the IRSL at relatively high 43 temperatures (Thomsen et al., 2008; Li, 2010; Jain and Ankjærgaard, 2011; Li and Li, 44 2013). Correspondingly, a two-step post IR IRSL (pIRIR) protocol (Buylaert et al., 45 2009; Thiel et al., 2011) and a multi-elevated-temperature post-IR IRSL (MET-pIRIR) 46 47 protocol (Li and Li, 2011a) have been proposed to overcome anomalous fading for dating K-feldspar from sediments, which offer the promising potential for extending 48 the luminescence dating limit (Thiel et al., 2011; Li and Li, 2012; Li et al., 2013, 49 However, the high temperature pIRIR signals (e.g. >200 °C) isare found to be more 50 difficult to bleach than the IRSL signals measured at lower temperatures (Li and Li, 51 2011a; Buylaert et al., 2012; Murray et al., 2012), and it usually requires up to several 52 hours or even days of exposure to sunlight or a solar simulator to bleach the pIRIR 53 signals down to a stable level (here the term "bleach" means to reduce the 54 luminescence intensity by optical stimulation). For some samples, a significant 55 non-bleachable (or residual) component in the pIRIR signals was left even after a 56 prolonged bleaching period using solar simulator or sunlight (Buylaert et al., 2011; 57 Lowick et al., 2012; Chen et al., 2013; Li et al., 2014b). These studies suggest that the 58 IRSL signals recorded at relatively high temperature have different luminescence 59 behavior compared with the IRSL signals at room temperature. 60

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There have been several studies conducted to explore the relationship between

62 luminescence with IR stimulation and luminescence with visible wavelength light stimulation. It was demonstrated that the majority of green light stimulated 63 luminescence (GLSL) can be bleached by prolonged IR light and an upper limit of \sim 64 90% GLSL was depleted as a result of IR bleaching at room temperature (Duller and 65 Bøtter-Jensen, 1993; Galloway, 1994). Jain and Singhvi (2001) concluded that the 66 blue-green (BG) stimulated luminescence measured at 125 °C is associated with at 67 least two trap populations. One trap population is responsive to both IR stimulation 68 and BG stimulation. Another trap population is only responsive to BG stimulation. 69 Gong et al. (2012) conducted a study on the relationship between the infrared 70 stimulated luminescence (IRSL) and blue light stimulated luminescence (BLSL) at 71 60 °C. They observed that most of the IRSL signals at 60 °C can be bleached by BL at 72 60 °C, while the BLSL signals at 60 °C can only be partially bleached by IR at 60 °C. 73 The sources for the IRSL at 60 °C are mainly associated with the fast and medium 74 components of the BLSL at 60 °C. 75

In this study, in order to better understand the sources of the traps associated with the IRSL and BLSL, we further explore the relationship between IRSL and BLSL using K-feldspar from two aeolian sand samples. The luminescence properties, in terms of thermal stability, dose response and laboratory fading rate, are also examined for the different IRSL components at a relatively high temperature of 200 °C.

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82 2. Samples and equipment

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Two aeolian sand samples (HSDK-11 and SY) from the Hunshandake desert in northeast China were used in this study. Both samples have been investigated in previous studies (Li et al., 2002; Gong et al., 2013). The samples are from the same environmental settings of the same region and have similar luminescence behaviors, so the experimental results obtained from them should be comparable. The samples were treated with 10 % hydrochloric acid (HCl) and 10 % hydrogen peroxide (H₂O₂) to remove carbonate and organic matter, respectively, in subdued red light in the Luminescence Dating Laboratory, the University of Hong Kong. Grains of 150-180 μ m in diameter were obtained by dry sieving. The K-feldspar grains were separated with heavy liquids (2.58 g·cm⁻³) and then etched for 40 min with diluted (10 %) hydrofluoric acid (HF) to clean the grains. HCl (10 %) was used again to dissolve any contaminating fluorides after etching before final rinsing and drying. K-feldspar grains were prepared by mounting the grains in a monolayer, on a 9.8 mm diameter aluminum disc with "Silkospay" silicone oil.

98 The luminescence measurements of the sample HSDK-11 were carried out with an automated Risø TL-DA-15 reader equipped with an IR LED array (880 nm, FWHM 99 40 nm) and a blue LED array (470 nm, FWHM 20 nm) in the Luminescence Dating 100 Laboratory, the University of Hong Kong. The IR and BL stimulations deliver ~135 101 $mW \cdot cm^{-2}$ and ~50 $mW \cdot cm^{-2}$ at the sample position, respectively (Bøtter-Jensen et al., 102 2003). To keep our results comparable with those from Gong et al. (2012), 90% of the 103 full power was used for stimulation in this study. Irradiations were carried out within 104 the reader using a 90 Sr/ 90 Y beta source which delivered a dose rate of 0.0761 Gy·s⁻¹ to 105 106 K-feldspar on aluminum discs. The IRSL and the BLSL signals were both detected after passing through 7.5-mm-thick U-340 filters, which mainly pass light from 290 107 nm to 370 nm with peak transmission at ~340 nm (Li et al., 2007b). The experimental 108 work on the other sample SY was performed in the Luminescence Dating Laboratory, 109 Institute of Geology and Geophysics, Chinese Academy of Sciences. The 110 luminescence measurements of the sample SY were carried out with an automated 111 Risø TL/OSL reader (TL/OSL-DA-15) using the similar equipment setting. The 112 90 Sr/ 90 Y beta source in the equipment delivered a dose rate of 0.0837 Gy·s⁻¹ to 113 K-feldspar on aluminum discs. 114

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117 3. Experimental details and results

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3.1 The relationship between the IRSL and the BLSL at different stimulationtemperatures

Two sets of experiments, namely post-IR BLSL (pIR-BLSL) and post-blue light IRSL (pBL-IRSL), are conducted to investigate the relationship between the IRSL and the BLSL at different stimulation temperatures. For simplification, we describe the stimulation temperatures used in the prior IR and post-IR BLSL as $pIR(T_1)$ -BLSL(T_2), where T_1 is the stimulation temperature used in the prior IR measurement and T_2 is the temperature used in post-IR BLSL measurement.

- 127
- 128 3.1.1 pIR-BLSL experiments
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The pIR-BLSL experiments were carried out using the procedure listed in Table 1. 130 Four aliquots of of K-feldspar grains HSDK-11 were firstly heated to 500 °C and then 131 given a dose of 30.4 Gy. These aliquots were subsequently preheat at 280 °C for 10 s 132 and then bleached using IR stimulation at a temperature of T_1 for different periods 133 ranging from 0 to 5000 s. The pIR-BLSL signal (L_x) was then measured at a 134 temperature of T₂. After that, a test dose of 15.2 Gy was applied and the induced 135 136 BLSL signal (T_x) was measured following the same preheat to monitor sensitivity change for L_x . The signals for both L_x and T_x were calculated from the integrated 137 photon counts in the first 1 s of stimulation, with subtraction of the instrumental 138 background signal. The experiments are conducted at a set of different temperature 139 combinations, i.e. pIR(60)-BLSL(60), pIR(100)-BLSL(60), pIR(150)-BLSL(60), 140 pIR(200)-BLSL(60) and pIR(200)-BLSL(200), respectively. 141

The IR bleaching effects on the pIR-BLSL signal for different periods of time are 142 shown in Fig. 1. It is observed that the IR bleaching at higher temperatures can 143 deplete the BLSL at 60 °C at a faster rate than IR stimulation at lower temperatures. 144 The BLSL at 60 °C was bleached to about 5 % of the initial intensity after IR 145 bleaching at 200 °C for 5000 s. In comparison, the BLSL at 60 °C was bleached to 146 about 15 % of the initial intensity after IR bleaching at 60 °C for 5000 s. If we 147 increase the stimulation temperature in BLSL from 60 to 200 °C, i.e. pIR(200)-BLSL 148 (200), the IR stimulation at 200 °C can bleach the most of the traps associated with 149 the BLSL at 200 °C and only 6 % of the initial intensity of the BLSL at 200 °C was 150

remaining after IR bleaching at 200 °C for 5000 s (Fig. 1). The results suggest that both the BLSL measured at 60 °C and the BLSL at 200 °C can only be partially bleached by prolonged (up to 5000 s) IR stimulation even at a relatively high temperature (i.e. 200 °C).

In our previous study (Gong et al., 2012), it was found that the BLSL signals 155 measured at 60 °C for the K-feldspar from sample HSDK-11 can be described using 156 three first-order exponential components, which are termed as fast (F), medium (M) 157 158 and slow (S) components. Gong et al. (2012) demonstrated that the sources for the IRSL at 60 °C are mainly associated with the fast and medium components of the 159 BLSL at 60 °C. To further demonstrate the relationship between IRSL signal at 160 relatively high temperatures and BLSL at 60 °C, the residual BLSL at 60 °C after IR 161 bleaching for different time from 0 s to 5000 s were then fitted using three OSL 162 components. It is found that the pIR-BLSL signals can be well described by the three 163 exponential functions (all $R^2 > 0.96$). The relative ratios of the decay rates of the 164 components of BLSL at 60 °C, i.e. b_f/b_m and b_m/b_s , are calculated at 4.87±0.14 and 165 166 10.69 \pm 0.41, respectively (here the parameters of b_f, b_m and b_s refer to the decay rate of the fast, medium and slow components of BLSL at 60 °C, respectively). It is noted 167 that the assumption of that the BLSL process is first-order may not be true. However, 168 this will not influence our conclusion because it is the relationship between the 169 different parts of BLSL (represented by the fast, medium and slow components) and 170 IRSL that is crucial for our study, rather than whether these components are first-order 171 or not. We, however, acknowledge that there may be some uncertainty associated with 172 the fitting and some results demonstrated by Fig. 2 and Fig. 6 might be partially 173 174 influenced if these components are not first-order.

