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Keywords

quartz, osl, unstable, component, observation, fast, CAS

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Abstract

Optically stimulated luminescence (OSL) dating has been applied to quartz grains extracted from a sedimentary layer containing stone tools from the bank of Salawusu River, Mu Us desert in central China. Severe age underestimation was observed by applying the single-aliquot regenerative dose (SAR) dating method when compared with the isochron infrared stimulated luminescence (iIRSL) dating results using potassium-rich feldspar grains of different grain sizes. Preheating plateau and dose recovery tests suggest that the SAR protocol is robust for this sample. Component resolving indicates that the OSL signals were dominated by the fast component. However, the fast component is thermally unstable as shown by pulse annealing measurements and single-grain study. This leads to OSL age underestimation. To overcome this problem, a method was proposed by combining equivalent dose (D_e) determination and pulse annealing experiments using single-grain measurements to select only those grains with the thermally stable signals. For those quartz grains with thermally stable OSL signals, the ages obtained are consistent with iIRSL results.

Key words: Quartz; OSL, Fast component, Thermal instability

Introduction

In the last decade, the single-aliquot regenerative-dose (SAR) dating protocol (Murray and Wintle, 2000) has been successfully applied to quartz grains from a wide variety of Quaternary sediments (Murray and Olley, 2002; Vandenberghe et al., 2004). Since the fast component in quartz optically stimulated luminescence (OSL) signals is the most easily bleached by sunlight, an underlying assumption of the SAR protocol is that the signals measured are fast-component dominant (Wintle and Murray, 2006).

Unsuccessful applications have been reported when samples are dominated by non-fast components (Choi et al., 2003; Li and Li, 2006a; Tsukamoto et al., 2007). These have been shown to be associated with the presence of slower decaying components (e.g. medium component and slow components) in the initial OSL signals. The presence of such components could be detected using the D_e (t) plot, D_e value against stimulation time. When the initial signals contain significant contributions from thermally unstable OSL components (e.g. M1 or S3) (Jain et al., 2003; Li and Li, 2006a), the D_e (t) plot would show a decrease and an underestimation of D_e will be resulted. The fast component can be isolated either by mathematically fitting OSL signals (Singarayer and Bailey, 2004; Li and Li, 2006b) or instrumentally using infrared (IR) stimulation (Jain et al., 2003; Fan et al., 2009), although these methods are time-consuming.

Age underestimations using SAR protocol have also been reported in quartz samples for which OSL signals are dominant by the fast component (Buylaert et al., 2007; Murray et al., 2007; Qin and Zhou, 2007). Recently studies on quartz OSL dating of a Danish Eemian (132-116 ka in northern Europe) sediments showed significant age underestimations (Murray and Funder, 2003). Similar trend of age underestimation was also found in quartz samples from Chinese Loess Plateau (Lu et al., 2007). Significant underestimations of quartz D_e were reported for samples in the same region (Buylaert et al., 2007; Qin and Zhou, 2007). Based on comparison of OSL and IRSL dating results and independent age control, Buylaert et al. (2007)

concluded that SAR-OSL dating on quartz in the region should be restricted to samples younger than 40-50 ka. Samples above that age would yield underestimations of more than 30%. In a detailed investigation on the performance of SAR on quartz samples from Chinese Loess Plateau, Qin and Zhou (2007) reported underestimations of 30-50% on samples from palaeosols (S1) bottom and the top of loess layer (L2) with stratigraphical ages around 130ka. It is worth pointing out here that growth curves analysis in above studies showed no sign of saturation in the regenerative dose range adopted in D_e measurements. Kinetic studies on quartz samples from different regions suggest that the lifetime of the fast component of OSL signals should be stable over a few million years (Li and Chen, 2001; Murray and Wintle, 1999).

In this study, we report the observation of thermally unstable OSL signals of quartz grains, which leads to the age underestimations.

2. Samples and Instruments

2.1 Samples

Sample FJGW1 from Fanjiagouwan site at the south edge of the Mu Us Desert was used in this study (Figure 1). Metal pipes were used to extract the sediments. The surface sample at both ends of the pipe was scraped away in the laboratory for dose rate and water content measurements. The central part of the sample was treated with 10% hydrochloric acid (HCl) and 10% hydrogen peroxide (H_2O_2) to remove carbonates and organic materials, respectively. Fractions of grains of different sizes were separated by sieving. Density separations using sodium polytungstate solutions were applied to isolate the K-feldspar (KF) and quartz grains.

For quartz, grain size range of 125-150μm were separated and etched with 40% hydrofluoric acid (HF) solution for at least 40 minutes prior to mounting on aluminum discs. The purpose of the HF etching was to remove any possible feldspar grains and to etch away the alpha irradiated outer layer (about 10μm) of the quartz grains. The

absence of K-feldspar contamination was then checked with IR stimulation on the aliquots (None showed any significant IRSL signals compared with the background). For K-feldspar, grains of 125-150μm was used in K-feldspar SAR-IRSL measurements. A 40 minutes etching using 10% HF solution was used to remove the alpha-irradiated layer and adhesive clays.

2.2 Instruments

The single-aliquot OSL measurements of quartz were performed using a Risø automated TL/OSL system (TL-DA-15) equipped with stimulation units containing blue light-emitting diodes (LEDs, 470 nm) and IR LEDs (880 nm). The total power delivered by the blue LEDs to the sample position was 45mW/cm^2 (Botter-Jensen et al., 2003).The OSL signals were measured through three 2.5-mm-thick U-340 filters and detected using a bialkali EMI 9635Q photomultiplier (PM) tube. Beta irradiation was performed using a ^{90}Sr/^{90}Y beta source delivering 0.08 Gy/s to grains loaded on 9.7-mm-diameter aluminum discs. The K-feldspar grains were measured using another Risø TL/OSL system (TL-DA-12), which a separated calibration of the dose rate for each grains size has been done to fulfill the requirements of iIRSL method (Li et al., 2008b). The IRSL signals were detected through a filter pack of Schott BG-39 and Corning 7-59.

The single-grain OSL measurements were carried out using a Risø automated TL/OSL system (TL-DA-20) with a single-grain attachment. The green laser used for single-grain measurements is a 10 mW Nd:YVO$_4$ solid state diode-pumped laser emitting at 532 nm focused to a spot with diameter about 20μm (Bøtter-Jensen et al., 2003). The maximum energy fluency rate at the sample is around 50W/cm^2 (Duller et al., 1999). The IR laser used for single-grain measurements is a 150 mW IR laser (830 nm), with an additional filter (3mm-thick RG 780) mounted at the end of the rail in the single-grain attachment to eliminate the small resonance of IR laser at 415 nm. All

of the heating were performed at a rate of 5°C/s unless specified.

3. Single-aliquot results

3.1. Age determination using quartz

Before applying the SAR protocol, a preheat plateau test was performed to check whether there was any dependence of D_e on the preheat temperature. As shown in Figure 2a and 2b, the measured D_e values showed no significant changes in the temperature range. Thus, a preheat of 260 ºC for 10s and a cutheat to 220 ºC were used. A dose recovery test was also conducted. The aliquots were first bleached at 125 ºC, which is followed by a laboratory irradiation of 90 Gy. Such a dose was then measured as an "unknown" dose using the same SAR procedure. It was found that the SAR protocol can recover a laboratory given dose (recovery ratio: 1.04±0.03).

For D_e determination, totally 30 aliquots of quartz grains were measured. The results showed good characteristics in SAR protocol, in terms of recycling ratio, recuperation and signal intensity. The recycling ratio (R) yielded values within 0.9-1.1 (R=1.06 in average) and all recuperated signal (i.e. D=0 Gy) yielded values <5% of the natural signals. All these results indicate that the SAR protocol is suitable for the quartz grains of the sample. An equivalent dose of 71.2±2.9 Gy (n=30) was obtained by quartz SAR-OSL, and the derived age is 27.4±1.5 ka (Table 1). The calculated over-dispersion (OD) value is 21±3 (%). The large over-dispersion value indicates a non-single population of D_e values (Figure 3).

