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Effects of thermally transferred signals in the post-IR IRSL SAR protocol

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ABSTRACT

The recently developed post-IR IRSL SAR protocol is promising to isolate more stable IRSL signals of feldspars. However, the high temperature thermal treatments used will inevitably induce thermally transferred post-IR $IRSL_{295}$ (TT-post-IR $IRSL_{295}$) signal, which would contribute to the measured post-IR $IRSL_{295}$ signal of the test dose and may lead to inaccurate sensitivity correction. In this study, the effects of TT-post-IR $IRSL_{295}$ signal in the post-IR $IRSL_{295}$ SAR protocol are investigated using medium polyminerals from a loess section at Caoxian, northwestern Loess Plateau in China. The sensitivity changes of the $IRSL_{50}$ and the post-IR $IRSL_{295}$ signals are different from natural cycle to regenerative cycles, which can be attributed to the interference of the TT-post-IR $IRSL_{295}$ signal. Such difference is dependent on the test dose and the post-IR IR stimulation time. The TT-post-IR $IRSL_{295}$ signal is also shown to affect the post-IR $IRSL_{295}$ D_o and D_e values and to cause overestimation of the fading rates. Our study therefore highlights the need of serious consideration on the effects of TT-post-IR IRSL signal when the post-IR IRSL SAR protocol is employed for dating.

1. Introduction

In an attempt to extend the upper age range of luminescence dating, a new approach for determining equivalent dose (D_e) using feldspar signals, the post-IR IRSL SAR protocol, has been recently applied to aeolian (Buylaert et al., 2009; Stevens et al., 2011; Thiel et al., 2011), fluvial (Buylaert et al., 2009) and coastal sediments (Reimann et al., 2011). In this protocol, a prior IR stimulation at 50 °C is made to eliminate the easy-to-fade electrons, then the second IR stimulation (post-IR IRSL) is performed at an elevated temperature (e.g. 225 °C or 290 °C with the preheat temperature at 250 °C or 320 °C correspondingly). It has been shown that the fading of the post-IR $IRSL_{225}$ signal is much less severe than that of the conventional $IRSL_{50}$ signal (Thomsen et al., 2008; Buylaert et al., 2009) and the post-IR $IRSL_{290}$ signal does not show any obvious signs of fading (Stevens et al., 2011; Thiel et al., 2011; Thomsen et al., 2011). While the new approach seems to hold promise for extending the dating range, the high temperature incorporated will inevitably generate unwanted signals as a result of the thermal treatment. In an earlier study, after bleaching the natural signal by IR_{225} for 200 s, Nian et al. (2009) heated the sample to 250 °C and carried out IR_{50} and post-IR IR_{225} stimulation again, and a significant post-IR $IRSL_{225}$ signal was observed. They termed such signal as thermally transferred post-IR $IRSL_{225}$ (TT-post-IR $IRSL_{225}$) signal. Stevens et al. (2011) found that the residual doses generated by using the $IRSL_{50}$ signal were less than 5 Gy, while those induced by the post-IR $IRSL_{290}$ signal were much larger and very sensitive to the previous dose. They attributed the residual signal to the effect of thermal transfer. Qin and Zhou (submitted for publication) observed non-negligible TT-post-IR $IRSL_{225}$ from polymineral grains in loess of Luochuan, central Loess Plateau of China. They argued that the thermally transferred signal may distort the observed sensitivity change and lead to the underestimation of the post-IR $IRSL_{225}$ SAR ages. In an earlier study, Auclair et al. (2003) had speculated that the thermally transferred IRSL signal may also lead to inaccurate estimation of the fading rate of conventional IRSL signal. In this study, the intensity of the TT-post-IR $IRSL_{295}$ signal is investigated and its effect on sensitivity correction and fading rate estimation in the post-IR $IRSL_{295}$ SAR protocol is studied.

2. Samples, instruments and methods

The samples used in this study were taken from a loess section at Caoxian located in the northwestern Loess Plateau of China. L1846 and L985 were taken from the stratigraphical levels equivalent to marine isotope stage (MIS) 2 and early MIS 3, and samples L1013 and L1015 were from the bottom of L1 and the top of L2, which are correlated to early MIS 4 and late MIS 6, respectively.

