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Simulations of thermally transferred OSL experiments and of the ReSAR dating protocol for quartz

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ABSTRACT

In several recent studies, a thermally transferred OSL (TT-OSL) signal has been proposed as the basis of a new OSL dating procedure. In this paper we present simulations of several published TT-OSL experiments. The first simulation is of repeated cycles of preheating (260 °C for 10 s) and TT-OSL measurements in order to separate the recuperated OSL (ReOSL) and basic-transferred OSL (BT-OSL) signals. The second simulation is of the temperature dependence of the TT-OSL signal after the aliquots were annealed for 1 s at temperatures from 200 to 400 °C. In addition, we simulate the construction of a dose response curve using the ReOSL signal in a single-aliquot regenerative-dose (SAR) protocol used for dating. The results of the three simulations are in general qualitative agreement with the experiments and confirm that a single charge transfer mechanism is responsible for the ReOSL signal.

1. Introduction

Several recent studies have shown that a thermally transferred OSL (TT-OSL) signal from quartz grains can be used as the basis of a new OSL dating procedure (Wang et al., 2006a; Tsukamoto et al., 2008). By using this signal, it is possible to extend the dating range for Chinese loess by almost an order of magnitude (Wang et al., 2006b). Wang et al. (2006b) investigated the properties of TT-OSL signals from fine-grained quartz extracted from Chinese loess and concluded that the TT-OSL was made up of a recuperated OSL (ReOSL) component and a basic-transferred OSL (BT-OSL) component, using the terminology of Aitken (1998). Wang et al. (2006b) designed a procedure to separate the BT-OSL signal and the ReOSL signal, either by using a sequence of repeated thermal and optical treatments, or by employing a single thermal treatment. They found that the recuperated TT-OSL is the major contributor to the measured TT-OSL signal, when heating at 260 °C for 10 s was used to induce the TT-OSL. The TT-OSL signal had the same optical decay characteristics as the OSL signal and it was concluded that the electrons that had been thermally released into the conduction band were then trapped in the main OSL traps.

Aitken (1998) suggested that the ReOSL may result from a "double transfer" process involving photo-transfer during optical stimulation and thermal transfer during preheating. He also suggested that the BT-OSL was due to electrons trapped originally in light-insensitive traps and which had been transferred into the OSL traps by the heating at 260 °C for 10 s. An alternative "single transfer" process for the production of ReOSL has been suggested by Adamiec et al. (2008) and was modeled by Pagonis et al. (2008). In the single transfer process, a light-sensitive trap (but one much less light sensitive than the main OSL traps) acts as the source of electrons for the ReOSL, rather than as the "refuge trap" hypothesized by Aitken (1998) in the "double transfer" process. It is assumed that during a 10 s preheat at 260 °C, a small percentage of the charge in this source trap transfers into the main OSL traps and subsequently creates the ReOSL part of the TT-OSL signal measured during blue light stimulation at 125 °C.

Adamiec et al. (2008) examined these two possible mechanisms. By analyzing the decay rates of the OSL and the TT-OSL signals as a function of stimulation time, they concluded that the two signals are derived from the same traps, and are dominated by the signal from the fast OSL component traps. They also presented experimental data that allowed them to conclude that a single transfer

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process was appropriate for the production of the ReOSL signal observed above 120 °C.

Pagonis et al. (2007) gave a mathematical description of several possible thermal transfer processes for ReOSL in quartz when stimulation was at room temperature, and when stimulation was at a temperature at 125 °C following a laboratory heating. The results, obtained using a comprehensive model for quartz luminescence (Bailey, 2001), were compared with three different sets of previously published experimental data. Subsequently, Pagonis et al. (2008) used a modified version of the Bailey (2001) model to simulate the dose response curves of the OSL, TT-OSL and BT-OSL signals for doses up to 4000 Gy.

The purpose of the present paper is to extend the work of Pagonis et al. (2008) and model published data from annealing experiments and repeated heat/OSL cycle experiments (Wang et al., 2006b; Tsukamoto et al., 2008). Furthermore, we simulate a newly proposed dating protocol that uses the ReOSL (Wang et al., 2007). The overall goal of this paper is to show that the results of these experiments are consistent with the single transfer mechanism for the experimentally observed ReOSL signals in quartz extracted from Chinese loess.