3.2. Age determination using K-feldspar

The age of K-feldspar using IRSL signals from our sample was also measured for comparison. The equivalent dose was measured using the SAR protocol described

by Blair et al. (2005), in which a preheat at 280 ºC for 10 s was used following both regenerative and test doses (Li et al., 2007). A central D_e value of 112 ± 2 Gy was obtained. This gives an apparent age of 35.0 ± 2.3 ka using an environmental dose rate of 3.2 Gy/ka (Table 1). It is interesting to note that such an apparent age is already older than the quartz OSL age of 27.4 ± 1.5 ka (Table 1). To test if the IRSL age was underestimated as a result of anomalous fading, fading rate (g value) was measured using the method proposed by Auclair et al. (2003). A fading rate of 3.5 ± 0.5 %/decade was obtained, suggesting that the IRSL age from KF should be younger than the true age. It is concluded that the quartz OSL age (27.4 ± 1.5 ka) has been severely underestimated.

To confirm this phenomenon further, iIRSL dating using different grain sizes (90-125, 125-150, 150-180, 180-212 and 212-250 μm) of K-feldspar, as proposed by Li et al. (2008b), was conducted. It has been shown that such method can avoid the problem of anomalous fading and changes in environmental dose rate (Li et al., 2008a; Li et al., 2008b), and it was successfully applied to sediments from the similar area. The D_e values from of all grain sizes (90-125μm, 125-150μm, 150-180μm, 180-212μm and 212-250μm) were plotted against the internal dose rate calculated using concentrations of 13 ± 1% and 400 μg/g for K and Rb, respectively (Figure 4). An IRSL Isochron age of 54 ± 7 ka was derived. The isochron dating result suggests that the quartz OSL age has been underestimated by about 49%. According to field observation and stratigraphy evidence, it is unlikely that the sample was incompletely bleached before deposition, or has been disturbed after deposition.

3.2. Luminescence dating characteristic

3.2.1 Dose response curves

Four representative growth curves in D_e measurements are shown in Figure 5. Although the curves showed a large aliquot-to-aliquot variation, there was no onset of

saturation. The sensitivity-corrected natural OSL (L_N/T_N) can be interpolated on the growth curves. The normalized OSL signals (L_i/T_i) were fitted using equation: $L_i/T_i=A[1-\exp(-D/D_0)]$, where D_0 is the dose level that is characteristic of the dose response curve and A is a constant related with normalization. The obtained values of $2D_0$ ranged from 127 to 363 Gy, which are larger than the obtained D_e values around 70Gy. Previous study on loess OSL dating using silt-sized quartz has also shown that the saturation dose could be larger than 800 Gy (Watanuki et al., 2003). Therefore, we conclude that underestimation of D_e values is not due to dose saturation.

3.2.2 D_e (t) plot

The underestimation of D_e could be a result of the existence of thermally unstable traps if they contribute a significant amount to the measured OSL. D_e (t) plot is a useful analytical tool to identify if there are any potential effects of such components. Figure 6 shows a typical D_e (t) plot from one aliquot of our sample. Every point was calculated using an integral of 0.2s of the OSL signal at each different stimulation time. Previous researches reported falling patterns in D_e (t) plot, which can be ascribed to the insatiability of S2 (Singarayer and Bailey, 2003) or medium (Li and Li, 2006a) components in their OSL signals. The plot of sample FJGW1 shows no trends of rising or falling in D_e values against stimulation time, indicating there is no apparent discrepancy of absorbed dose in different OSL components at the initial OSL signals.

3.2.3 OSL signal composition

The quartz OSL signals were fitted using several exponentially decaying components (Bailey et al., 1997). At least three components are necessary to fit the CW-OSL curves. The average value of detrapping probability of the fastest component

is 1.5±0.1 for all OSL curves measured, including both natural signals and regenerative ones. Note that the stimulation source used here is the blue LEDs (470nm) set at 50% of its maximum power. A photoionization cross-section α of 2.51±0.22 $\times 10^{-17}$ cm^2 was obtained. This is a representative value for the sample and is consistent with that obtained by previous studies (Jain et al., 2003; Singarayer and Bailey, 2003; Li and Li, 2006b).

Curve fitting was used to investigate relative contribution of each component in the OSL signals. As shown in Figure 7, the fast component contributes to over 90% of the OSL signals measured in the first 0.6 second from the aliquot, which was used in D_e measurement. Therefore, the OSL signal from this sample is fast-component dominant.

3.2.4 Thermal stability

To investigate the thermal stability of measured OSL signals, pulse-annealing experiment was carried out using 4 bleached quartz aliquots from the sample. The detailed pulse annealing experiment procedures are summarized in Table 2. Each aliquot was given a laboratory dose of 32 Gy. The aliquot was then subject to a cut heat to T ºC. The aliquot was stimulated with 100 s blue stimulation at 125 ºC to measure the CW-OSL signal. At the end of each run, the aliquot was bleached with blue stimulation at 280 ºC to remove all potential signals. This measurement cycle was repeated with the cutheat temperature T being increased from 200 to 400 ºC, in increments of 20 ºC. Correction for sensitivity changes during repeated irradiation, heating and illumination in the pulse-annealing experiments was made using OSL response to a test dose of 8 Gy. The luminescence remaining after heating to each temperature was normalized to the initial value. For comparison, pulse annealing results from a heated quartz sample is also shown as aliquot 0# (Figure 8), which OSL signal is typically from stable fast component(Fan et al., 2009).

The normalized OSL signals of aliquots from FJGW1 sample (1#, 3#, 5# and 7#) showed a early decrease at low temperature range (below 260 ºC), which is in contrast with the results of 0# sample. Curve patterns of FJGW1 sample also showed a large aliquot-to-aliquot variation between temperatures ranging from 200 ºC to 300 ºC. Significant reductions in the OSL signals were observed for all aliquots after temperature was raised above 300 ºC.

4. Single-grain results

4.1 Single-grain D_e measurements

Research on single-grain quartz have shown that the luminescence properties, e.g., signal brightness, sensitivity changes, relative proportions of different components and dose responses, can vary greatly from grain to grain (Duller et al., 2000; Bulur et al., 2002). D_e values obtained from single grains could provide higher resolution of variation of luminescence characteristics than the single aliquots of multiple grains. The SAR protocol (Murray and Wintle, 2000) was applied to single grains to obtain D_e values of each grain. Considering the high power of green laser used in single-grain measurement, 1 second stimulation at 125 ºC was adopted. The OSL signal used for dating was the integral of first 0.06 second of the OSL decay curve, with subtracting a background of the last 0.2 second. IR diodes stimulation was performed before measuring the recycling point to check the existence of feldspar contamination using the IR depletion ratio (Duller, 2003). A direct IR laser stimulation was also used after the D_e measurements for checking any feldspar contaminations.

Totally 2500 grains (25 discs, one disc loaded with 10x10 arrays of grains) were measured. 2108 grains were rejected during D_e analysis by at least one of the following rejecting criteria:

1) Initial T_N signal is lower than 3 times of the background counts measured by PM

tube (around 2 counts/0.02s);

2) Not giving a dose response curve;

3) Recycling ratio (IR depletion ratio) falls out of $1\pm10\%$;

4) Recuperation ratio is larger than 10%.

5) No intersection of L_N/T_N on the growth curve.

6) PM counts during IR laser stimulation are higher than 3 times of the background counts measured without IR stimulation;

After screening with the criteria, D_e values of 392 grains were accepted for further analysis (Figure 9). A D_e value of 87.7±3.0 Gy was calculated using the Central Age Model (Galbraith et al., 1999). Although the age of 34 ka derived from the equivalent dose is larger than the average age of 27 ka from single aliquots, it is still 37% lower than the K-feldspar iIRSL age. Despite the underestimation, D_e distribution is much wider than the single-aliquot results. The value of over-dispersion is 45.3%, indicating the possibility of more than one populations existing in the measured grains.

4.2 Single-grain pulse annealing measurements

Pulse annealing analysis was performed on all grains after D_e measurements. Experimental details were similar as mentioned in section 3.2.4, except green laser was used as the stimulation light source for single-grain measurements. Pulse annealing curves from 7 representative grains are shown in Figure 10, with their D_e values indicated on the right. The pulse annealing results of individual grains showed grain-to-grain variations, significantly larger than those observed from single-aliquots (Figure 8). Some grains, e.g. grain 2 and 7 in Figure 10, show typical pulse annealing curves of quartz, i.e. their OSL signals are thermally stable up to 300 ºC, while some grains (e.g. grain 4 and 6 in Figure 10) show an early decrease in their OSL signals in temperature as low as 200 ºC.