Fig. 2a illustrates four representative pIR-BLSL signals, which are fitted into three components. The results of IR bleaching for the fast, medium and slow component of BLSL at 60 °C are shown in Fig. 2b. It is observed that the IR stimulation at 200 °C for 5000 s can deplete 99 % of the fast component, ~99 % of the medium component but only ~38 % of the slow component for the BLSL at 60 °C, while IR stimulation at 60 °C for 5000 s can only deplete ~97 % of fast component, ~91 % of medium component and ~12 % of slow component, respectively, for the BLSL at 60 °C. These results indicate that IRSL obtained at 200 °C involves more traps associated with hard-to-bleach components (i.e. the medium and slow components) of BLSL at 60 °C than does the IR stimulation at 60 °C. The results are consistent with previous studies that the IRSL signals at high temperatures (e.g. >200 °C) are relatively harder to bleach than the IRSL at 60 °C (Buylaert et al., 2011; Li and Li, 2011a; Chen et al., 2013).

188 The relationship between the IRSL and BLSL at different temperatures is further studied by investigating the relationship between the emitted light counts from the 189 IRSL and the corresponding lost counts obtained from the $pIR(T_1)$ -BLSL(T_2) 190 experiments (T_1 = 60, 100, 150, 200 °C; T_2 = 60, 200 °C). This is similar to the method 191 applied to study the relation between IRSL and thermoluminescence (TL) by Duller 192 (1995). In Fig. 3, we plot the emitted counts from the IRSL, against the corresponding 193 lost counts of the pIR-BLSL as a result of IR bleaching. It is observed that, if the 194 stimulation temperature for IR and BL was identical in both cases (i.e. 195 196 pIR(60)-BLSL(60) and pIR(200)-BLSL(200)), the emitted counts of the IRSL have a nearly 1:1 relationship with the corresponding lost counts in the pIR-BLSL. However, 197 in the case of $T_1 > T_2$, the emitted counts of the IRSL are larger than the corresponding 198 lost counts in pIR-BLSL, indicating that the relationship between BLSL and IRSL is 199 dependent on the stimulation temperature. It is to be noted that such a relationship 200 between IRSL and BLSL is not influenced by the interference of isothermal TL, 201 because the preheat at 280 °C for 10 s is sufficient to remove any isothermal TL at 202 200 °C. One straightforward explanation for the temperature dependency of the 203 204 relationship is that at least two components are involved in the IRSL at the relatively high temperature (such as the IRSL at 200 °C). One component is responsive to the 205 BL at 60 °C. The other is hard to reach by BL at 60 °C, but can be accessed at higher 206 temperatures. The results further support fact that the IRSL signals at relatively high 207 temperatures are relatively harder to bleach than the IRSL at 60 °C (e.g. Chen et al., 208 2013). 209

The effects of BL bleaching at 60 °C and 200 °C on the IRSL signals at different 213 temperatures (60, 100, 150 and 200 °C) are investigated using pBL-IRSL experiments 214 (see the procedures listed in Table 1). The experiments conducted are 215 pBL(60)-IRSL(60), pBL(60)-IRSL(100), pBL(60)-IRSL(150), pBL(60)-IRSL(200) 216 and pBL(200)-IRSL(200), respectively. Four aliquots of K-feldspar grains of 217 HSDK-11 were firstly heated to 500 °C to remove any residual signals and then given 218 the same irradiation dose of 30.4 Gy. These aliquots were then held at 280 °C for 10 s. 219 They were subsequently bleached with BL at 60, 200 °C for different periods from 0 220 to 320 s before IRSL measurements. After that, the IRSL sensitivity was monitored 221 and measured following a test dose of 15.2 Gy and preheat at 280 °C for 10 s. 222

The remnant IRSL at different temperatures (50, 100, 150 200 °C) as a result of 223 BL bleaching at 60, 200 °C for different periods of times are shown in Fig. 4. It is 224 demonstrated that the IRSL at 60 °C can be bleached to a negligible level (~ 0.2 %) by 225 BL stimulation at 60 °C for 320 s, while 3.5 % of the initial IRSL at 200 °C still 226 remains after BL bleaching at 60 °C for 320 s. These results indicate that, compared 227 with the IRSL at 60 °C, the IRSL at 200 °C involves more traps that are harder to 228 bleach by BL at 60 °C. However, the IRSL at 200 °C can be bleached to a negligible 229 level (~0.2 %) by BL stimulation at 200 °C for 320 s. In addition, the decay rates in 230 the pBL(200)-IRSL (200) and the pBL(60)-IRSL(60) are very similar and they are 231 calculated at 0.23 ± 0.02 s⁻¹ and 0.21 ± 0.01 s⁻¹, respectively. These results further 232 suggest that the relationship between the IRSL and the BLSL is dependent on 233 234 stimulation temperature.

Further investigation is made on the relationship between the emitted counts from the BLSL and the corresponding lost counts from pBL(T₁)-IRSL(T₂) (T₁= 60, 200 °C; T₂= 60, 100, 150, 200 °C) (Fig. 5). It is observed that the emitted counts from the BLSL measured both at 60 °C and at 200 °C are significantly larger than the corresponding lost counts from pBL(T₁)-IRSL(T₂). These results indicate that BL can access much more traps than IR stimulation. Only part of traps associated with the BLSL at 60 °C and at 200 °C is accessible by IR stimulation, which is similar to the results of IRSL observed at 60 °C (Gong et al, 2012). It is also demonstrated that relationship between emitted BLSL counts and lost counts of pBL-IRSL changes as the stimulation temperature changes.

To further demonstrate the relationship between different OSL components of the 245 BLSL signal at 60 °C and the IRSL signals at relatively high temperatures, the emitted 246 light counts from different OSL components of the BLSL signal at 60 °C are 247 compared with the corresponding lost counts from the pBL(60)-IRSL(200) and 248 pBL(60)-IRSL(60) as a result of BL bleaching at 60 °C for different periods. We plot 249 the emitted counts from the various OSL components of the BLSL at 60 °C, against 250 251 the lost counts of IRSL at 60 °C and IRSL at 200 °C as a result of BL bleaching in Fig. 6. It is observed that the lost counts in pBL(60)-IRSL(200) are larger than the sum of 252 the emitted light counts of the fast and medium components of BLSL at 60 °C, while 253 the lost counts in pBL(60)-IRSL(60) have a nearly 1:1 relationship with the sum of 254 255 the emitted light counts of the fast and medium components of BLSL at 60 °C. These results indicate that the IRSL signals at 200 °C are involved with not only the fast and 256 medium components of BLSL at 60 °C, but also some other OSL components (e.g. 257 slower components of BLSL at 60 °C). In contrast, there is a close relationship 258 between IRSL at 60 °C and the fast and medium components of BLSL at 60 °C (Gong 259 et al., 2012). The results are consistent with the observations in previous section 3.1.1. 260 In summary, the results from the pIR-BLSL and pBL-IRSL bleaching experiments 261 suggest that the relationship between IRSL and BLSL is dependent on stimulation 262 263 temperature. At least two components of traps are involved in the IRSL measured at elevated temperatures (e.g., 200 °C). One component can be easily bleached by BL at 264 60 °C, and the other of the IRSL is relatively harder to access by BL at 60 °C. The 265

results show that the IRSL signals at relatively high temperatures are harder to bebleached than the IRSL at room temperature.

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269 3.2 Luminescence properties of IRSL at relatively high temperature

The luminescence characteristics of the IRSL at 200 °C, the pIR(60)-IRSL(200) and the pBL(60)-IRSL(200), including thermal stability, dose response and laboratory fading rate, were further investigated. In both the pIR(60)-IRSL(200) and the pBL(60)-IRSL(200) experiments, the IR and BL bleaching time was both fixed at 200 s.

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277 3.2.1 Thermal stability study

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The thermal stability studies are carried out using the pulse annealing test (Table 2) 279 (Li et al., 1997; Li and Tso, 1997). The tests were conducted for the IRSL at 60 °C, 280 the IRSL at 200 °C, the pIR(60)-IRSL(200) and the pBL(60)-IRSL(200), respectively. 281 An aliquot of K-feldspar of SY was firstly heated to 500 °C and then given an 282 irradiation dose of 30.4 Gy. After that, it was preheated at 280 °C for 10 s and then 283 heated to a temperature at T °C before the remaining IRSL was measured at 60 °C for 284 285 160 s. The sensitivity change was monitored by measuring the IRSL signal at 60 °C from a test dose of 30.4 Gy. The same preheat condition (280 °C for 10 s) was applied 286 for the test dose IRSL measurement. This cycle was repeated by increasing the 287 annealing temperature (T) from 160 °C to 500 °C in steps of 20 °C. The similar pulse 288 annealing test procedures were also conducted for the IRSL at 200 °C, the 289 pIR(60)-IRSL(200) and the pBL(60)-IRSL(200) (Table 2). The heating rate for all 290 these pulse annealing experiments was 3 °C \cdot s⁻¹. 291

The typical decay curve of the pBL(60)-IRSL(200) signal is shown in Fig. 7. The 292 results of the pulse annealing test of the IRSL at 60 °C, the IRSL at 200 °C, the 293 pIR(60)-IRSL(200) and the pBL(60)-IRSL(200) are shown in Fig. 8. It is observed 294 that the thermal stability of the IRSL at 200 °C is relatively more stable than that of 295 the IRSL at 60 °C. Li and Li (2011b; 2013) also observed the different thermal 296 stabilities among the IRSL at different stimulation temperatures. In addition, it is 297 found that both pIR(60)-IRSL(200) and pBL(60)-IRSL(200) is more thermally stable 298 than IRSL at 200 °C. The results suggest that at least two components are involved in 299

the IRSL at 200 °C and the components have significantly different thermal stability.
Both IR at 60 °C and BL at 60 °C can remove the thermally relatively unstable
component of IRSL 200 °C. It is interesting to be noted that the pBL(60)-IRSL(200)
is significantly more thermally stable than pIR(60)-IRSL(200), indicating that the BL
at 60 °C is more efficient than IR at 60 °C to reduce thermally unstable component in
the IRSL at 200 °C.