2. The numerical model

A modified version of the original Bailey (2001) model is used in all the simulations presented in this paper. This model was used recently by Pagonis et al. (2008) to simulate the complete ReSAR multiple aliquot dating protocol of Wang et al. (2006a). The computer code, the set of differential equations and the choice of parameters were presented recently in this journal by Pagonis et al. (2008), and will not be repeated here. For easy reference we briefly describe here the levels in the model, as well as present the values of the parameters in Table 1.

The original model by Bailey (2001) consists of 5 electron traps and 4 hole centers. Level 1 in the model represents a shallow electron trapping level, which gives rise to a TL peak at ~100 °C with a heating rate of 5 K/s. The corresponding OSL signal plays only a minor role in the current simulations, since OSL measurements are carried out at 125 °C. Level 2 represents a generic "230 °C TL" trap, typically found in many quartz samples. Levels 3 and 4 are the traps that give rise to the fast and medium OSL components and they would give rise to TL peaks at ~330 °C. Level 5 is a deep, thermally disconnected, electron center. Levels 6 and 7 are thermally unstable, non-radiative recombination centers ("hole reservoirs"). Level 8 is a thermally stable, radiative recombination center termed the "luminescence center" (L). Level 9 is a thermally stable, non-radiative recombination center termed a "killer" center (K). Levels 10 and 11 are the two new levels added to the original model by Pagonis et al. (2008), and play an essential role in the production of the TT-OSL signals and BT-OSL signals observed experimentally by Wang et al. (2006a,b). Level 10 represents the source trap for the charge giving rise to the TT-OSL signal and is a slightly less thermally stable trap with high dose saturation. It is assumed that a proportion of the electrons from level 10 are thermally transferred to the empty fast component trap (level 3) via the conduction band. This trap (level 10) is assumed to be emptied optically in nature by long sunlight exposure. Level 11 is assumed to contribute the majority of the BT-OSL signal with a proportion of the electrons from these light-insensitive traps being transferred via the conduction band to the less thermally stable level 3; level 10 is also assumed to be less thermally stable than level 11.

The parameters are as defined by Bailey (2001); N_i are the concentrations of electron traps or hole centers (cm^{-3}), n_i are the concentrations of trapped electrons or holes (cm^{-3}), s_i are the frequency factors (s^{-1}), E_i are the electron trap depths below the conduction band or hole trap depths above the valence band (eV), A_i ($i=1,\ldots,5$, and $i=10$, 11) are the conduction band to electron trap transition probability coefficients (cm^3 s^{-1}), A_j ($j=6,\ldots,9$) are the valence band to hole trap transition probability coefficient (cm^3 s^{-1}), valence band to trap transition probability coefficients (cm^3 s^{-1}) and B_j ($j=6,\ldots,9$) are the conduction band to hole center transition probability coefficients (cm^3 s^{-1}). Other parameters related to the photoionization cross-sections of the optically sensitive traps are the photo-eviction constant θ_{0i} (s^{-1}) at $T=\infty$, the thermal assistance energy E_i^{th} (eV). The numerical values given in Table 1 are those given in Table 2 of Pagonis et al. (2008).

Table 1
The parameters of Pagonis et al. (2008) are shown together with modified values for N_{10} and N_{11} introduced in the present simulations shown in bold.

	N_i, cm^{-3}	E_i, eV	s_i, s^{-1}	A_i, cm^3 s^{-1}	B_i, cm^3 s^{-1}	θ_{0i}, s^{-1}	E_i^{th}, eV
1	1.5e7	0.97	5e12	1e−8		0.75	1
2	1e7	1.55	5e14	1e−8		0	0
3	4e7	1.73	6.5e13	5e−9		6	0.1
4	2.5e8	1.8	1.5e13	5e−10		4.5	.13
5	5e10	2	1e10	1e−10		0	0
6	3e8	1.43	5e13	5e−7	5e−9	0	0
7	1e10	1.75	5e14	1e−9	5e−10	0	0
8	3e10	5	1e13	1e−10	1e−10	0	0
9	1.2e12	5	1e13	1e−14	3e−10	0	0
10	5e9 (**1.5e9**)	1.65	6.5e13	1e−11		0.01	0.2
11	4e9 (**1.2e9**)	1.6	5e12	6e−12		0	0