In order to characterize the thermal stability of the OSL signals from different grains further, we calculated a thermal remnant ratio (R_T), defined as the ratio of the remnant OSL signals measured after heated to 280 ºC to those measured after heated to 240 ºC. The 392 grains accepted were divided into Group A (107 grains) and Group B (285 grains), respectively. Group A grains have R_T ratios larger than or equal to 0.9; Group B grains have R_T ratios less than 0.9. For a thermally stable OSL signal, it is expected that heating to 280 ºC would not affect the signal. Hence, the R_T ratio should be close to unity, as those grains in Group A. Grains with relatively thermally unstable signals are in Group B.

To check whether the unstable OSL signal observed in group B is a result of presence of thermally unstable component (e.g. ultra-fast, medium or slow components) (Singarayer and Bailey, 2003; Li and Li, 2006a; Jain et al., 2008), the single-grain OSL decay curves in both groups were examined. Figure 11 shows the OSL decay curves of the same grains in Figure 10. There is no distinguishable difference among these grains in the initial part of the decay curves. The OSL signals rapidly decreased to the 5% background level within the first 0.1s of the 1s stimulation (90% power). The OSL curves can be fitted with two exponential decaying components. The calculated detrapping rate for the fast component was 46 s^{-1}, which is similar as reported for a typical quartz sample in previous study (Bulur et al., 2002). This result confirms that the large discrepancy of the thermal stabilities between grains in group A and B is not a result of presence of the thermally unstable components of ultra-fast component or medium component.

4.3 Relationship between D_e and thermal stability

In addition to the differences in thermal stability, grains in group B give smaller D_e values than those in Group A. This indicates that the underestimation of D_e relates to the thermal stability of OSL signals. D_e results of the 392 grains were

plotted in Figure 12. The grains in Group A are shown as filled circles and grains in Group B as open triangles. Central D_e and the corresponding over-dispersion values from both groups are also summarized in Table 3.

For group A, the central D_e value of 147.1±2.6 Gy with over-dispersion value of 13.1% was obtained. The small over-dispersion value suggested that the D_e values measured was dominated by one population. The derived central age is 56.6±2.4 ka, which is consistent with the results obtained by iIRSL dating (54 ±7 ka). However, for group B, the calculated central D_e is 69.8±2.4 Gy, which give an age of 26.8±1.4 ka. There is still a large scatter of the D_e distribution as indicated by the over-dispersion value (37.7%).

In calculating the value of R_T, the temperatures of 280 and 240 ºC were chosen arbitrarily. It is necessary to test whether the D_e results are influenced by the temperatures chosen for calculation. Here we re-calculate the value of R_T as the ratio of the remnant OSL signals measured after heating to 280 ºC and those after heating to 200 ºC, and re-group the grains using the new criterion, as shown in Table 3. Although there is a slightly difference in the number of grains accepted in each group (Group A and A*), the derived central D_e and over-dispersion values are indistinguishable. We conclude that the D_e values obtained for both groups does not change when different criterion are used. The R_T ratio ($OSL_{280ºC}/OSL_{240ºC}$) can be adopted to distinguish the grains with stable OSL signal from the ones with unstable signals.

5. Discussion

The studying sample gives an apparent IRSL age of 35.0±2.3 ka for KF and an age of 27.4±1.5 ka for quartz using SAR protocol. It is expected that the quartz OSL age should be older than the apparent KF IRSL age, because of anomalous fading effect in K-feldspar. Such discrepancy indicated our quartz OSL age was

severely underestimated. This was further supported by our iIRSL dating result from different grain sizes K-feldspar.

The underestimation of quartz OSL age is not due to feldspar contamination, because the quartz sample has been checked using the IR depletion ratio (Duller, 2003) in the SAR protocol. The age underestimation is not a result of other thermally unstable signals, such as ultra-fast, medium and slow components. The ultra-fast component can be removed effectively by heating to 220°C (Jain et al., 2003). A preheat at 260°C has been applied in our D_e measurements. The medium component (Li and Li, 2006a) and the slow component (Jain et al., 2003) could also result in D_e underestimation, which could be manifested by the D_e (t) plot. D_e (t) plot for our sample (Figure 6) showed no dependence of D_e values on the stimulation time. This is further supported by curve-fitting analysis on the CW-OSL curves (Figure 7). It is shown that the total contribution from medium or other slower components in the first 0.2s is estimated to be less than 5% of the total signals. It is also noted that a photoionization cross-section α of $2.51\pm0.22 \times 10^{-17}$ cm^2 was obtained from the single-aliquot CW-OSL curves from quartz sample, which is consistent with the value of the fast component obtained in previous studies (Jain et al., 2003; Singarayer and Bailey, 2003; Li and Li, 2006b). Therefore, the only way yet to discriminate the unstable component from the stable ones for our sample is their difference in thermal property, not by their decay rates. Sensitivity change during the measurement of the OSL signals also can result in D_e underestimation. This phenomenon can be tested by the recycling ratio in the SAR protocol. Quartz sample used in this study has a recycling ratio of 1.06, which indicates that the sensitivity correction is robust in SAR D_e measurements. Therefore, we conclude that the D_e underestimation, when using SAR protocol of fast component in quartz OSL, is a result of lack of thermal stability of OSL signals in the fast component from some of quartz grains. Such unstable signal cannot eliminated completely by applying high preheat temperatures (Figure 2a), although a noticeable increase in D_e with higher preheat temperature could be

achieved. Our single-grain measurements show that there is a large variety in the thermal stability from grain to grain (Figure 10). The pulse annealing results indicated that the OSL from some quartz grains is unstable at heating temperature as low as 200°C.

The OSL signals from a single aliquot, which contains a large number of grains (500-1000), would inevitably have contributions from the grains with unstable OSL signals. The D_e measured is thus underestimated. By conducting single-grain pulse annealing measurements, those grains with stable and unstable OSL signal could be distinguished. By selecting those grains with stable OSL signals, an age of 56.6±2.4 ka was obtained. This is in excellent agreement with the isochron dating result (54 ±7 ka) obtained using KF.

In a comparison of quartz OSL ages and KF IRSL ages (Buylaert et al., 2007), underestimations in quartz grains were found in samples with expected ages >70 ka. A similar underestimation was also reported by Lai (2010). By comparing OSL ages with independent ages from palaeo-magnetic data, Lai (2010) pointed out that the reliable ages obtained in quartz from the Luochuan section is younger than 70 ka (i.e. 230 Gy). Our sample was taken from the south edge of the Mu Us Desert, which is adjacent to the north part of the Chinese Loess Plateau. It is believed that the materials in the Chinese Loess Plateau is partly originated from the same as those in Mu Us Desert (Sun, 2002). It is therefore expected that a similar luminescence behavior would be observed in both areas. Our results suggest that the thermal instability in the quartz OSL signal could explain the underestimation observed in the Chinese Loess Plateau and the adjacent areas.

6. Conclusions

Severe age underestimation accompanied with large scatter in D_e values was observed when the SAR dating protocol was applied to the quartz grains from a sample from Mu Us Desert, northern China. This is attributed to the unstable fast

component from a part of quartz grains, indicated by pulse annealing results. The grains with stable OSL signals can be separated in single-grain pulse annealing measurements. The age calculated from these grains with stable OSL signals is in agreement with the feldspar iIRSL dating results.

References

Adamiec, G., Aitken, M. J., 1998. Dose-rate conversion factors: update. Ancient TL 16, 37-50.

Auclair, M., Lamothe, M., Huot, S., 2003. Measurement of anomalous fading for feldspar IRSL using SAR. Radiation Measurements 37, 487-492.

Bailey, R. M., Smith, B. W., Rhodes, E. J., 1997. Partial bleaching and the decay form characteristics of quartz OSL. Radiation Measurements 27, 123-136.

Blair, M. W., Yukihara, E. G., McKeever, S. W. S., 2005. Experiences with single-aliquot OSL procedures using coarse-grain feldspars. Radiation Measurements 39, 361-374.