- 306
- 307 3.2.2 Dose response curves
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Different shapes of dose response curve (DRC) may provide an indication of 309 different origins of different luminescence signals (Gong et al., 2012). Here we 310 compare the DRC of the IRSL at 200 °C from K-feldspar with that of the 311 pBL(60)-IRSL(200). Regenerative doses ranging from 0 to 1950 Gy were employed 312 in a single aliquot regeneration (SAR) protocol for the IRSL at 200 °C. A test dose of 313 52 Gy was applied and the test dose signal (T_x) was measured to monitor and correct 314 315 for sensitivity change. A recycle dose at 26 Gy was used and the recycling ratios all fall within the range of 1.0 ± 0.05 for the sample. The preheat temperature (held at 316 280 °C for 10 s) was the same for regeneration and test dose measurements. A 317 cut-heat to 500 °C was used between each of the SAR cycles to clean the residual 318 signals from the previous cycle. The IRSL signals L_x and T_x were calculated from the 319 integrated photon counts in the first 1 s of stimulation, with subtraction of a 320 background signal derived from the last 10 s of the 160 s stimulation. For construction 321 the DRC of the pBL(60)-IRSL(200), a similar SAR procedure was applied, except 322 that a BL bleaching at 60 °C for 200 s was added before each IRSL measurement for 323 both the regenerative and test dose measurements. The dose response curves for the 324 two signals are shown in Fig. 9. It is found that the pBL(60)-IRSL(200) signal have a 325 different dose saturation level with the IRSL at 200 °C. 326

327 If the two dose response curves are fitted with double saturation exponential328 function (equation 1),

329
$$I = I_0 + I_a (1 - \exp(-D/D_{0,a}) + I_b (1 - \exp(-D/D_{0,b}))$$
 (1)

The dose saturation level of two D_0 ($D_{0,a}$ and $D_{0,b}$) parameters are 42.9±5.8 Gy and 289.7±22.4 Gy for the pBL(60)-IRSL(200) signal, while the values of two D_0 ($D_{0,a}$ and $D_{0,b}$) parameters of the IRSL at 200 °C are significantly higher at 214.6±9.9 Gy and 806.1±69.6 Gy, respectively. The results indicate that at least two components are involved in the IRSL at elevated temperature. One group is easy to bleach by BL at 60 °C and they have a higher dose saturation level, while the other group is hard to bleach by BL at 60 °C and they have a lower dose saturation level.

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338 3.2.3 Laboratory fading test

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Anomalous fading was observed for both IRSL and BLSL signals in previous 340 studies (e.g. Thomsen et al., 2008). Here we studied the laboratory fading rates for the 341 342 IRSL at 200 °C, the pIR(60)-IRSL(200) and the pBL(60)-IRSL(200) signals. In measurement of the IRSL at 200 °C, six aliquots of SY were heated to 500 °C to 343 remove any residual signals (similar to a hot-bleach between SAR cycles). Then these 344 aliquots were given 50.8 Gy and immediately preheated at 280 °C for 10 s. The 345 sensitivity corrected signals were then measured after delays of different periods. For 346 the test dose, 12.7 Gy was given and the same preheat condition was applied. The 347 IRSL signals $L_{(x)}$ and $T_{(x)}$ were calculated from the integrated photon counts in the 348 first 1 s of stimulation, with subtraction of a background signal derived from the last 349 350 10 s of the 160 s stimulation. The first measurement of the IRSL at 200 °C signal took place at a time $t_c = 562$ s after the mid-point of the irradiation time. A similar 351 measurement procedure was adopted for measuring the fading rate for the 352 pIR(60)-IRSL(200) and pBL(60)-IRSL(200) signals. For the pIR(60)-IRSL(200) 353 signal, an IR bleaching at 60 °C for 200 s was added before the IRSL measurement at 354 200 °C for both the regenerative and test dose measurements. The first measurement 355 of the pIR(60)-IRSL(200) signal took place at a time t_c = 669 s after the mid-point of 356

the irradiation time. For the pBL(60)-IRSL(200) signal, a BL bleaching at 60 °C for 357 200 s was added before the IRSL measurement at 200 °C for both the regenerative 358 and test dose measurements. The first measurement of the pBL(60)-IRSL(200) signal 359 took place at a time $t_c = 669$ s after the mid-point of the irradiation time. The decay of 360 the IRSL at 200 °C, the pIR(60)-IRSL(200) and the pBL(60)-IRSL(200) signals after 361 normalization as a function of storage time is shown in Fig 10. The corresponding 362 anomalous fading rates (g-value) are calculated based on the data sets and are also 363 shown in Fig. 10. It is observed that the IRSL at 200 °C, the pIR(60)-IRSL(200) and 364 the pBL(60)-IRSL(200) have significantly different laboratory fading rates. The g 365 value for the IRSL at 200 °C was detected at 4.0±0.3 %/decade, the g value of the 366 pIR(60)-IRSL(200) was at 1.6±0.4 %/decade and the pBL(60)-IRSL(200) was 0.4± 367 0.4 %/decade. This result indicates that there are at least two components for the IRSL 368 at 200 °C. One component is easy to bleach by IR at 60 °C and BL at 60 °C and it has 369 higher laboratory fading rate, while the other is hard to bleach by IR at 60 °C and BL 370 371 at 60 °C and it has a significantly lower fading rate.

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373 4. Discussion

The sources and process of the traps associated with IRSL from feldspar are 374 important for developing reliable dating methods. Different models have been 375 proposed to explain the various luminescence behaviors of feldspars. A single trap 376 model has been proposed recently to explain the luminescence characteristics for 377 feldspar (e.g., Jain and Ankjærgaard, 2011; Anderson et al., 2012), while a multi-trap 378 model is suggested alternatively by others (e.g., Duller and Bøtter-Jensen, 1993; Li 379 and Li, 2011; Thomsen et al., 2011; Li et al., 2014). These studies were based on their 380 own experimental designs with limited experimental conditions and the explanations 381 are based on different assumptions, so a unique interpretation cannot be reached. It is 382 hoped that the study of the relationship between BLSL and IRSL could be helpful for 383 understanding the source and process of IRSL, because, unlike IRSL process, BLSL is 384 expected to be a simpler and delocalized process due to the higher photon energy of 385

BL (~2.64 eV) compared to the main IRSL trap depth (~2.5 eV) (e.g. Baril and Huntley, 2003; Kars et al., 2013). Based on our results, we are in favor of the multiple-trap model to explain the experimental data obtained in this study, which cannot be well explained using a simple single-trap model. The pieces of evidences are given as follows:

(1) If we assume that IRSL at 200 °C and 60 °C originate the same traps and then 391 both signals should be depleted by BL at a similar rate, because BL have energy high 392 393 enough to evict the trapped electron to the conduction band and then the electron can randomly recombine with both close and distant holes. In Fig. 4, it is clearly showned 394 that, compared with the IRSL at 60 °C, the IRSL at 200 °C is bleached at the 395 significantly slower rate by BL at 60 °C, suggesting that IRSL signals at 200 °C are 396 involved with traps which are very hard to bleach by BL at 60 °C. This could be due 397 to either that the hard-to-bleach component has a deeper trap depth (>2.5 eV) or that 398 the component has a different photoionization cross-section, which both indicate a 399 different trap from the easy to bleach component. 400

401 (2) During the pIR(60)-BLSL(60) experiments, the emitted counts of the IRSL
402 have a nearly 1:1 relationship with the corresponding lost counts in the pIR-BLSL.
403 However, this is not the case for the pIR(200)-BLSL(60) (Fig. 3). This indicates that
404 IRSL at elevated temperature can access more traps that are more difficult to bleach
405 by BL at 60 °C.

(3) The pBL(60)-IRSL(200) and IRSL signals at 200 °C have very different 406 luminescence properties, such as thermal stability, dose response and fading rate. 407 Since BL have energy high enough to evict the trapped electron to the conduction 408 409 band, the electron will randomly recombine with close or distant holes after excitation. 410 Hence, BL will cause not only recombination of spatially close electron-hole pairs, but also recombination of distant electron-hole pairs. As a result, BL bleaching should 411 not change the relative proportions between close and distant electron-hole pairs. 412 Correspondingly, it is expected that the pBL-IRSL should have a similar thermal 413 stability as IRSL, and the pIR-IRSL should have a higher thermal stability than 414 pBL-IRSL. Our results, however, showed that the pBL(60)-IRSL(200) is significantly 415

416 more thermally stable than both the IRSL at 200 °C and pIR(60)-IRSL(200) (Fig. 8), which cannot be explained by the single-trap model. Similarly, a similar fading rate 417 should be expected for the IRSL(200) and pBL(60)-IRSL(200) signals based on a 418 single-trap model. For our samples, the g values for the IRSL at 200 °C are greatly 419 reduced after the BL bleaching at 60 °C for 200 s (Fig. 10). It is interesting to be 420 noted that the laboratory fading rate of pBL(60)-IRSL(200) is significantly lower than 421 that of pIR(60)-IRSL(200), suggesting that the BL at 60 °C is more efficiently than 422 the IR at 60 °C to remove spatially close electron-hole pairs (easy-to-fade), which 423 cannot be explained by a single trap model. 424

Based on the above arguments, we think that a single trap model is not sufficient to explain all the luminescence phenomena in feldspar. In the future, it is maybe helpful to use time-resolved optically stimulated luminescence (TR-OSL) technique to further study the luminescence behaviors of K-feldspar (e.g. Chithambo and Galloway, 2001).