3. Simulation #1: the repeated OSL/heat cycles experiment

An experiment consisting of repeated cycles of preheating and TT-OSL measurement was shown in Fig. 5a of Wang et al. (2006b). The experiment consisted of repeated heating and bleaching measurements on the same aliquot of sample IEE266. This sample was obtained from a depth of 12.3 m and had a natural dose of 409 ± 18 Gy, as obtained using multiple aliquot OSL measurements (Lu et al., 2007). After several successive cycles the TT-OSL seemed to approach a limiting value which was originally thought to represent a non-bleachable signal, perhaps corresponding to the "basic-transfer" TT-OSL signal of Aitken (1998). Further work by Wang et al. (2007) showed that the magnitude of this remaining signal reached after several cycles, depends on the magnitude of the test dose used during the experiment. Similar results were obtained using sedimentary quartz by Tsukamoto et al. (2008).

The experiment consisted of 11 measurements of the TT-OSL signal using the same preheat (260 °C for 10 s) and optical stimulation conditions (blue diode stimulation at 125 °C for 360 s). In each cycle the TT-OSL signal is measured ($L_{\mathrm{TT\text{-}OSL}}$). The aliquot is then irradiated with a test dose of 7.8 Gy, preheated using a cutheat of 220 °C for 10 s, and optically stimulated with blue light at 125 °C for 100 s. The test dose response signal ($T_{\mathrm{TT\text{-}OSL}}$) is recorded. The complete process is repeated for the same aliquot 10 more times, and the sensitivity corrected TT-OSL signal ($L_{\mathrm{TT\text{-}OSL}}/T_{\mathrm{TT\text{-}OSL}}$) is obtained as a function of the cycle number.

The parameters used in the simulation are those of Table 2 in Pagonis et al. (2008), and they are listed in Table 1 of this paper. The complete sequence of steps in the simulation is shown in Table 2. Fig. 1a shows the results of the simulation as a broken line, and the experimental data of Wang et al. (2006b, Fig. 5a). The simulated results show the same qualitative behavior, but the simulated TT-OSL signal on the y-axis is approximately three times as large as the experimental data for the sensitivity corrected signal $L_{\mathrm{TT\text{-}OSL}}/T_{\mathrm{TT\text{-}OSL}}$.

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Table 2
The experiment of Wang et al. (2006b) simulated using the model of Pagonis et al. (2008). Steps 1–5 are a simulation of a "natural" quartz sample IEE266 with a natural dose of 409 Gy and steps 7–11 simulate the repeated cycle experiment.

1	Natural quartz sample. Set all trap populations to zero
2	Geological dose – 1000 Gy at 1 Gy/s at 20 °C
3	Geological time – heat to 350 °C
4	Illuminate for 100 s at 200 °C
Repeated daylight exposures over a long period of time	
5	Burial dose- 409 Gy at 20 °C at 10^{-6} Gy/s
6	Bleach sample for 360 s at 125 °C, to empty OSL traps
7	Preheat sample at 260 °C for 10 s
8	Blue stimulation at 125 °C for 100 s – Record OSL (5 s) (L)
9	Give test dose of 7.8 Gy at 1 Gy/s at 20 °C
10	Cutheat at 220 °C for 10 s
11	Blue stimulation at 125 °C for 100 s – Record OSL (5 s) (*T*)
12	Repeat steps 7–11 for the same aliquot 10 more times
Graph the sensitivity corrected signal *L*/*T* as a function of the cycle number	

4. Simulation #2: the annealing experiment

Wang et al. (2006b) carried out an experiment in order to investigate the thermal stability of the TT-OSL signal. Measurements were made on naturally irradiated discs of sample IEE266.