Botter-Jensen, L., Andersen, C. E., Duller, G. A. T., Murray, A. S., 2003. Developments in radiation, stimulation and observation facilities in luminescence measurements. Radiation Measurements 37, 535-541.

Bulur, E., Duller, G. A. T., Solongo, S., Botter-Jensen, L., Murray, A. S., 2002. LM-OSL from single grains of quartz: a preliminary study. Radiation Measurements 35, 79-85.

Buylaert, J. P., Vandenberghe, D., Murray, A. S., Huot, S., De Corte, F., Van den Haute,

P., 2007. Luminescence dating of old (> 70ka) Chinese loess: A comparison of single-aliquot OSL and IRSL techniques. Quaternary Geochronology 2, 9-14.

Choi, J. H., Murray, A. S., Cheong, C. S., Hong, D. G., Chang, H. W., 2003. The resolution of stratigraphic inconsistency in the luminescence ages of marine terrace sediments from Korea. Quaternary Science Reviews 22, 1201-1206.

Duller, G. A. T., Botter-Jensen, L., Murray, A. S., Truscott, A. J., 1999. Single grain laser luminescence (SGLL) measurements using a novel automated reader. Nuclear Instruments & Methods in Physics Research Section B-Beam Interactions with Materials and Atoms 155, 506-514.

Duller, G. A. T., Botter-Jensen, L., Murray, A. S., 2000. Optical dating of single sand-sized grains of quartz: sources of variability. Radiation Measurements 32, 453-457.

Duller, G. A. T., 2003. Distinguishing quartz and feldspar in single grain luminescence measurements. Radiation Measurements 37, 161-165.

Fan, A. C., Li, S.-H., Li, B., 2009. Characteristics of quartz infrared stimulated luminescence (IRSL) at elevated temperatures**.** Radiation Measurements 44, 434-438.

Galbraith, R. F., Roberts, R. G., Laslett, G. M., Yoshida, H., Olley, J. M., 1999. Optical dating of single and multiple grains of quartz from Jinmium rock shelter, northern austrilia: Part I, Experimental design and statistical models. Archaeometry 41, 339-364.

Jain, M., Murray, A. S., Botter-Jensen, L., 2003. Characterisation of blue-light stimulated luminescence components in different quartz samples: implications for dose measurement. Radiation Measurements 37, 441-449.

Jain, M., Choi, J. H., Thomas, P. J., 2008. The ultrafast OSL component in quartz: Origins and implications. Radiation Measurements 43, 709-714.

Lai, Z., 2010. Chronology and the upper dating limit for loess samples from Luochuan section in the Chinese Loess Plateau using quartz OSL SAR protocol. Journal of

Asian Earth Sciences 37, 176-185.

Li, B., Li, S. H., 2006a. Comparison of D-e estimates using the fast component and the medium component of quartz OSL. Radiation Measurements 41, 125-136.

Li, B., Li, S. H., Wintle, A. G., Zhao, H., 2007. Isochron measurements of naturally irradiated K-feldspar grains. Radiation Measurements 42, 1315-1327.

Li, B., Li, S. H., Wintle, A. G., 2008a. Overcoming environmental dose rate changes in luminescence dating of waterlain deposits. Geochronometria 30, 33-40.

Li, B., Li, S. H., Wintle, A. G., Zhao, H., 2008b. Isochron dating of sediments using luminescence of K-feldspar grains. Journal of Geophysical Research-Earth Surface. 113, F02026, doi:10.1029/2007JF000900.

Li, S. H., Chen, G., 2001. Studies of thermal stability of trapped charges associated with OSL from quartz. Journal of Physics D-Applied Physics 34, 493-498.

Li, S. H., Li, B., 2006b. Dose measurement using the fast component of LM-OSL signals from quartz. Radiation Measurements 41, 534-541.

Lu, Y. C., Wang, X. L., Wintle, A. G., 2007. A new OSL chronology for dust accumulation in the last 130,000 yr for the Chinese Loess Plateau. Quaternary Research. 67, 152-160.

Murray, A. S., Wintle, A. G., 1999. Isothermal decay of optically stimulated luminescence in quartz. Radiation Measurements 30, 119-125.

Murray, A. S., Wintle, A. G., 2000. Luminescence dating of quartz using an improved single-aliquot regenerative-dose protocol. Radiation Measurements 32, 57-73.

Murray, A. S., Olley, J. M., 2002. Precision and accuracy in the optically stimulated luminescence dating of sedimentary quartz: a status review. Geochronometria 21, 1-16.

Murray, A. S., Funder, S., 2003. Optically stimulated luminescence dating of a Danish Eemian coastal marine deposit: a test of accuracy. Quaternary Science Reviews 22, 1177-1183.

Murray, A. S., Svendsen, J. I., Mangerud, J., Astakhov, V. I., 2007. Testing the accuracy

of quartz OSL dating using a known-age Eemian site on the river Sula, northern Russia. Quaternary Geochronology 2, 102-109.

Qin, J. T., Zhou, L. P., 2007. Optically stimulated Luminescence Dating of Upper Part of a Thick Loess Section at Caoxian near The Northern Desert of China. Quaternary Sciences 27, 546-552 (In Chinese, with English abstract).

Singarayer, J. S., Bailey, R. M., 2003. Further investigations of the quartz optically stimulated luminescence components using linear modulation. Radiation Measurements 37, 451-458.

Singarayer, J. S., Bailey, R. M., 2004. Component-resolved bleaching spectra of quartz optically stimulated luminescence: preliminary results and implications for dating. Radiation Measurements 38, 111-118.

Sun, J. M., 2002. Provenance of loess material and formation of loess deposits on the Chinese Loess Plateau. Earth and Planetary Science Letters 203, 845-859.

Tsukamoto, S., Murray, A. S., Huot, S., Watanuki, T., Denby, P. M., Botter-Jensen, L., 2007. Luminescence property of volcanic quartz and the use of red isothermal TL for dating tephras. Radiation Measurements 42, 190-197.

Vandenberghe, D., Kasse, C., Hossain, S. M., De Corte, F., Van den Haute, P., Fuchs, M., Murray, A. S., 2004. Exploring the method of optical dating and comparison of optical and C-14 ages of Late Weichselian coversands in the southern Netherlands. Journal of Quaternary Science 19, 73-86.

Watanuki, T., Murray, A. S., Tsukamoto, S., 2003. A comparison of OSL ages derived from silt-sized quartz and polymineral grains from Chinese loess. Quaternary Science Reviews 22, 991-997.

Wintle, A. G., Murray, A. S., 2006. A review of quartz optically stimulated luminescence characteristics and their relevance in single-aliquot regeneration dating protocols. Radiation Measurements 41, 369-391.

Figure captions

Figure 1. Location of sample FJGW1. Fanjiagouwan is an archaeological site on the bank of Salawusu River, where stone tools were found.

Figure 2. (a) Preheat tests on sample FJGW1 of temperature ranging from 220 ºC to 280 ºC. The equivalent dose at each temperature was measured with 4 aliquots. The average values of the 4 aliquots are presented with standard error. (The overall average value is also indicated as the dashed line). (b) The average recycling ratio (filled circles) and recuperation ratio (filled squares) obtained at each preheat temperature.

Figure 3. Age distribution (30 aliquots) obtained with SAR-OSL for quartz grains. Dashed lines show the ages obtained using IRSL and iIRSL dating of K-feldspar of the same sample, respectively (data listed in Table 1).

Figure 4. The Isochron plots showing the D_e values of K-feldspar grains of different size plotted against the internal beta dose rate calculated by assuming that the concentrations of K and Rb are 13% and 400 µg/g, respectively. Filled symbols are measured data. The full line is the best fitted line of the data points. The dashed line is calculated using the method described by Li et al. (2008b) and the age calculated from the difference between the slopes from the two lines.

Figure 5. Growth curves of 4 representative aliquots in quartz single-aliquot measurements.

Figure 6. Typical D_e (t) plot of a quartz aliquot of sample FJGW1.

Figure 7. Relative contributions from different components in the CW-OSL decay curve from one a aliquot, plotted against the stimulation time.