Another outcome of our study is that we first demonstrate that the 430 431 pBL(60)-IRSL(200) has a high thermal stability and a negligible fading rate, which opens the potential of using this signal in sediments dating without the corrections for 432 anomalous fading. A potential advantage of using pBL(60)-IRSL(200) is that blue 433 bleaching at 60 °C can eliminate the contribution of quartz grains to IRSL at 434 elevatedion temperatures (Fan et al., 2009). Quartz grains can coexist with K-feldspar 435 after heavy liquid separation. The IRSL of quartz at elevatedion temperatures can be 436 effectively bleached by blue light at low temperatures, but not by infrared. Further 437 tests on the applicability in dating are required to confirm the suitability of using the 438 439 pBL-IRSL at relatively high temperatures.

440

441 5. Conclusions

442

From the pIR-BLSL and pBL-IRSL bleaching experiments, it is concluded that the relationship between IRSL and BLSL is dependent on the stimulation temperature. If stimulation temperatures for the IRSL increase from 60 to 200 °C, at least two

446 components are associated with the IRSL at 200 °C. One component is easy to bleach 447 by BL at 60 °C, and the other relative hard to bleach by BL at 60 °C. The two 448 components of the IRSL at 200 °C have significantly different luminescence 449 properties, in terms of thermal stability, dose saturation level and laboratory fading 450 rates.

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593 Figure captions

594

Figure 1: Remnant BLSL measured at 60 °C and 200 °C after IR bleaching at different temperature for different times. The temperatures for IR bleaching were set at 60, 100, 150 and 200 °C, respectively.

598

Figure 2: (a) four representative pIR-BLSL signals, which are then deconvoluted into 599 three components. For each of the fitting, the F-statistics are provided and they are all 600 significantly larger than $F_{0.01}$ (e.g. Adamiec, 2005). The corresponding residuals are 601 shown at the right. (b) The residual fast, medium and slow components of BLSL at 602 60 °C after IR bleaching for different time from 0 s to 5000 s. To better demonstrate 603 the data, the residual fast and medium components of BLSL at 60 °C after IR 604 bleaching for different time from 0 s to 320 s were further shown in the insets, while 605 the y-axis in the insets is on the logarithmic scale. The data were from sample 606 HSDK-11 and the fast, medium and slow components of BLSL at 60 °C were fitted 607 with the decay rates of 0.375 ± 0.004 s⁻¹, 0.077 ± 0.002 s⁻¹ and 0.0072 ± 0.0002 s⁻¹, 608 respectively, the same as Gong et al. (2012). 609

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Figure 3: The relationship between emitted counts of the IRSL and the corresponding lost counts of pIR(T₁)-BLSL(T₂) as a result of IR bleaching for different time. T₁= 60, 100, 150, 200 °C, T₂= 60, 200 °C respectively.

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Figure 4: Remnant IRSL after blue light bleaching at 60 °C and 200 °C for different
times. The temperatures for IR stimulations were set at 60, 100, 150 and 200 °C,
respectively.

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Figure 5: The relationship between emitted counts of the BLSL and the corresponding
lost counts of pBL(T₁)-IRSL(T₂) as a result of blue light bleaching for different time.

621 T_1 = 60, 200 °C, T_2 = 60, 100, 150, 200 °C, respectively.

Figure 6: The relationship between emitted counts of OSL components of BLSL at 60 °C and the lost counts of pBL(60)-IRSL(200) and pBL(60)-IRSL(60) as a result of blue light bleaching at 60 °C for different times. F+M: The sum of fast and medium components of the BLSL at 60 °C; S: slow component of the BLSL at 60 °C. The data were from sample HSDK-11.

Figure 7: The typical decay curves of the pBL(60)-IRSL(200) from sample HSDK-11.
All the signals were normalized using the initial intensity of the pBL(60)-IRSL(200).

Figure 8: Pulse annealing curves based on the IRSL signal at 60 °C, the IRSL signal at 200 °C, pIR(60)-IRSL(200) and the pBL(60)-IRSL(200) signal; In the pIR(60)-IRSL(200) and pBL(60)-IRSL(200) experiments, the previous IR stimulation and BL stimulation at 60 °C are both at 200 s. The heating rate was 3 °C·s⁻¹.

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Figure 9: Dose response curves of the IRSL signal at 200 °C and the pBL(60)-IRSL(200) signal. The two dose response curves could be fitted well by the double saturation exponential function ($R^2>0.99$; residuals are shown in the inset).

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Figure 10: Anomalous fading tests for IRSL signal at 200 °C, the pIR(60)-IRSL(200)
and the pBL(60)-IRSL(200) signal using six aliquots from sample SY as a function of
delayed period (t).

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683 Figure 2a



686 Figure 2b













806 Figure 6



812 Figure 7



830 Figure 8



848 Figure 9



Figure 10



881 Table 1

Experimental procedures for the $pIR(T_1)$ -BLSL(T₂) and $pBL(T_2)$ -pIRSL(T₁) experiments. T₁ were set at 60,100, 150, 200 °C respectively, while T₂ were set at 60 and 200 °C.

	$pIR(T_1)$ -BLSL(T_2)		pBL(T ₂)-pIRSL(T ₁)	
Step	Treatment	Observed	Treatment	Observed
(1)	Cut-heat to 500 °C		Cut-heat to 500 °C	
(2)	Regenerative dose (30.4 Gy)		Regenerative dose (30.4 Gy)	
(3)	Preheat to 280 °C for 10 s		Preheat to 280 °C for 10 s	
(4)	IR bleaching at T_1 for different time (0-5000 s)		BL bleaching at T_2 for different time (0-320 s)	
(5)	BLSL measurement at T_2 for 200 s	L pIR-BLSL	IRSL measurement at T ₁ for 160 s	L _{pBL-IRSL}
(6)	Test dose (15.2 Gy)		Test dose (15.2 Gy)	
(7)	Preheat to 280 °C for 10s		Preheat to 280 °C for 10s	
(8)	BLSL measurement at T_2 for 200 s	T _{BLSL}	IRSL measurement at T ₁ for 160 s	T _{IRSL}
(9)	Return to step 1 and time for bleaching changes		Return to step 1 and time for bleaching changes	

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899 Table 2

Pulse annealing procedures for the IRSL at 60 °C, the IRSL at 200 °C, the pIR(60)-IRSL(200) and the pBL(60)-IRSL(200). Note that the sequence of IRSL at 60 °C is steps 1, 2, 3, 4, 5a, 6, 7, 8a and 9, the sequence of IRSL at 200 °C is steps 1, 2, 3, 4, 5b, 6, 7, 8b and 9, the sequence of pIR(60)-IRSL(200) is steps 1, 2, 3, 3a, 4, 5b, 6, 7, 8b and 9 and the sequence of pBL(60)-IRSL(200) is steps 1, 2, 3, 3b, 4, 5b, 6, 7, 8b and 9.

Step	Treatment	Observed
(1)	Cut-heat to 500 °C	
(2)	Regenerative dose (30.4 Gy)	
(3)	Preheat to 280 °C for 10 s	
(3a)	IR bleaching at 60 °C for 200 s	
(3b)	BL bleaching at 60 °C for 200 s	
(4)	Cut-heat to T °C (160 °C -500 °C)	
(5a)	IRSL measurement at 60 °C for 160 s	L _(IRSL 60 °C)
(5b)	IRSL measurement at 200 °C for 160 s	L _(IRSL 200 °C)
(6)	Test dose (30.4 Gy)	
(7)	Preheat to 280 °C for 10 s	
(8a)	IRSL measurement at 60 °C for 160 s	T _(IRSL 60 °C)
(8b)	IRSL measurement at 200 °C for 160 s	T _(IRSL 200 °C)
(9)	Return to step 1 and $T = T + 20 $ °C	

Further studies on the relationship between IRSL and BLSL at

2	relatively high temperatures for potassium-feldspar from sediments
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10	

11 Abstract:

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In optical dating of potassium-feldspar, the luminescence signals can be stimulated 12 by both infrared (IR) light and blue light (BL). To develop reliable dating methods 13 using different stimulation light sources for feldspars, it is important to understand the 14 sources of the traps associated with the infrared stimulated luminescence (IRSL) and 15 blue light stimulated luminescence (BLSL) and their relationship. In this study, we 16 explored the luminescence characteristics of IRSL and BLSL at different stimulation 17 temperatures (from 60 °C to 200 °C) and their relationship based on five sets of 18 experiments, i.e. post-IR BLSL, post-BL IRSL experiments, pulse annealing test, dose 19 response test and laboratory fading rate test. Our results suggest that the luminescence 20 characteristics of IRSL and BLSL and their relationship are dependent on stimulation 21 temperature. For IR stimulation at a relatively high temperature of 200 °C, at least two 22 components of IRSL signals are involved in the process. One component of IRSL 23 signals can be easily bleached by BL stimulation at 60 °C, while the other is relatively 24 25 hard to be bleached by BL stimulation at 60 °C. The two components have different luminescence properties, such as thermal stability, dose response and laboratory fading 26 27 rate.