These aliquots were preheated for 10 s at 260 °C and then optically stimulated at 125 °C for 300 s in order to measure their natural OSL signal and to reduce the OSL signal to less than 1% of its initial value. The measured natural signal $L_{NATURAL}$ was used to normalize the data from the different aliquots. In the next step of the experiment, the aliquots were annealed for 1 s at temperatures from 200 to 400 °C and the TT-OSL intensity ($L_{TT\text{-}OSL}$) was measured as a function of the annealing temperature. The normalized TT-OSL signal ($L_{TT\text{-}OSL}/L_{NATURAL}$) was obtained as a function of the annealing temperature and the data of Wang et al. (2006b, Fig. 4a) are shown in Fig. 1b. For low temperatures the TT-OSL signal was found to increase with annealing temperature, and to reach a maximum at about 300 °C, with a subsequent fast drop of the signal at higher temperatures. Tsukamoto et al. (2008) carried out similar experiments for several types of quartz and obtained similar results.

We have simulated this experiment using the parameters in Table 1, and the results are shown in Fig. 1b as dashed lines. The parameters used in this simulation are those of Pagonis et al. (2008), and the complete sequence of steps in this simulation is shown in Table 3. Once more the results of the simulation in Fig. 1b follow the same qualitative behavior, but the simulated values of ($L_{TT\text{-}OSL}/L_{NATURAL}$) are much larger than the experimental data.

The simulation results shown in Fig. 1a and b led us to adjust the concentrations of the source trap and BT-OSL trap (N_{10} and N_{11} correspondingly), reducing both of them by a factor of 3.3. One can attribute these differences in the concentrations N_i to natural variations expected in real samples. The new simulations (shown smoothed as a continuous line in Fig. 1a and b) are in reasonable qualitative agreement with the experimental data.

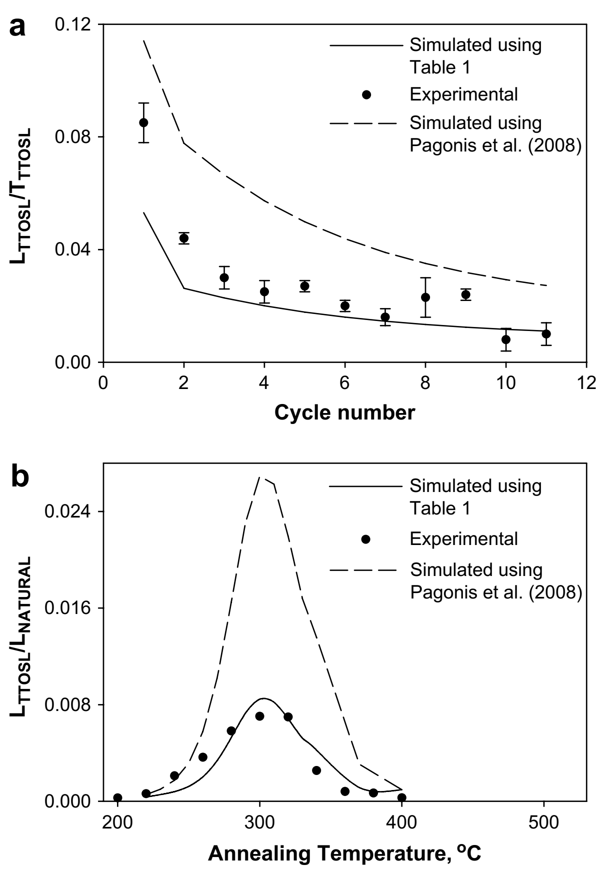

Fig. 1. (a) Simulation of the experiment with repeated cycles of preheating and OSL-bleaching using the parameters of Pagonis et al. (2008) and modified parameters given in bold in Table 1. Also shown are the experimental data from Fig. 5a of Wang et al. (2006b). The detailed steps in the simulation are shown in Table 2. (b) Simulation of the annealing experiment using the parameters of Pagonis et al. (2008) and modified parameters are given in bold in Table 1. Also shown are the experimental data of Wang et al. (2006b), their Fig. 4a. The detailed steps in this simulation are shown in Table 3.

4.1. Further results from the simulations

Fig. 2a shows the charge concentrations n_{10} in the source trap (level 10) and n_{11} in the BT-OSL trap (level 11) at the end of each successive cycle during the repeated cycles experiment shown in Fig. 1a. The values of n_{10} decrease by ~13% in each cycle, while the corresponding values of n_{11} decrease at a much slower rate of ~5%/cycle. This result is consistent with the BT-OSL level being more thermally stable than the source trap, and with the single transfer mechanism proposed by Adamiec et al. (2008).