Following the treatments with 10% HCl and 30% H_2O_2, 45–63 μm polymineral grains were extracted by wet sieving. All

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luminescence measurements were made on a Risø TL/OSL-DA-20 reader equipped with $^{90}Sr/^{90}Y$ beta source. The IRSL and post-IR IRSL signals were stimulated with an array of twenty one IR diodes emitting at 870 nm (FWHM 40 nm) and detected by an EMI 9235QB photomultiplier tube with Schott BG39/Corning 7-59 filters combination in front (Botter-Jensen et al., 2003). The post-IR IRSL SAR protocol was used to determine the equivalent dose (Stevens et al., 2011; Thiel et al., 2011). Post-IR IR stimulation was carried out at 295 °C following a preheat at 320 °C and a prior IR stimulation at 50 °C. The 325 °C IR bleach at the end of each cycle was not incorporated in this study.

The sensitivities of post-IR IRSL signals are monitored by their responses to a test dose in the post-IR IRSL SAR protocol. When the test dose signals are stimulated, the TT-post-IR $IRSL_{295}$ signal inherited from previous doses would be detected indistinguishably and mask the true sensitivity. In the following section, the intensity of the TT-post-IR $IRSL_{295}$ signal is evaluated. Subsequently, the sensitivity change characteristics of the $IRSL_{50}$ and post-IR $IRSL_{295}$ signals throughout the post-IR IRSL SAR cycles are studied using samples with different natural dose. Then, the effect of TT-post-IR $IRSL_{295}$ signal on sensitivity correction, equivalent dose (D_e) characteristic saturation dose (D_o) and fading rate (g value) measurements are investigated by varying the size of test dose and post-IR IR stimulation time. In particular, its effect of on g value estimation is investigated by varying the order of preheat and laboratory storage.

3. Results and discussion

3.1. Observation of thermally transferred IRSL signals

Sample L1013 was used to investigate the intensity of the TT-post-IR $IRSL_{295}$ signal. After measuring the $IRSL_{50}$ and post-IR $IRSL_{295}$ signals (stimulation time is 100 s for both) of 190 Gy regenerative dose, the aliquot was heated at 320 °C for 60 s (equal to the preheat for test dose) and was stimulated by IR at 50 °C and 295 °C successively. The $IRSL_{50}$ and post-IR $IRSL_{295}$ signals observed in these two steps are the thermally transferred $IRSL_{50}$ (TT-$IRSL_{50}$) and the TT-post-IR $IRSL_{295}$ signal, respectively (Fig. 1a, dash and dotted lines). Then, a test dose of 25 Gy was administered and the $IRSL_{50}$ and post-IR $IRSL_{295}$ responses were measured as shown in Fig. 1a. The ratio of the TT-post-IR $IRSL_{295}$ to the post-IR $IRSL_{295}$ signal is larger than 25%, while that of the TT-$IRSL_{50}$ to the $IRSL_{50}$ signal is less than 10%. The TT-post-IR $IRSL_{295}$ signal would contribute much more significantly to the measured post-IR $IRSL_{295}$ signal of the test dose than the TT-$IRSL_{50}$ signal.

The relationship between the TT-post-IR $IRSL_{295}$ and TT-$IRSL_{50}$ signal was investigated. These two signals were measured repeatedly for three times, after the natural signal and regenerative signals (250 Gy) were measured respectively. The proportional relationship is identified between the TT-post-IR $IRSL_{295}$ and TT-$IRSL_{50}$ signal, and the ratio of them is higher following natural dose than that following regenerative dose (Fig. 1b).