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29 Keywords: K-feldspar, IRSL, BLSL, component

30

31 1. Introduction

Both quartz and potassium-rich feldspar (K-feldspar) have been widely used as 33 natural dosimeters for optically stimulated luminescence (OSL) dating (Aitken, 1998). 34 Compared with quartz OSL, the infrared stimulated luminescence (IRSL) signal from 35 K-feldspar (Hütt et al., 1988) has advantages of much brighter luminescence signals 36 and much higher dose saturation level, making feldspar as an attractive candidate for 37 luminescene dating of the natural sedimentary samples. However, the usage of 38 39 K-feldspar for dating has long been hindered by the anomalous fading of the trapped charges related to the IRSL signals (e.g. Spooner, 1994; Huntley and Lamonthe, 2001; 40 Li and Li, 2008). 41

More recently, progress in understanding anomalous fading in feldspar has raised 42 the prospect of isolating a non-fading component from the IRSL at relatively high 43 temperatures (Thomsen et al., 2008; Li, 2010; Jain and Ankjærgaard, 2011; Li and Li, 44 2013). Correspondingly, a two-step post IR IRSL (pIRIR) protocol (Buylaert et al., 45 2009; Thiel et al., 2011) and a multi-elevated-temperature post-IR IRSL (MET-pIRIR) 46 47 protocol (Li and Li, 2011a) have been proposed to overcome anomalous fading for dating K-feldspar from sediments, which offer the promising potential for extending 48 the luminescence dating limit (Thiel et al., 2011; Li and Li, 2012; Li et al., 2013, 49 However, the high temperature pIRIR signal (e.g. >200 °C) is found to be more 50 to bleach than the IRSL signal measured at lower temperatures (Li and Li, 2011a; 51 Buylaert et al., 2012; Murray et al., 2012), and it usually requires up to several hours 52 even days of exposure to sunlight or a solar simulator to bleach the pIRIR signal 53 down to a stable level (here the term "bleach" means to reduce the luminescence 54 55 intensity by optical stimulation). For some samples, a significant non-bleachable (or residual) component in the pIRIR signals was left even after a prolonged bleaching 56 period using solar simulator or sunlight (Buylaert et al., 2011; Lowick et al., 2012; 57 Chen et al., 2013; Li et al., 2014b). These studies suggest that the IRSL signals 58 recorded at relatively high temperature have different luminescence behavior 59 compared with the IRSL signals at room temperature. 60

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There have been several studies conducted to explore the relationship between

62 luminescence with IR stimulation and luminescence with visible wavelength light stimulation. It was demonstrated that the majority of green light stimulated 63 luminescence (GLSL) can be bleached by prolonged IR light and an upper limit of \sim 64 90% GLSL was depleted as a result of IR bleaching at room temperature (Duller and 65 Bøtter-Jensen, 1993; Galloway, 1994). Jain and Singhvi (2001) concluded that the 66 blue-green (BG) stimulated luminescence measured at 125 °C is associated with at 67 least two trap populations. One trap population is responsive to both IR stimulation 68 and BG stimulation. Another trap population is only responsive to BG stimulation. 69 Gong et al. (2012) conducted a study on the relationship between the infrared 70 stimulated luminescence (IRSL) and blue light stimulated luminescence (BLSL) at 71 60 °C. They observed that most of the IRSL signals at 60 °C can be bleached by BL at 72 60 °C, while the BLSL signals at 60 °C can only be partially bleached by IR at 60 °C. 73 The sources for the IRSL at 60 °C are mainly associated with the fast and medium 74 components of the BLSL at 60 °C. 75

In this study, in order to better understand the sources of the traps associated with the IRSL and BLSL, we further explore the relationship between IRSL and BLSL using K-feldspar from two aeolian sand samples. The luminescence properties, in terms of thermal stability, dose response and laboratory fading rate, are also examined for the different IRSL components at a relatively high temperature of 200 °C.

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82 2. Samples and equipment

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Two aeolian sand samples (HSDK-11 and SY) from the Hunshandake desert in northeast China were used in this study. Both samples have been investigated in previous studies (Li et al., 2002; Gong et al., 2013). The samples are from the same environmental settings of the same region and have similar luminescence behaviors, so the experimental results obtained from them should be comparable. The samples were treated with 10 % hydrochloric acid (HCl) and 10 % hydrogen peroxide (H₂O₂) to remove carbonate and organic matter, respectively, in subdued red light in the Luminescence Dating Laboratory, the University of Hong Kong. Grains of 150-180 μ m in diameter were obtained by dry sieving. The K-feldspar grains were separated with heavy liquids (2.58 g·cm⁻³) and then etched for 40 min with diluted (10 %) hydrofluoric acid (HF) to clean the grains. HCl (10 %) was used again to dissolve any contaminating fluorides after etching before final rinsing and drying. K-feldspar grains were prepared by mounting the grains in a monolayer, on a 9.8 mm diameter aluminum disc with "Silkospay" silicone oil.

98 The luminescence measurements of the sample HSDK-11 were carried out with an automated Risø TL-DA-15 reader equipped with an IR LED array (880 nm, FWHM 99 40 nm) and a blue LED array (470 nm, FWHM 20 nm) in the Luminescence Dating 100 Laboratory, the University of Hong Kong. The IR and BL stimulations deliver ~135 101 $mW \cdot cm^{-2}$ and ~50 $mW \cdot cm^{-2}$ at the sample position, respectively (Bøtter-Jensen et al., 102 2003). To keep our results comparable with those from Gong et al. (2012), 90% of the 103 full power was used for stimulation in this study. Irradiations were carried out within 104 the reader using a 90 Sr/ 90 Y beta source which delivered a dose rate of 0.0761 Gy·s⁻¹ to 105 106 K-feldspar on aluminum discs. The IRSL and the BLSL signals were both detected after passing through 7.5-mm-thick U-340 filters, which mainly pass light from 290 107 nm to 370 nm with peak transmission at ~340 nm (Li et al., 2007b). The experimental 108 work on the other sample SY was performed in the Luminescence Dating Laboratory, 109 Institute of Geology and Geophysics, Chinese Academy of Sciences. The 110 luminescence measurements of the sample SY were carried out with an automated 111 Risø TL/OSL reader (TL/OSL-DA-15) using the similar equipment setting. The 112 90 Sr/ 90 Y beta source in the equipment delivered a dose rate of 0.0837 Gy·s⁻¹ to 113 K-feldspar on aluminum discs. 114

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117 3. Experimental details and results

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3.1 The relationship between the IRSL and the BLSL at different stimulationtemperatures

Two sets of experiments, namely post-IR BLSL (pIR-BLSL) and post-blue light IRSL (pBL-IRSL), are conducted to investigate the relationship between the IRSL and the BLSL at different stimulation temperatures. For simplification, we describe the stimulation temperatures used in the prior IR and post-IR BLSL as $pIR(T_1)$ -BLSL(T_2), where T_1 is the stimulation temperature used in the prior IR measurement and T_2 is the temperature used in post-IR BLSL measurement.

- 127
- 128 3.1.1 pIR-BLSL experiments
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The pIR-BLSL experiments were carried out using the procedure listed in Table 1. 130 Four aliquots of of K-feldspar grains HSDK-11 were firstly heated to 500 °C and then 131 given a dose of 30.4 Gy. These aliquots were subsequently preheat at 280 °C for 10 s 132 and then bleached using IR stimulation at a temperature of T_1 for different periods 133 ranging from 0 to 5000 s. The pIR-BLSL signal (L_x) was then measured at a 134 temperature of T₂. After that, a test dose of 15.2 Gy was applied and the induced 135 136 BLSL signal (T_x) was measured following the same preheat to monitor sensitivity change for L_x . The signals for both L_x and T_x were calculated from the integrated 137 photon counts in the first 1 s of stimulation, with subtraction of the instrumental 138 background signal. The experiments are conducted at a set of different temperature 139 combinations, i.e. pIR(60)-BLSL(60), pIR(100)-BLSL(60), pIR(150)-BLSL(60), 140 pIR(200)-BLSL(60) and pIR(200)-BLSL(200), respectively. 141

The IR bleaching effects on the pIR-BLSL signal for different periods of time are 142 shown in Fig. 1. It is observed that the IR bleaching at higher temperatures can 143 deplete the BLSL at 60 °C at a faster rate than IR stimulation at lower temperatures. 144 The BLSL at 60 °C was bleached to about 5 % of the initial intensity after IR 145 bleaching at 200 °C for 5000 s. In comparison, the BLSL at 60 °C was bleached to 146 about 15 % of the initial intensity after IR bleaching at 60 °C for 5000 s. If we 147 increase the stimulation temperature in BLSL from 60 to 200 °C, i.e. pIR(200)-BLSL 148 (200), the IR stimulation at 200 °C can bleach the most of the traps associated with 149 the BLSL at 200 °C and only 6 % of the initial intensity of the BLSL at 200 °C was 150

remaining after IR bleaching at 200 °C for 5000 s (Fig. 1). The results suggest that both the BLSL measured at 60 °C and the BLSL at 200 °C can only be partially bleached by prolonged (up to 5000 s) IR stimulation even at a relatively high temperature (i.e. 200 °C).