Fig. 2b shows the corresponding values of n_{10} and n_{11} for the annealing experiment shown in Fig. 1b. It can be seen that both the source trap and the BT-OSL trap contribute to the TT-OSL signal measured during the annealing experiment. The charge in the source trap (n_{10}) contributes more to the low-temperature side of the peak than the charge in the BT-OSL trap (n_{11}), since the former is thermally less stable.

Table 3
The annealing experiment of Wang et al. (2006b) simulated using the model of Pagonis et al. (2008). Steps 1–5 are a simulation of the "natural" quartz sample IEE266 with a natural dose of 409 Gy. This is simulating a multiple aliquot procedure.

1	Natural quartz sample. Set all trap populations to zero
2	Geological dose – 1000 Gy at 1 Gy/s at 20 °C
3	Geological time – heat to 350 °C
4	Illuminate for 100 s at 200 °C
Repeated daylight exposures over a long period of time	
5	Burial dose – 409 Gy at 20 °C at 10^{-6} Gy/s
6	Preheat sample at 260 °C for 10 s
7	Optical bleach in laboratory: Blue stimulation at 125 °C for 300 s – Record NATURAL OSL (0.5 s) ($L_{NATURAL}$)
8	Anneal sample at annealing temperature (200–400 °C in steps of 20 °C) for 1 s
9	Blue stimulation at 125 °C for 100 s – Record OSL (0.5 s) ($L_{TT\text{-}OSL}$)
10	Calculate normalized TT-OSL as $L_{TT\text{-}OSL}/L_{NATURAL}$
Repeat the complete simulation in steps 1–10 using a higher annealing temperature in step 8	

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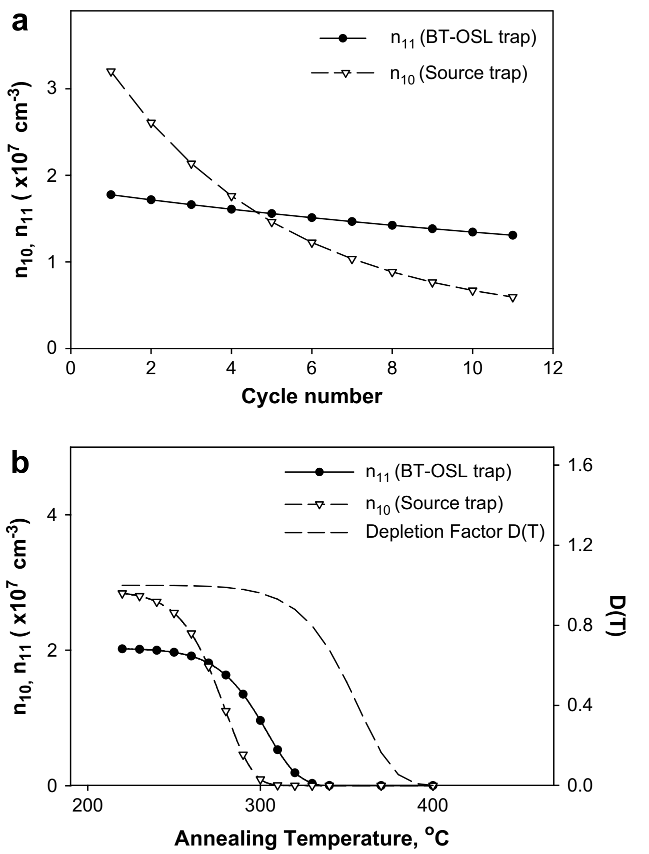

Fig. 2. (a) The simulated temperature dependence of the charge concentrations in the source traps (n_{10}), and the BT-OSL traps (n_{11}), during the repeated cycles experiment of Wang et al. (2006b). (b) Same as (a), for the annealing experiment of Wang et al. (2006b). The slightly modified parameters of Table 1 are used in both simulations shown in (a) and (b).

