Effect of single-grain versus multi-grain aliquots in determining age for K-feldspars from southwestern British Columbia

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Introduction

Using simulated data, Arnold and Roberts (2009) have argued that multi-grain aliquots, because of averaging effects among grains, cannot correctly distinguish different dose components in mixed-age samples. They argued that averaging effects may have less effect for determining the correct depositional age of partially bleached sediments, using the minimum age model (MAM), provided that partial bleaching is the main cause of dispersion. This paper presents empirical evidence to show that MAM on multi-grain aliquots may produce grossly over-estimated ages even from small aliquots in the range of 2-3 grains each. The samples are K-feldspar extracts from fluvial sediments collected from the Chilliwack Valley, southwestern British Columbia.

The Chilliwack Valley is a large mountainous watershed in the North Cascade Mountains. The valley was under 2 km of ice during the last glacial maximum, but during a late glacial phase of alpine glaciation, a series of retreats and advances of Fraser ice into Chilliwack Valley led to a moraine blocking the valley mouth and to the development of a sequence of intermittent glacial lakes and an outwash plain in the mid-valley (Saunders et al., 1987). With further ice retreat, valley base-level was lowered considerably (100 m or more) as the Chilliwack River began to cut down through (or incise) these glacial sediments, a process that extended well into the Holocene (Tunnicliffe et al., submitted). To strengthen the chronology of these events, nine luminescence samples were collected from a series of mid-valley terraces deposited during the incision process. Dates for eight of these samples are evaluated here.

Procedures

The samples form two sets, UW1326-29, which were collected in summer 2005, and UW1863-66, which were collected in spring 2008. All were retrieved from between 35 and 60 cm below the modern surface. Because quartz in these samples had little sensitivity using UV emission, K-feldspars were prepared. For UW1326-29, both 125-150µm and 180-212µm fractions were prepared, while for UW1863-66 only the larger fraction was prepared. This report focuses on the differences in results between the two size fractions of UW1326-29, mentioning the results of the other samples only for comparison.

Luminescence was measured using the single-grain attachment to a Risø TL/OSL DA-20 reader, using a 150mW 830nm IR laser for excitation. Because the single-grain disks have 300µm diameter holes, the 180-212µm grains generally provide single-grain resolution, but 2-3 grains of the 125-150µm grains will fit in each hole, providing effectively small multi-grain aliquots.

K-feldspars were isolated by sieving, treatment with HCl and H_2O_2, and density separation using a lithium metatungstate solution of 2.58 specific gravity. No HF was applied. For measuring luminescence, the laser was passed through a RG780 filter and set at variable power from 30-90%. A 7.5 mm blue filter pack (Scott 7-59 and BG-39) allowed emission in the 350-450 nm range. The variable power did not appear to have a detectable effect. Equivalent dose (D_e) values for two samples were 21.5±2.1 and 5.8±1.0 Gy for 30% power and 18.1±1.6 and 5.8±0.9 Gy for 70% power, respectively. The values were based on 20-60 grains for each sample, using the central age model. No significant differences in over-dispersion or ages corrected for anomalous fading were detected either.

D_e was determined using a single aliquot regenerative dose (SAR) method as adapted for feldspars (Auclair et al. 2003), with stimulation at 50°C and using a preheat of 250°C for 1 minute (after either

Table 1: *Luminescence dating data for samples arranged in stratigraphic order of terraces from oldest to youngest*

Sample	Terrace	U (ppm)	Th (ppm)	K (%)	Total dose rate (Gy/ka)*	Grain size (µm)	N	Age (ka) Central age model	Over-dispersion (%)	Age (ka) Minimum age model
UW1326	T1-outwash surface	1.38±0.11	3.77±0.76	1.40±0.01	2.64±0.20	180-212	36	10.9±0.7	1.3	11.5±2.2
					2.52±0.16	**125-150**	**245**	**87.1±4.2**	**53.3**	**36.1±2.8**
UW1863	T2-upper	1.03±0.08	1.63±0.49	1.35±0.05	2.27±0.05	180-212	108	15.1±2.2	132.6	2.9±0.8
UW1866	T2-upper	1.58±0.11	1.90±0.54	1.58±0.08	2.44±0.05	180-212	197	28.0±2.8	119.0	5.0±0.9
UW1327	T3-mid	1.39±0.10	1.85±0.53	1.21±0.05	2.36±0.20	180-212	86	23.8±3.4	119.1	3.5±0.7
					2.24±0.15	**125-150**	**328**	**64.1±2.7**	**65.6**	**20.0±1.3**
UW1865	T3-mid	1.85±0.17	9.77±1.17	1.73±0.08	2.34±0.06	180-212	188	24.9±2.2	106.3	4.4±0.6
UW1328	T3a-mid-eroded edge	1.76±0.12	2.90±0.68	1.20±0.04	2.69±0.20	180-212	85	30.4±4.4	117.7	4.3±0.8
					2.57±0.16	**125-150**	**250**	**46.6±3.1**	**87.0**	**10.5±1.0**
UW1864	T3a-mid-eroded edge	0.96±0.07	1.56±0.44	1.63±0.11	2.78±0.06	180-212	117	15.6±1.8	109.5	3.0±0.5
UW1329	T4-lower	1.70±0.12	3.02±0.68	1.21±0.02	1.95±0.07	180-212	100	11.5±0.6	109.6	1.1±0.2
					1.84±0.07	**125-150**	**38**	**31.6±5.4**	**91.3**	**7.0±1.8**