In our previous study (Gong et al., 2012), it was found that the BLSL signals 155 measured at 60 °C for the K-feldspar from sample HSDK-11 can be described using 156 three first-order exponential components, which are termed as fast (F), medium (M) 157 158 and slow (S) components. Gong et al. (2012) demonstrated that the sources for the IRSL at 60 °C are mainly associated with the fast and medium components of the 159 BLSL at 60 °C. To further demonstrate the relationship between IRSL signal at 160 relatively high temperatures and BLSL at 60 °C, the residual BLSL at 60 °C after IR 161 bleaching for different time from 0 s to 5000 s were then fitted using three OSL 162 components. It is found that the pIR-BLSL signals can be well described by the three 163 exponential functions (all $R^2 > 0.96$). The relative ratios of the decay rates of the 164 components of BLSL at 60 °C, i.e. b_f/b_m and b_m/b_s , are calculated at 4.87±0.14 and 165 166 10.69 \pm 0.41, respectively (here the parameters of b_f, b_m and b_s refer to the decay rate of the fast, medium and slow components of BLSL at 60 °C, respectively). It is noted 167 that the assumption of that the BLSL process is first-order may not be true. However, 168 this will not influence our conclusion because it is the relationship between the 169 different parts of BLSL (represented by the fast, medium and slow components) and 170 IRSL that is crucial for our study, rather than whether these components are first-order 171 or not. We, however, acknowledge that there may be some uncertainty associated with 172 the fitting and some results demonstrated by Fig. 2 and Fig. 6 might be partially 173 174 influenced if these components are not first-order.

Fig. 2a illustrates four representative pIR-BLSL signals, which are fitted into three components. The results of IR bleaching for the fast, medium and slow component of BLSL at 60 °C are shown in Fig. 2b. It is observed that the IR stimulation at 200 °C for 5000 s can deplete 99 % of the fast component, ~99 % of the medium component but only ~38 % of the slow component for the BLSL at 60 °C, while IR stimulation at 60 °C for 5000 s can only deplete ~97 % of fast component, ~91 % of medium component and ~12 % of slow component, respectively, for the BLSL at 60 °C. These results indicate that IRSL obtained at 200 °C involves more traps associated with hard-to-bleach components (i.e. the medium and slow components) of BLSL at 60 °C than does the IR stimulation at 60 °C. The results are consistent with previous studies that the IRSL signals at high temperatures (e.g. >200 °C) are relatively harder to bleach than the IRSL at 60 °C (Buylaert et al., 2011; Li and Li, 2011a; Chen et al., 2013).

188 The relationship between the IRSL and BLSL at different temperatures is further studied by investigating the relationship between the emitted light counts from the 189 IRSL and the corresponding lost counts obtained from the $pIR(T_1)$ -BLSL(T_2) 190 experiments (T_1 = 60, 100, 150, 200 °C; T_2 = 60, 200 °C). This is similar to the method 191 applied to study the relation between IRSL and thermoluminescence (TL) by Duller 192 (1995). In Fig. 3, we plot the emitted counts from the IRSL, against the corresponding 193 lost counts of the pIR-BLSL as a result of IR bleaching. It is observed that, if the 194 stimulation temperature for IR and BL was identical in both cases (i.e. 195 196 pIR(60)-BLSL(60) and pIR(200)-BLSL(200)), the emitted counts of the IRSL have a nearly 1:1 relationship with the corresponding lost counts in the pIR-BLSL. However, 197 in the case of $T_1 > T_2$, the emitted counts of the IRSL are larger than the corresponding 198 lost counts in pIR-BLSL, indicating that the relationship between BLSL and IRSL is 199 dependent on the stimulation temperature. It is to be noted that such a relationship 200 between IRSL and BLSL is not influenced by the interference of isothermal TL, 201 because the preheat at 280 °C for 10 s is sufficient to remove any isothermal TL at 202 200 °C. One straightforward explanation for the temperature dependency of the 203 204 relationship is that at least two components are involved in the IRSL at the relatively high temperature (such as the IRSL at 200 °C). One component is responsive to the 205 BL at 60 °C. The other is hard to reach by BL at 60 °C, but can be accessed at higher 206 temperatures. The results further support fact that the IRSL signals at relatively high 207 temperatures are relatively harder to bleach than the IRSL at 60 °C (e.g. Chen et al., 208 2013). 209

The effects of BL bleaching at 60 °C and 200 °C on the IRSL signals at different 213 temperatures (60, 100, 150 and 200 °C) are investigated using pBL-IRSL experiments 214 (see the procedures listed in Table 1). The experiments conducted are 215 pBL(60)-IRSL(60), pBL(60)-IRSL(100), pBL(60)-IRSL(150), pBL(60)-IRSL(200) 216 and pBL(200)-IRSL(200), respectively. Four aliquots of K-feldspar grains of 217 HSDK-11 were firstly heated to 500 °C to remove any residual signals and then given 218 the same irradiation dose of 30.4 Gy. These aliquots were then held at 280 °C for 10 s. 219 They were subsequently bleached with BL at 60, 200 °C for different periods from 0 220 to 320 s before IRSL measurements. After that, the IRSL sensitivity was monitored 221 and measured following a test dose of 15.2 Gy and preheat at 280 °C for 10 s. 222

The remnant IRSL at different temperatures (50, 100, 150 200 °C) as a result of 223 BL bleaching at 60, 200 °C for different periods of time are shown in Fig. 4. It is 224 demonstrated that the IRSL at 60 °C can be bleached to a negligible level (~ 0.2 %) by 225 BL stimulation at 60 °C for 320 s, while 3.5 % of the initial IRSL at 200 °C still 226 remains after BL bleaching at 60 °C for 320 s. These results indicate that, compared 227 with the IRSL at 60 °C, the IRSL at 200 °C involves more traps that are harder to 228 bleach by BL at 60 °C. However, the IRSL at 200 °C can be bleached to a negligible 229 level (~0.2 %) by BL stimulation at 200 °C for 320 s. In addition, the decay rates in 230 the pBL(200)-IRSL (200) and the pBL(60)-IRSL(60) are very similar and they are 231 calculated at 0.23 ± 0.02 s⁻¹ and 0.21 ± 0.01 s⁻¹, respectively. These results further 232 suggest that the relationship between the IRSL and the BLSL is dependent on 233 234 stimulation temperature.

Further investigation is made on the relationship between the emitted counts from the BLSL and the corresponding lost counts from pBL(T₁)-IRSL(T₂) (T₁= 60, 200 °C; T₂= 60, 100, 150, 200 °C) (Fig. 5). It is observed that the emitted counts from the BLSL measured both at 60 °C and at 200 °C are significantly larger than the corresponding lost counts from pBL(T₁)-IRSL(T₂). These results indicate that BL can access much more traps than IR stimulation. Only part of traps associated with the BLSL at 60 °C and at 200 °C is accessible by IR stimulation, which is similar to the results of IRSL observed at 60 °C (Gong et al, 2012). It is also demonstrated that relationship between emitted BLSL counts and lost counts of pBL-IRSL changes as the stimulation temperature changes.

To further demonstrate the relationship between different OSL components of the 245 BLSL signal at 60 °C and the IRSL signals at relatively high temperatures, the emitted 246 light counts from different OSL components of the BLSL signal at 60 °C are 247 compared with the corresponding lost counts from the pBL(60)-IRSL(200) and 248 pBL(60)-IRSL(60) as a result of BL bleaching at 60 °C for different periods. We plot 249 the emitted counts from the various OSL components of the BLSL at 60 °C, against 250 251 the lost counts of IRSL at 60 °C and IRSL at 200 °C as a result of BL bleaching in Fig. 6. It is observed that the lost counts in pBL(60)-IRSL(200) are larger than the sum of 252 the emitted light counts of the fast and medium components of BLSL at 60 °C, while 253 the lost counts in pBL(60)-IRSL(60) have a nearly 1:1 relationship with the sum of 254 255 the emitted light counts of the fast and medium components of BLSL at 60 °C. These results indicate that the IRSL signals at 200 °C are involved with not only the fast and 256 medium components of BLSL at 60 °C, but also some other OSL components (e.g. 257 slower components of BLSL at 60 °C). In contrast, there is a close relationship 258 between IRSL at 60 °C and the fast and medium components of BLSL at 60 °C (Gong 259 et al., 2012). The results are consistent with the observations in previous section 3.1.1. 260 In summary, the results from the pIR-BLSL and pBL-IRSL bleaching experiments 261 suggest that the relationship between IRSL and BLSL is dependent on stimulation 262 263 temperature. At least two components of traps are involved in the IRSL measured at elevated temperatures (e.g., 200 °C). One component can be easily bleached by BL at 264 60 °C, and the other of the IRSL is relatively harder to access by BL at 60 °C. The 265

results show that the IRSL signals at relatively high temperatures are harder to bebleached than the IRSL at room temperature.

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269 3.2 Luminescence properties of IRSL at relatively high temperature

The luminescence characteristics of the IRSL at 200 °C, the pIR(60)-IRSL(200) and the pBL(60)-IRSL(200), including thermal stability, dose response and laboratory fading rate, were further investigated. In both the pIR(60)-IRSL(200) and the pBL(60)-IRSL(200) experiments, the IR and BL bleaching time was both fixed at 200 s.