Also shown in Fig. 2b is the calculated thermal depletion factor $D(T)$ for the charge in the fast OSL trap (level 3). $D(T)$ is calculated assuming first order kinetics and using the expression:

$$D(T) = \exp(-s\exp(-E/kT)t_{Anneal}). \qquad (1)$$

The values of the constants in this expression are taken as the thermal parameters of the fast OSL trap in the model (level 3), namely $E_3 = 1.73$ eV, $s_3 = 6.5 \times 10^{13}$ s^{-1}, and $t_{\mathrm{Anneal}} = 1$ s is the annealing time that was used in the experiments of Wang et al. (2006b). From Fig. 2b it is clear that the fast OSL trap starts depleting at ~300 °C and gradually becomes completely empty at ~400 °C. This result is in agreement with the steep drop on the high temperature side of the data in Fig. 1b.

It is also appropriate to consider the lifetime associated with the source trap. The ReOSL signal not only needs to grow with dose, but also have sufficient thermal stability. However, from Fig. 2b it can be seen that the source trap is much less thermally stable than the fast OSL trap. Using the values of E and s in Table 1 and assuming first order kinetics, we calculate that the lifetimes at 10 °C (a reasonable temperature for a thick loess deposit on the Chinese Loess Plateau) for the source trap and the fast OSL trap are 120 Ma and 3100 Ma, respectively. Whether these lifetimes are relevant can be investigated by applying ReSAR to samples of known age.

5. Simulation #3: the ReSAR protocol

Wang et al. (2007) developed and tested a single-aliquot regenerative-dose (SAR) procedure for measuring the equivalent dose in fine-grained quartz using the ReOSL; it was called the ReSAR protocol. These authors carried out dose recovery experiments and obtained equivalent doses (D_e) using the ReSAR protocol for samples dating back to 100 ka, with D_e ~350 Gy. We have simulated the recovery of a natural dose of 1000 Gy using the ReSAR protocol (Table 1 of Wang et al., 2007) and results of our simulation are shown in Fig. 3. The parameters used in the model are those in Table 1, and the simulated protocol is given in Table 4. The sequence of regenerative doses given to the sample during the ReSAR protocol was 0, 500, 1000, 1500, 0, 500 Gy and the test dose was 7 Gy. The preheat after the regenerated dose was 260 °C for 10 s, and the preheat after the test dose was 220 °C for 20 s.