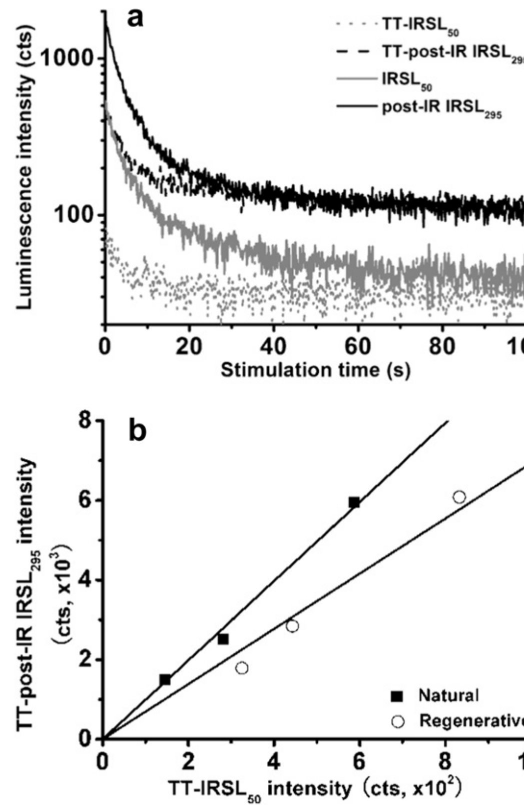

Fig. 1. a) The observation of the TT-IRSL and TT-post-IR IRSL signal. After measuring the $IRSL_{50}$ and post-IR $IRSL_{295}$ signals of 190 Gy regenerative dose, the aliquot was preheated and stimulated by IR at 50 °C (gray dotted) and 295 °C (black dotted) successively. Then, the $IRSL_{50}$ (gray solid) and post-IR $IRSL_{295}$ (black solid) signals of 25 Gy test dose were measured; b) the proportional relationship between the TT-$IRSL_{50}$ and TT-post-IR $IRSL_{295}$ signals. The ratio of the TT-post-IR $IRSL_{295}$ to TT-$IRSL_{50}$ signal is higher for natural dose than for regenerative dose.

3.2. Sensitivity changes of $IRSL_{50}$ and post-IR $IRSL_{295}$ signals

The sensitivity changes of the $IRSL_{50}$ and post-IR $IRSL_{295}$ signals throughout the post-IR IRSL SAR cycles were traced using samples L1846 (MIS 2) and L985 (MIS 3), and the same regenerative doses ranging from 48 Gy to 335 Gy were adopted for the two samples. The $IRSL_{50}$ and post-IR $IRSL_{295}$ signals measured following the test dose were used as indicators for their sensitivities, and all data were normalized to the values of the natural cycle (N). Fig. 2(a) and (c) show that the sensitivity changes of the $IRSL_{50}$ and post-IR $IRSL_{295}$ signals are similar during the regenerative cycles, which

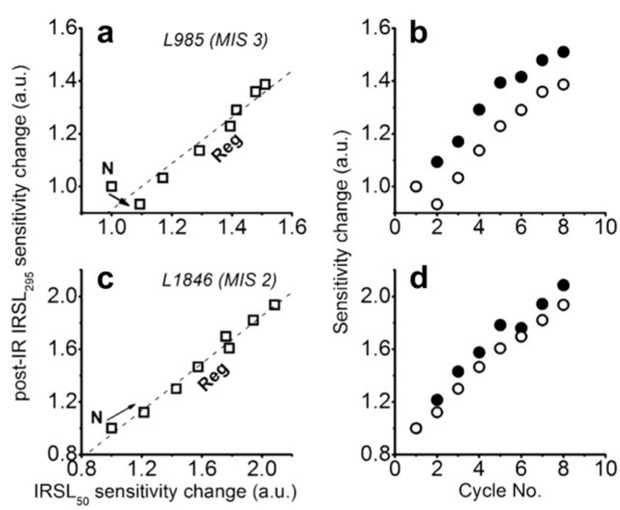

Fig. 2. The sensitivity changes of the $IRSL_{50}$ and post-IR $IRSL_{295}$ signals throughout the post-IR IRSL SAR cycles for sample L985 (a and b) and L1846 (c and d) from the strata corresponding to MIS 3 and MIS 2, respectively. All data were normalized to the value of the natural cycle (N). The regenerative doses were the same for the two samples. The sensitivity changes of the $IRSL_{50}$ and post-IR $IRSL_{295}$ signal show similar pattern during regenerative cycles for sample (a) L985 (MIS 3) and (c) L1846 (MIS 2), while the sensitivity change of these two signals differed from the natural cycle to the first regenerative cycle, and the difference was larger for sample L985, of which the natural dose was much larger. In (b) and (d), the sensitivity of $IRSL_{50}$ (solid circle) and post-IR $IRSL_{295}$ (open circle) are plotted against the cycle number of SAR protocol.