Figure 8. Pulse-annealing curves for four aliquots (1#, 3#, 5#, 7#) from sample FJGW1, plotted as the remnant luminescence versus annealing temperature. (Integral of the first 0.2 s OSL signals was used). Aliquot 0# is a heated quartz sample with stable OSL signals for comparison.

Figure 9. Radialplot of equivalent dose values for all 392 grains of FJGW1 after sceening with the criteria. D_e values falling in the 2σ region of the central D_e are identified as filled circles.

Figure 10. Pulse-annealing curves for 7 representative grains in sample FJGW1, plotted as the remnant luminescence versus annealing temperature. Integral of the first 0.02 s OSL signals was used.

Figure 11. OSL decay curves for grains in Figure 10 measured during the D_e determination. The data was normalized to the largest OSL signal point of each grain.

Figure 12. Radialplot of equivalent dose values for all 392 grains of FJGW1. Grains in Group A and Group B are labeled as filled circles and open triangles, respectively.

Table 1. Summary of single-aliquot dating results using quartz and KF extracted from FJGW1 using SAR technique.

Dating material	alpha counting rate[a] counts/ks	K content (%)	Water content (%)	Internal dose rate[b] (Gy/ka)	Cosmic ray (Gy/ka)	Equivalent dose (Gy)	Dose rate (Gy/ka)	OSL age (ka)
Quartz	6.2±0.2	1.6±0.1	10±5	-	0.2	71.2±2.9	2.6±0.1	27.4±1.5
K-feldspar	6.2±0.2	1.6±0.1	10±5	0.6 ±0.1	0.2	112 ± 2[c]	3.2±0.2	35.0±2.3

[a]The alpha counting rate is measured using 42-mm-diameter ZnS screens. The counts were converted into alpha, beta and gamma contributions with the conversion factors given by Adamiec and Aitken (1998).

[b]The internal dose rate calculated using concentrations of 13±1% and 400 ug/g for K and Rb, respectively

[c]The apparent D_e of KF (without any correction for anomalous fading)

Table 2. The pulse annealing measurement procedures

Step	Treatment	Observed
1	Give regenerative dose, $D_0{}^a$	
2	Heat to T °C^b	
3	OSL measurement at 125°C for 100 s	L_i
4	Give test dose, $D_t{}^c$	
5	Cut-heat to 200 °C	
6	OSL measurement at 125°C for 100 s	T_i
7	Blue LED bleach at 280 °C for 40 s	
8	Return to step 1	

^{a}Given dose D_0 is 32 Gy.

^{b}T °C is from 200 to 400°C in steps of 20°C.

^{c}Test dose for each cycle: $D_t = 8$ Gy.

Table 3. Number of grains in Group A (stable) and Group B (unstable) identified using the R_T ratio. Central D_e, over-dispersion values and the corresponding ages from both groups are also presented.

Grains	Number	D_e (Gy)	Over-dispersion (%)	Age (ka)
Total	392	87.7±3.0	45.3	33.7
Group B	285 (73%)	69.8±2.4	37.7	26.8
Group A	107 (27%)	147.1±2.6	13.1	56.6
Group A*	88 (22%)	143.8±5.2	15.6	55.3