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277 3.2.1 Thermal stability study

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The thermal stability studies are carried out using the pulse annealing test (Table 2) 279 (Li et al., 1997; Li and Tso, 1997). The tests were conducted for the IRSL at 60 °C, 280 the IRSL at 200 °C, the pIR(60)-IRSL(200) and the pBL(60)-IRSL(200), respectively. 281 An aliquot of K-feldspar of SY was firstly heated to 500 °C and then given an 282 irradiation dose of 30.4 Gy. After that, it was preheated at 280 °C for 10 s and then 283 heated to a temperature at T °C before the remaining IRSL was measured at 60 °C for 284 285 160 s. The sensitivity change was monitored by measuring the IRSL signal at 60 °C from a test dose of 30.4 Gy. The same preheat condition (280 °C for 10 s) was applied 286 for the test dose IRSL measurement. This cycle was repeated by increasing the 287 annealing temperature (T) from 160 °C to 500 °C in steps of 20 °C. The similar pulse 288 annealing test procedures were also conducted for the IRSL at 200 °C, the 289 pIR(60)-IRSL(200) and the pBL(60)-IRSL(200) (Table 2). The heating rate for all 290 these pulse annealing experiments was 3 °C \cdot s⁻¹. 291

The typical decay curve of the pBL(60)-IRSL(200) signal is shown in Fig. 7. The 292 results of the pulse annealing test of the IRSL at 60 °C, the IRSL at 200 °C, the 293 pIR(60)-IRSL(200) and the pBL(60)-IRSL(200) are shown in Fig. 8. It is observed 294 that the thermal stability of the IRSL at 200 °C is relatively more stable than that of 295 the IRSL at 60 °C. Li and Li (2011b; 2013) also observed the different thermal 296 stabilities among the IRSL at different stimulation temperatures. In addition, it is 297 found that both pIR(60)-IRSL(200) and pBL(60)-IRSL(200) is more thermally stable 298 than IRSL at 200 °C. The results suggest that at least two components are involved in 299

the IRSL at 200 °C and the components have significantly different thermal stability.
Both IR at 60 °C and BL at 60 °C can remove the thermally relatively unstable
component of IRSL 200 °C. It is interesting to be noted that the pBL(60)-IRSL(200)
is significantly more thermally stable than pIR(60)-IRSL(200), indicating that the BL
at 60 °C is more efficient than IR at 60 °C to reduce thermally unstable component in
the IRSL at 200 °C.

- 306
- 307 3.2.2 Dose response curves
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Different shapes of dose response curve (DRC) may provide an indication of 309 different origins of different luminescence signals (Gong et al., 2012). Here we 310 compare the DRC of the IRSL at 200 °C from K-feldspar with that of the 311 pBL(60)-IRSL(200). Regenerative doses ranging from 0 to 1950 Gy were employed 312 in a single aliquot regeneration (SAR) protocol for the IRSL at 200 °C. A test dose of 313 52 Gy was applied and the test dose signal (T_x) was measured to monitor and correct 314 315 for sensitivity change. A recycle dose at 26 Gy was used and the recycling ratios all fall within the range of 1.0 ± 0.05 for the sample. The preheat temperature (held at 316 280 °C for 10 s) was the same for regeneration and test dose measurements. A 317 cut-heat to 500 °C was used between each of the SAR cycles to clean the residual 318 signals from the previous cycle. The IRSL signals L_x and T_x were calculated from the 319 integrated photon counts in the first 1 s of stimulation, with subtraction of a 320 background signal derived from the last 10 s of the 160 s stimulation. For construction 321 the DRC of the pBL(60)-IRSL(200), a similar SAR procedure was applied, except 322 that a BL bleaching at 60 °C for 200 s was added before each IRSL measurement for 323 both the regenerative and test dose measurements. The dose response curves for the 324 two signals are shown in Fig. 9. It is found that the pBL(60)-IRSL(200) signal have a 325 different dose saturation level with the IRSL at 200 °C. 326

327 If the two dose response curves are fitted with double saturation exponential328 function (equation 1),

329
$$I = I_0 + I_a (1 - \exp(-D/D_{0,a}) + I_b (1 - \exp(-D/D_{0,b}))$$
 (1)

The dose saturation level of two D_0 ($D_{0,a}$ and $D_{0,b}$) parameters are 42.9±5.8 Gy and 289.7±22.4 Gy for the pBL(60)-IRSL(200) signal, while the values of two D_0 ($D_{0,a}$ and $D_{0,b}$) parameters of the IRSL at 200 °C are significantly higher at 214.6±9.9 Gy and 806.1±69.6 Gy, respectively. The results indicate that at least two components are involved in the IRSL at elevated temperature. One group is easy to bleach by BL at 60 °C and they have a higher dose saturation level, while the other group is hard to bleach by BL at 60 °C and they have a lower dose saturation level.

337

338 3.2.3 Laboratory fading test

339

Anomalous fading was observed for both IRSL and BLSL signals in previous 340 studies (e.g. Thomsen et al., 2008). Here we studied the laboratory fading rates for the 341 342 IRSL at 200 °C, the pIR(60)-IRSL(200) and the pBL(60)-IRSL(200) signals. In measurement of the IRSL at 200 °C, six aliquots of SY were heated to 500 °C to 343 remove any residual signals (similar to a hot-bleach between SAR cycles). Then these 344 aliquots were given 50.8 Gy and immediately preheated at 280 °C for 10 s. The 345 sensitivity corrected signals were then measured after delays of different periods. For 346 the test dose, 12.7 Gy was given and the same preheat condition was applied. The 347 IRSL signals $L_{(x)}$ and $T_{(x)}$ were calculated from the integrated photon counts in the 348 first 1 s of stimulation, with subtraction of a background signal derived from the last 349 350 10 s of the 160 s stimulation. The first measurement of the IRSL at 200 °C signal took place at a time $t_c = 562$ s after the mid-point of the irradiation time. A similar 351 measurement procedure was adopted for measuring the fading rate for the 352 pIR(60)-IRSL(200) and pBL(60)-IRSL(200) signals. For the pIR(60)-IRSL(200) 353 signal, an IR bleaching at 60 °C for 200 s was added before the IRSL measurement at 354 200 °C for both the regenerative and test dose measurements. The first measurement 355 of the pIR(60)-IRSL(200) signal took place at a time t_c = 669 s after the mid-point of 356

the irradiation time. For the pBL(60)-IRSL(200) signal, a BL bleaching at 60 °C for 357 200 s was added before the IRSL measurement at 200 °C for both the regenerative 358 and test dose measurements. The first measurement of the pBL(60)-IRSL(200) signal 359 took place at a time $t_c = 669$ s after the mid-point of the irradiation time. The decay of 360 the IRSL at 200 °C, the pIR(60)-IRSL(200) and the pBL(60)-IRSL(200) signals after 361 normalization as a function of storage time is shown in Fig 10. The corresponding 362 anomalous fading rates (g-value) are calculated based on the data sets and are also 363 shown in Fig. 10. It is observed that the IRSL at 200 °C, the pIR(60)-IRSL(200) and 364 the pBL(60)-IRSL(200) have significantly different laboratory fading rates. The g 365 value for the IRSL at 200 °C was detected at 4.0±0.3 %/decade, the g value of the 366 pIR(60)-IRSL(200) was at 1.6±0.4 %/decade and the pBL(60)-IRSL(200) was 0.4± 367 0.4 %/decade. This result indicates that there are at least two components for the IRSL 368 at 200 °C. One component is easy to bleach by IR at 60 °C and BL at 60 °C and it has 369 higher laboratory fading rate, while the other is hard to bleach by IR at 60 °C and BL 370 371 at 60 °C and it has a significantly lower fading rate.

372

373 4. Discussion

The sources and process of the traps associated with IRSL from feldspar are 374 important for developing reliable dating methods. Different models have been 375 proposed to explain the various luminescence behaviors of feldspars. A single trap 376 model has been proposed recently to explain the luminescence characteristics for 377 feldspar (e.g., Jain and Ankjærgaard, 2011; Anderson et al., 2012), while a multi-trap 378 model is suggested alternatively by others (e.g., Duller and Bøtter-Jensen, 1993; Li 379 and Li, 2011; Thomsen et al., 2011; Li et al., 2014). These studies were based on their 380 own experimental designs with limited experimental conditions and the explanations 381 are based on different assumptions, so a unique interpretation cannot be reached. It is 382 hoped that the study of the relationship between BLSL and IRSL could be helpful for 383 understanding the source and process of IRSL, because, unlike IRSL process, BLSL is 384 expected to be a simpler and delocalized process due to the higher photon energy of 385

BL (~2.64 eV) compared to the main IRSL trap depth (~2.5 eV) (e.g. Baril and Huntley, 2003; Kars et al., 2013). Based on our results, we are in favor of the multiple-trap model to explain the experimental data obtained in this study, which cannot be well explained using a simple single-trap model. The pieces of evidence are given as follows:

(1) If we assume that IRSL at 200 °C and 60 °C originate the same traps and then 391 both signals should be depleted by BL at a similar rate, because BL have energy high 392 393 enough to evict the trapped electron to the conduction band and then the electron can randomly recombine with both close and distant holes. In Fig. 4, it is clearly shown 394 that, compared with the IRSL at 60 °C, the IRSL at 200 °C is bleached at the 395 significantly slower rate by BL at 60 °C, suggesting that IRSL signals at 200 °C are 396 involved with traps which are very hard to bleach by BL at 60 °C. This could be due 397 to either that the hard-to-bleach component has a deeper trap depth (>2.5 eV) or that 398 the component has a different photoionization cross-section, which both indicate a 399 different trap from the easy to bleach component. 400

401 (2) During the pIR(60)-BLSL(60) experiments, the emitted counts of the IRSL
402 have a nearly 1:1 relationship with the corresponding lost counts in the pIR-BLSL.
403 However, this is not the case for the pIR(200)-BLSL(60) (Fig. 3). This indicates that
404 IRSL at elevated temperature can access more traps that are more difficult to bleach
405 by BL at 60 °C.