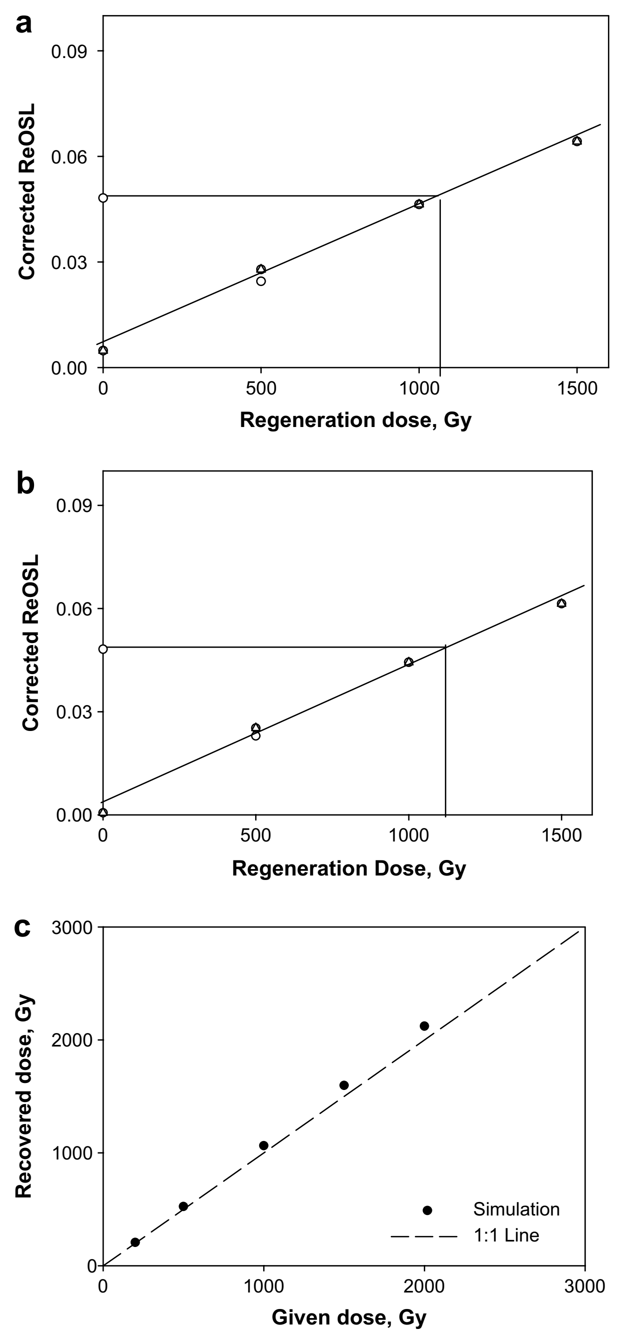

Fig. 3. (a) Simulation of the ReSAR protocol of Wang et al. (2007) shown in Table 4. (b) Same as (a), but including the high temperature step suggested by Tsukamoto et al. (2008). (c) Recovered doses as a function of the given dose for doses between 200 and 3000 Gy.

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Table 4
The ReOSL dating protocol of Wang et al. (2007).

1	Geological dose irradiation of 1000 Gy at 1 Gy/s
2	Geological time – heat to 350 °C
3	Illuminate for 100 s at 200 °C
4	Burial dose – 2000 Gy at 220 °C at 0.01 Gy/s
5	Bleaching of sample: blue stimulation at 125 °C for 2000 s
6	Regenerated dose D_i at 20 °C and at 1 Gy/s
7	Preheat to 260 °C for 10 s
8	Blue stimulation at 125 °C for 270 s
9	Preheat to 260 °C for 10 s
10	Blue stimulation at 125 °C for 90 s (L_{TT-OSL})
11	Test dose TD = 7 Gy
12	Preheat to 220 °C for 20 s
13	Blue stimulation at 125 °C for 90 s (T_{TT-OSL})
14	Anneal to 300 °C for 10 s
15	Blue stimulation at 125 °C for 90 s
16	Preheat at 260 °C for 10 s
17	Blue stimulation at 125 °C for 90 s (L_{BT-OSL})
18	Test dose TD = 7 Gy
19	Preheat to 220 °C for 20 s
20	Blue stimulation at 125 °C for 90 s (T_{BT-OSL})
21	Extra step suggested by Tsukamoto et al. (2008)
Blue stimulation at 280 °C for 90 s	
Repeat steps 6–21 for a sequence of different regenerative doses	
e.g. D_i = (0, 500, 1000, 1500, 0, 500 Gy)	

In the simulation in Fig. 3a, the estimated recovered dose using the ReSAR protocol was 1064 Gy (recovery ratio of 1.06) and the recycling ratio was 0.85. In addition, there was a significant signal when the zero dose step was applied. The simulation was repeated, but adding the high temperature stimulation step recommended by Tsukamoto et al. (2008). They found that by adding an optical bleach of 90 s at 280 °C at the end of the ReSAR protocol, the recycling ratio was improved and recuperation was eliminated. Our simulation (Fig. 3b) gave a slightly improved recycling ratio of 0.90, but a slightly worse recovery ratio of 1.13. However, there is no recuperation following the zero dose step. Fig. 3c gives the results of repeating the simulation in Table 4 for a range of given doses between 200 Gy and 2000 Gy given in step 6 in the first cycle, and using an appropriate range of regenerative doses for the dose response curve to permit interpolation. It can be seen that the ReSAR protocol systematically overestimates the value of the recovered dose for all given doses larger than ~500 Gy.

6. Conclusions

The simulations presented in this paper are consistent with a single charge transfer process of TT-OSL production during the experiments of Wang et al. (2006b). These experiments can be described in a qualitative manner using the model of Pagonis et al. (2008). Using their parameters, the simulations show that during the preheating stage (10 s at 260 °C), ~13% percentage of the charge in the source trap and a smaller percentage (~5%) of the charge in the BT-OSL trap is transferred into the main OSL trap, resulting in the subsequently observed TT-OSL signal. The results of the simulations also show that regenerative doses above ~500 Gy may be overestimated in a systematic manner by the single-aliquot ReSAR protocol.

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