*Showing the results when a different R_T ratio was used ($OSL_{280°C}/OSL_{200°C}$).
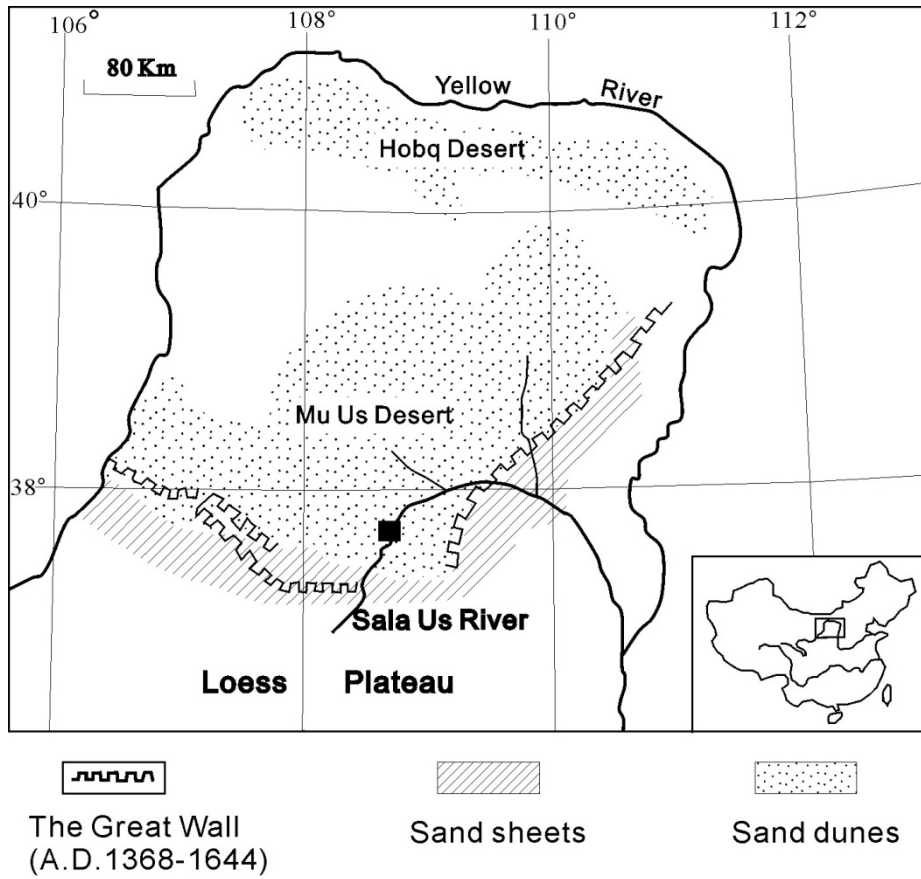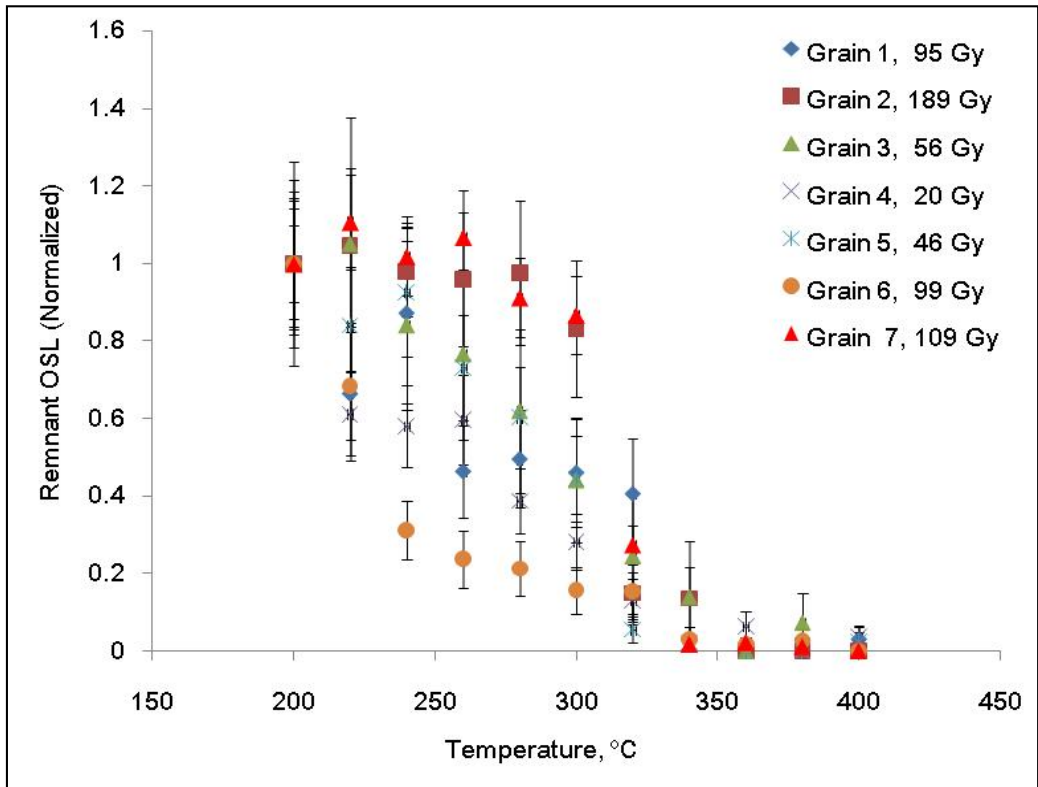
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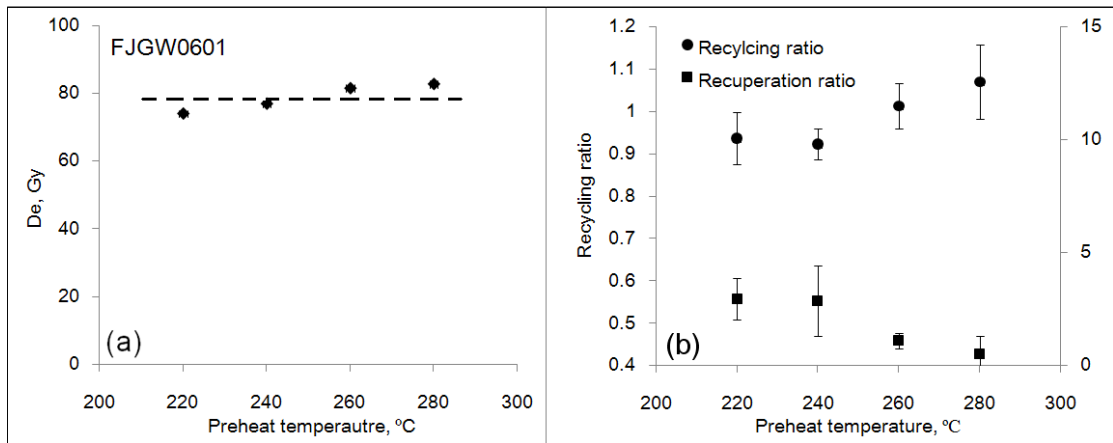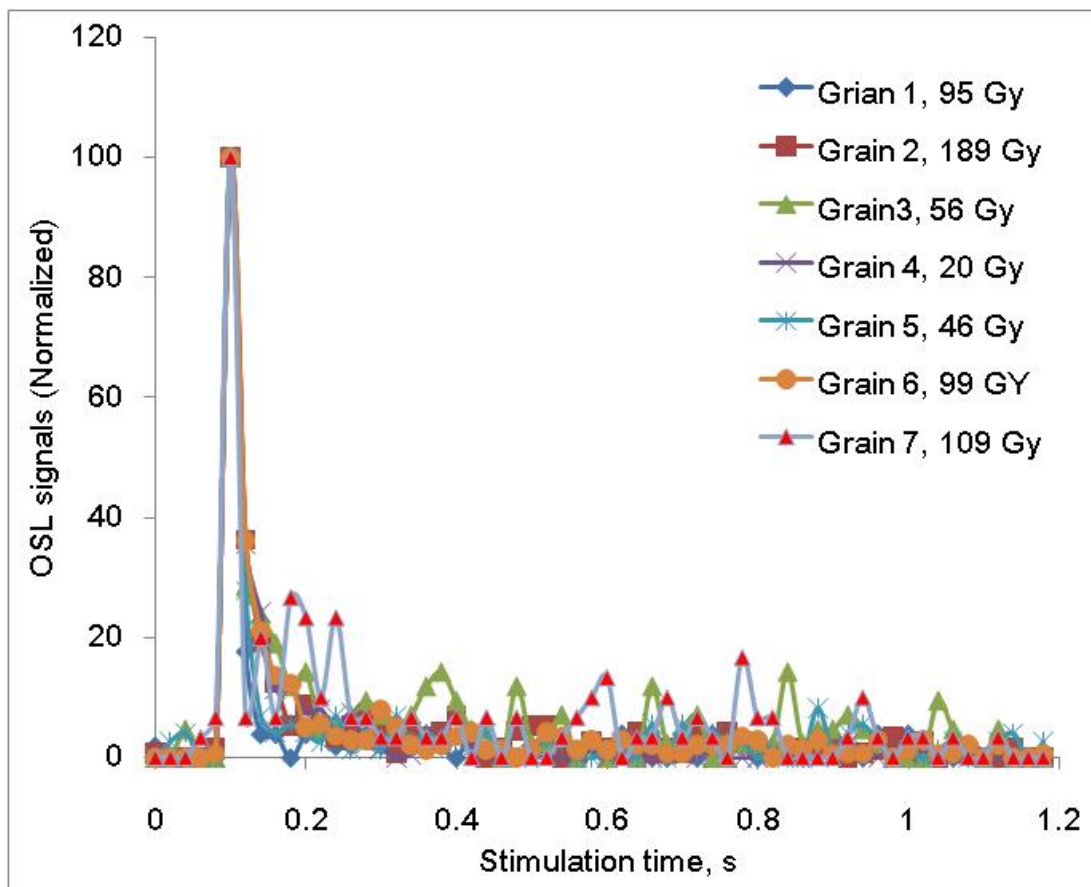