(3) The pBL(60)-IRSL(200) and IRSL signals at 200 °C have very different 406 luminescence properties, such as thermal stability, dose response and fading rate. 407 Since BL have energy high enough to evict the trapped electron to the conduction 408 409 band, the electron will randomly recombine with close or distant holes after excitation. 410 Hence, BL will cause not only recombination of spatially close electron-hole pairs, but also recombination of distant electron-hole pairs. As a result, BL bleaching should 411 not change the relative proportions between close and distant electron-hole pairs. 412 Correspondingly, it is expected that the pBL-IRSL should have a similar thermal 413 stability as IRSL, and the pIR-IRSL should have a higher thermal stability than 414 pBL-IRSL. Our results, however, showed that the pBL(60)-IRSL(200) is significantly 415

416 more thermally stable than both the IRSL at 200 °C and pIR(60)-IRSL(200) (Fig. 8), which cannot be explained by the single-trap model. Similarly, a similar fading rate 417 should be expected for the IRSL(200) and pBL(60)-IRSL(200) signals based on a 418 single-trap model. For our samples, the g values for the IRSL at 200 °C are greatly 419 reduced after the BL bleaching at 60 °C for 200 s (Fig. 10). It is interesting to be 420 noted that the laboratory fading rate of pBL(60)-IRSL(200) is significantly lower than 421 that of pIR(60)-IRSL(200), suggesting that the BL at 60 °C is more efficiently than 422 the IR at 60 °C to remove spatially close electron-hole pairs (easy-to-fade), which 423 cannot be explained by a single trap model. 424

Based on the above arguments, we think that a single trap model is not sufficient to explain all the luminescence phenomena in feldspar. In the future, it is maybe helpful to use time-resolved optically stimulated luminescence (TR-OSL) technique to further study the luminescence behaviors of K-feldspar (e.g. Chithambo and Galloway, 2001).

Another outcome of our study is that we first demonstrate that the 430 431 pBL(60)-IRSL(200) has a high thermal stability and a negligible fading rate, which opens the potential of using this signal in sediments dating without the corrections for 432 anomalous fading. A potential advantage of using pBL(60)-IRSL(200) is that blue 433 bleaching at 60 °C can eliminate the contribution of quartz grains to IRSL at elevated 434 temperatures (Fan et al., 2009). Quartz grains can coexist with K-feldspar after heavy 435 liquid separation. The IRSL of quartz at elevated temperatures can be effectively 436 bleached by blue light at low temperatures, but not by infrared. Further tests on the 437 applicability in dating are required to confirm the suitability of using the pBL-IRSL at 438 439 relatively high temperatures.

440

441 5. Conclusions

442

From the pIR-BLSL and pBL-IRSL bleaching experiments, it is concluded that the relationship between IRSL and BLSL is dependent on the stimulation temperature. If stimulation temperatures for the IRSL increase from 60 to 200 °C, at least two

446 components are associated with the IRSL at 200 °C. One component is easy to bleach 447 by BL at 60 °C, and the other relative hard to bleach by BL at 60 °C. The two 448 components of the IRSL at 200 °C have significantly different luminescence 449 properties, in terms of thermal stability, dose saturation level and laboratory fading 450 rates.

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593 Figure captions

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Figure 1: Remnant BLSL measured at 60 °C and 200 °C after IR bleaching at different temperature for different times. The temperatures for IR bleaching were set at 60, 100, 150 and 200 °C, respectively.

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Figure 2: (a) four representative pIR-BLSL signals, which are then deconvoluted into 599 three components. For each of the fitting, the F-statistics are provided and they are all 600 significantly larger than $F_{0.01}$ (e.g. Adamiec, 2005). The corresponding residuals are 601 shown at the right. (b) The residual fast, medium and slow components of BLSL at 602 60 °C after IR bleaching for different time from 0 s to 5000 s. To better demonstrate 603 the data, the residual fast and medium components of BLSL at 60 °C after IR 604 bleaching for different time from 0 s to 320 s were further shown in the insets, while 605 the y-axis in the insets is on the logarithmic scale. The data were from sample 606 HSDK-11 and the fast, medium and slow components of BLSL at 60 °C were fitted 607 with the decay rates of $0.375 \pm 0.004 \text{ s}^{-1}$, $0.077 \pm 0.002 \text{ s}^{-1}$ and $0.0072 \pm 0.0002 \text{ s}^{-1}$, 608 respectively, the same as Gong et al. (2012). 609

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Figure 3: The relationship between emitted counts of the IRSL and the corresponding lost counts of pIR(T₁)-BLSL(T₂) as a result of IR bleaching for different time. T₁= 60, 100, 150, 200 °C, T₂= 60, 200 °C respectively.

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Figure 4: Remnant IRSL after blue light bleaching at 60 °C and 200 °C for different
times. The temperatures for IR stimulations were set at 60, 100, 150 and 200 °C,
respectively.

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Figure 5: The relationship between emitted counts of the BLSL and the corresponding lost counts of $pBL(T_1)$ -IRSL (T_2) as a result of blue light bleaching for different time.

621 T_1 = 60, 200 °C, T_2 = 60, 100, 150, 200 °C, respectively.

Figure 6: The relationship between emitted counts of OSL components of BLSL at 60 °C and the lost counts of pBL(60)-IRSL(200) and pBL(60)-IRSL(60) as a result of blue light bleaching at 60 °C for different times. F+M: The sum of fast and medium components of the BLSL at 60 °C; S: slow component of the BLSL at 60 °C. The data were from sample HSDK-11.

Figure 7: The typical decay curves of the pBL(60)-IRSL(200) from sample HSDK-11.
All the signals were normalized using the initial intensity of the pBL(60)-IRSL(200).

Figure 8: Pulse annealing curves based on the IRSL signal at 60 °C, the IRSL signal at 200 °C, pIR(60)-IRSL(200) and the pBL(60)-IRSL(200) signal; In the pIR(60)-IRSL(200) and pBL(60)-IRSL(200) experiments, the previous IR stimulation and BL stimulation at 60 °C are both at 200 s. The heating rate was 3 °C·s⁻¹.

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Figure 9: Dose response curves of the IRSL signal at 200 °C and the pBL(60)-IRSL(200) signal. The two dose response curves could be fitted well by the double saturation exponential function ($R^2>0.99$; residuals are shown in the inset).

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Figure 10: Anomalous fading tests for IRSL signal at 200 °C, the pIR(60)-IRSL(200)
and the pBL(60)-IRSL(200) signal using six aliquots from sample SY as a function of
delayed period (t).

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683 Figure 2a



686 Figure 2b













806 Figure 6



812 Figure 7



830 Figure 8



848 Figure 9



Figure 10



881 Table 1

Experimental procedures for the $pIR(T_1)$ -BLSL(T₂) and $pBL(T_2)$ -pIRSL(T₁) experiments. T₁ were set at 60,100, 150, 200 °C respectively, while T₂ were set at 60 and 200 °C.

	pIR(T ₁)-BLSL(T ₂)		pBL(T ₂)-pIRSL(T ₁)	
Step	Treatment	Observed	Treatment	Observed
(1)	Cut-heat to 500 °C		Cut-heat to 500 °C	
(2)	Regenerative dose (30.4 Gy)		Regenerative dose (30.4 Gy)	
(3)	Preheat to 280 °C for 10 s		Preheat to 280 °C for 10 s	
(4)	IR bleaching at T_1 for different time (0-5000 s)		BL bleaching at T_2 for different time (0-320 s)	
(5)	BLSL measurement at T_2 for 200 s	L pIR-BLSL	IRSL measurement at T ₁ for 160 s	L _{pBL-IRSL}
(6)	Test dose (15.2 Gy)		Test dose (15.2 Gy)	
(7)	Preheat to 280 °C for 10s		Preheat to 280 °C for 10s	
(8)	BLSL measurement at T_2 for 200 s	T _{BLSL}	IRSL measurement at T ₁ for 160 s	T _{IRSL}
(9)	Return to step 1 and time for bleaching changes		Return to step 1 and time for bleaching changes	

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899 Table 2

Pulse annealing procedures for the IRSL at 60 °C, the IRSL at 200 °C, the pIR(60)-IRSL(200) and the pBL(60)-IRSL(200). Note that the sequence of IRSL at 60 °C is steps 1, 2, 3, 4, 5a, 6, 7, 8a and 9, the sequence of IRSL at 200 °C is steps 1, 2, 3, 4, 5b, 6, 7, 8b and 9, the sequence of pIR(60)-IRSL(200) is steps 1, 2, 3, 3a, 4, 5b, 6, 7, 8b and 9 and the sequence of pBL(60)-IRSL(200) is steps 1, 2, 3, 3b, 4, 5b, 6, 7, 8b and 9.

Step	Treatment	Observed
(1)	Cut-heat to 500 °C	
(2)	Regenerative dose (30.4 Gy)	
(3)	Preheat to 280 °C for 10 s	
(3a)	IR bleaching at 60 °C for 200 s	
(3b)	BL bleaching at 60 °C for 200 s	
(4)	Cut-heat to T °C (160 °C -500 °C)	
(5a)	IRSL measurement at 60 °C for 160 s	L _(IRSL 60 °C)
(5b)	IRSL measurement at 200 °C for 160 s	L _(IRSL 200 °C)
(6)	Test dose (30.4 Gy)	
(7)	Preheat to 280 °C for 10 s	
(8a)	IRSL measurement at 60 °C for 160 s	T _(IRSL 60 °C)
(8b)	IRSL measurement at 200 °C for 160 s	T _(IRSL 200 °C)
(9)	Return to step 1 and $T = T + 20 \text{ °C}$	