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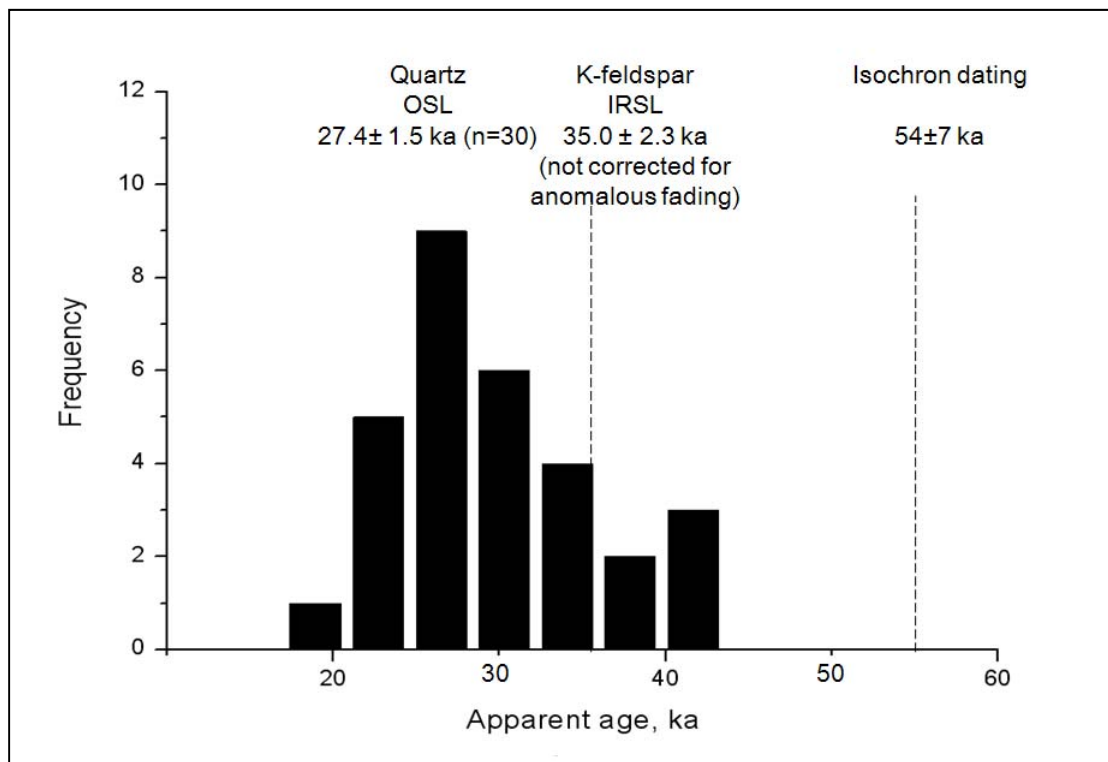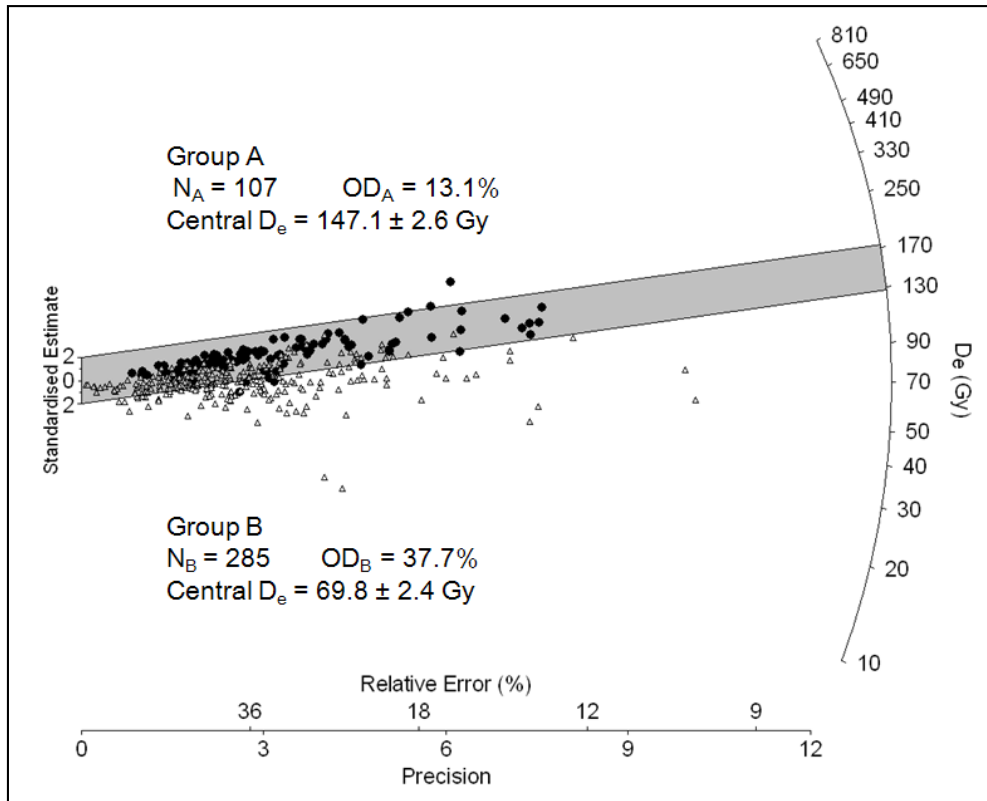
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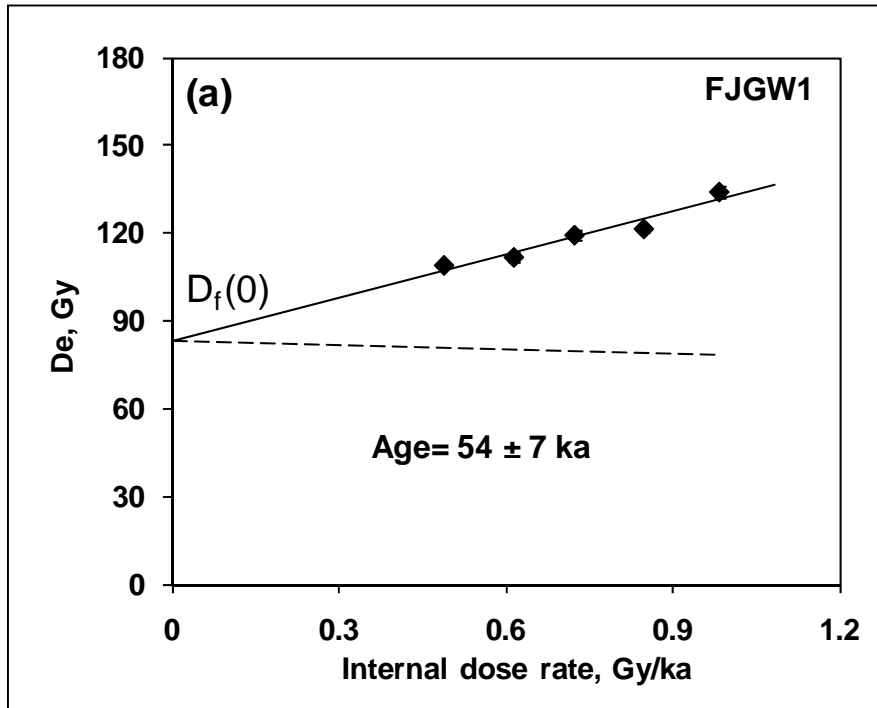
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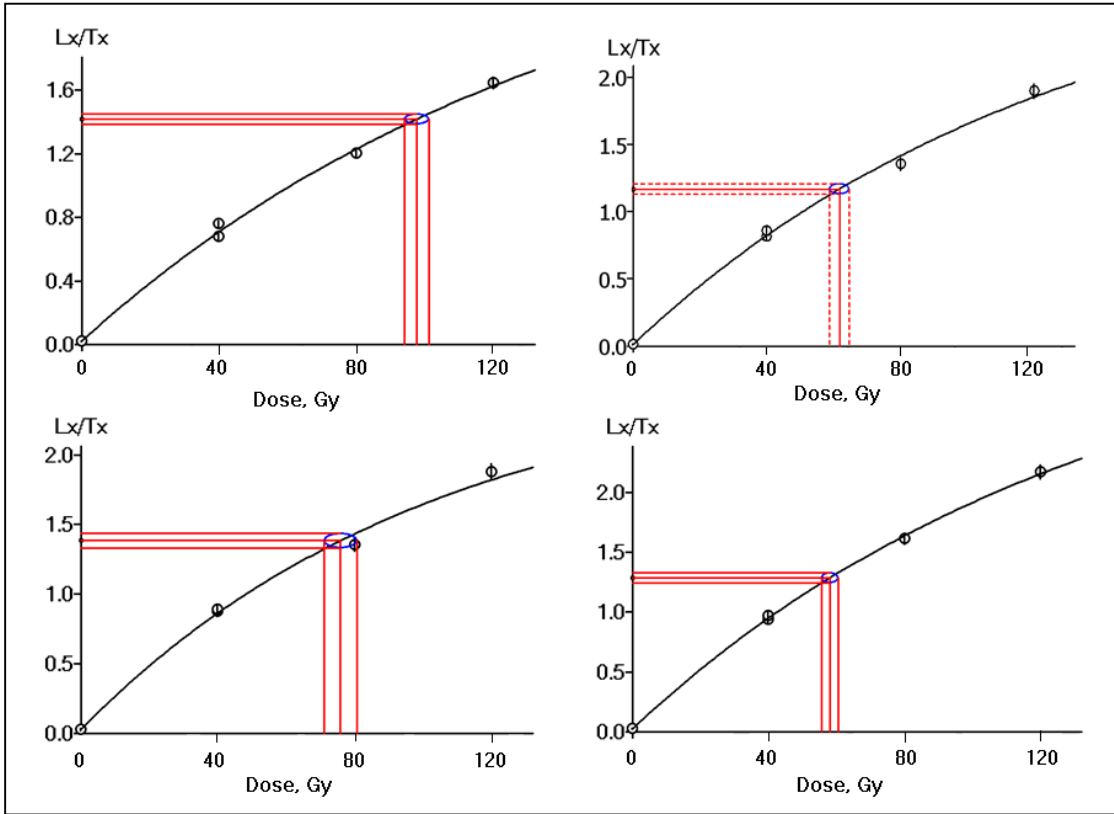
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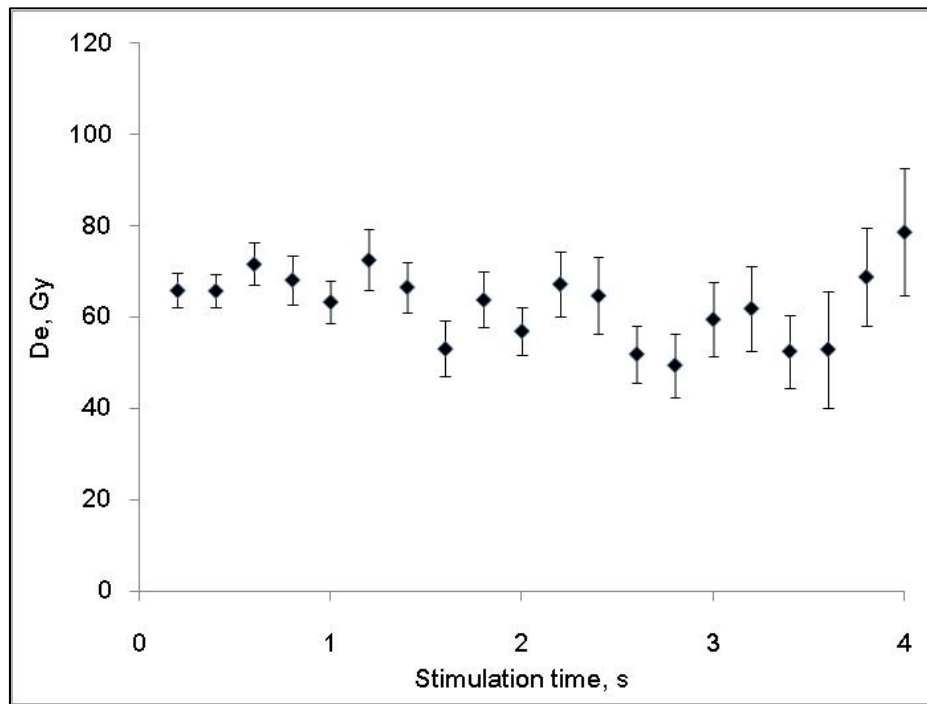
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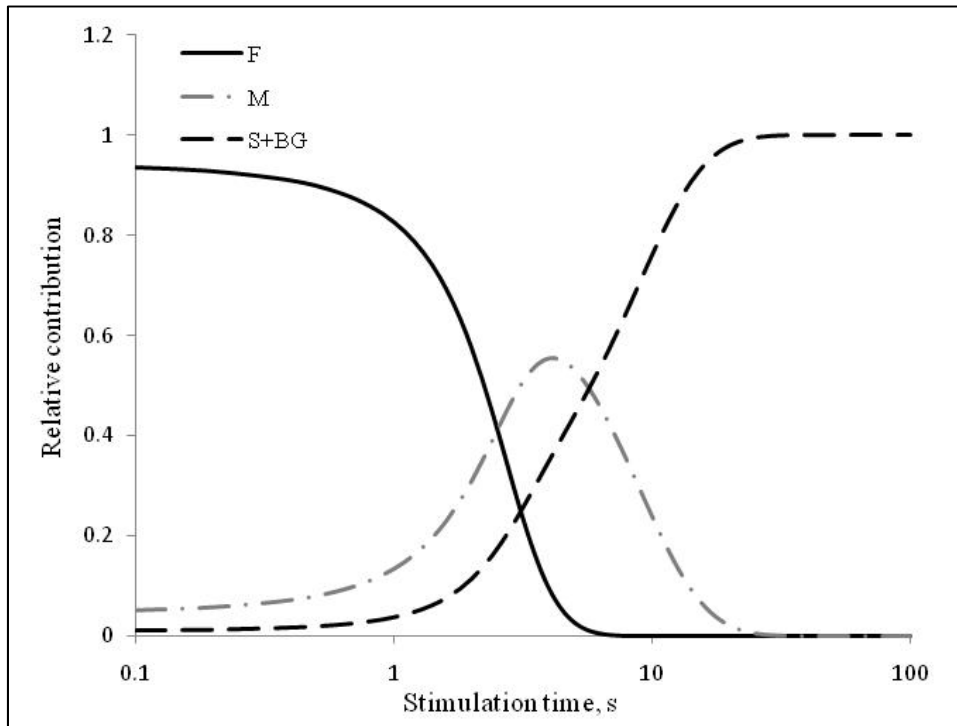
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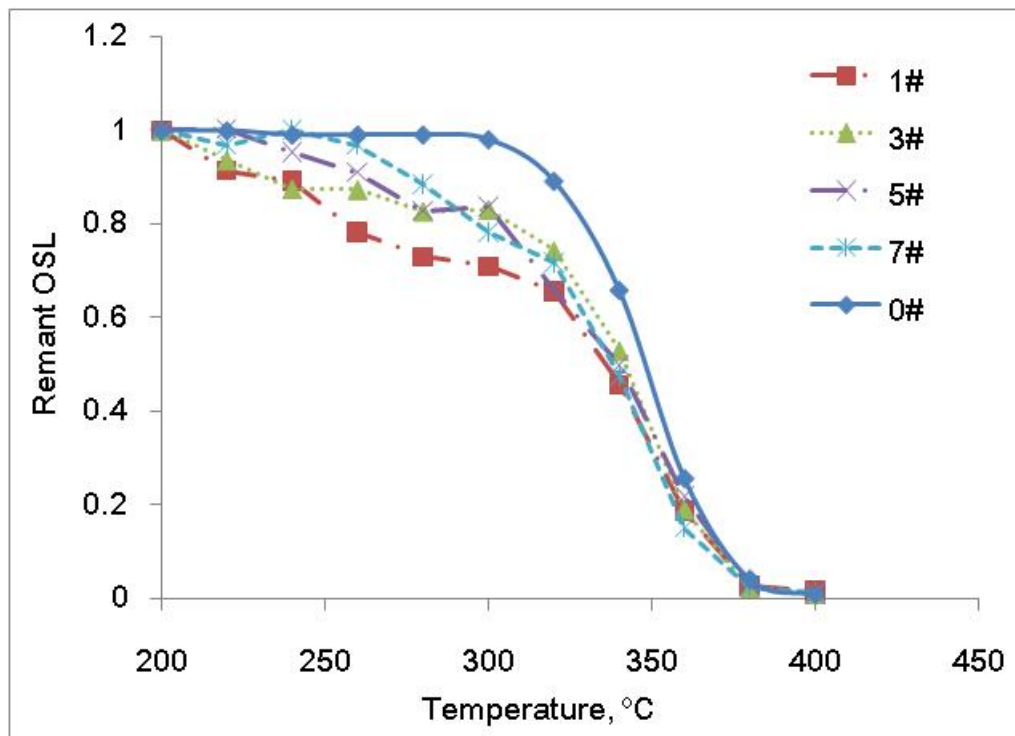
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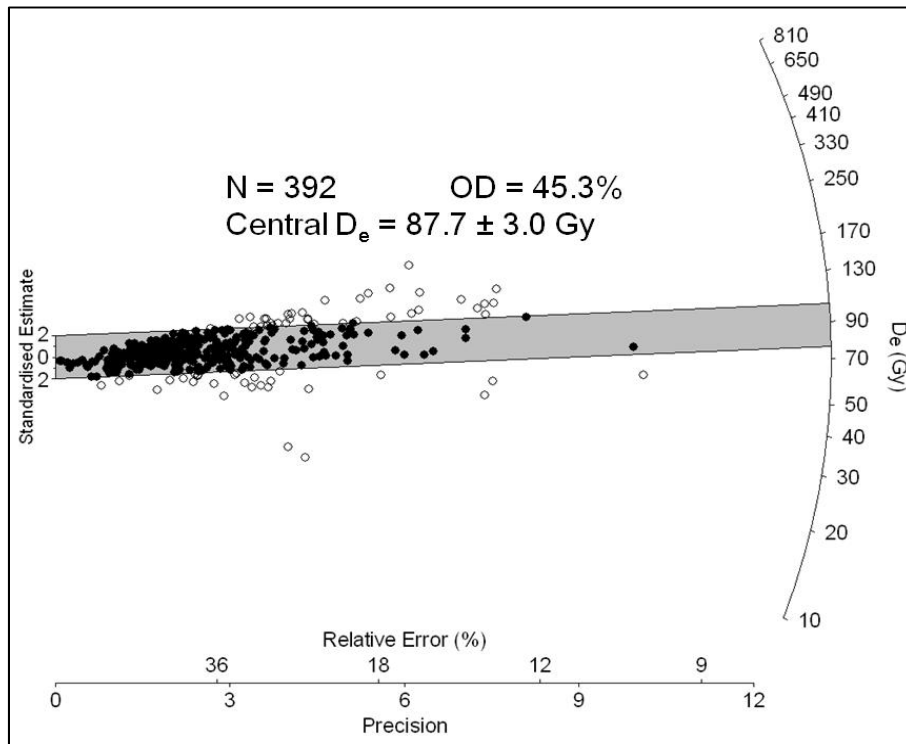
Figure 9

Figure 10

Figure 11

Figure 12