* For the alpha contribution, a b-value of 1.9 ± 1.0 was used to correct for lower efficiency. The dose rate is slightly higher for larger grains because of grain size effect on attenuations and internal K contribution.

regeneration or test dose). Anomalous fading rates were measured using the procedures of Auclair et al. (2003) for single aliquots. Ages were corrected following Huntley and Lamothe (2001). Storage times after irradiation of up to 3-5 days were employed to determine fading.

Dose rates were measured in the laboratory by alpha counting, beta counting and flame photometry and in situ by placing $CaSO_4$:Dy dosimeters. The dosimeters measured a somewhat higher external dose rate than that calculated from the laboratory measurement of the samples themselves, making a difference of about 12% in the total dose rate. The rate from the dosimeters, as a more direct measure, was used in the age calculation. The internal beta dose rate from K-feldspar grains was evaluated by determining the K-content on 39 grains (180-212µm) of samples UW1327-8 (also used for D_e measurement) using X-ray energy dispersive analysis attached to a scanning electron microscope. The K content averaged 8.2 ± 2.9%, compared to 14% for pure orthoclase. The dose rates in Table 1 were calculated using the former value. A moisture content of 10 ± 5% was used, which was about 2-5% higher than the measured values from summer-collected samples.

Results

Table 1 gives the samples, their provenance and a summary of results. The discussion here focuses on the age distributions. Aside from measurement of internal K on a few grains, no attempt (nor the ability to do so) was made to measure dose rate at a single-grain scale, so a bulk dose rate for each sample was used in the age calculations.

A well-noted advantage of single-grain dating is the opportunity to remove from analysis grains with unsuitable characteristics as judged by failure to meet a set of criteria. Grains were eliminated from this analysis if they (1) had poor signals (as judged from net natural signals less than three times above the standard deviation of the background), (2) had recycle ratios outside of the range 0.8-1.2, (3) yielded natural signals that did not intersect the regeneration growth curves, (4) had a signal larger than 10% of the natural signal after a zero dose, and (5) produced a D_e within 1-sigma of zero.

A total of 2951 single-grains of 180-212µm were measured. Of these, 1030 passed all the criteria, an acceptance rate of 35%. Of those rejected, 66% were because of poor signal. Of the other criteria mentioned, the largest number of rejections was due to the natural signal not intersecting the growth curve (17%). Most of these were probably due to the regeneration curves not being carried out far enough to capture the natural signal, although some showed the regeneration curve saturating below the natural level, a phenomenon well documented in quartz, but not published to our knowledge for feldspar. Figure 1 shows some examples. Early measurements used about 300 Gy as the highest regeneration dose, but later measurements (on the UW1326-1329 180-212µm grains) reduced this to about 185 (for UW1327-8) or even 25 Gy (for UW1326, UW1329) to save machine time when it was realized the samples were much younger. This had the effect of increasing the number of rejections due to lack of natural intersection. The full distributions, even for those with a maximum regeneration dose of 300 Gy, are clipped at the high end. This should not affect minimum age determinations as long as the highest regeneration dose is well above that required for the minimum ages. To measure the effect, the distributions for single-grain samples where the highest regeneration dose was 300 Gy were artificially clipped for maximum D_e of 185 and 25 Gy (Table 2). No significant difference in minimum age is observed. A 25 Gy dose translates into an uncorrected age of about 10 ka. Almost half the rejections by this criterion came from one sample, UW1326, the sample from the highest terrace (and presumably the oldest).

A total of 1787 single grain holes containing 125-150 µm grains (small aliquots) were measured for 4 samples, and 946 were accepted, a rate of 53%. Of the rejections 30% were because of the natural signal not intersecting the growth curve, again the largest number (about half) from UW1326. After fading tests, there were further rejections for both single grains and small aliquots because high fading rates produced infinite corrected ages. The number of measurable ages totalled 917 for the single grains and 861 for the multi-grain aliquots.

A dose recovery test was done on 400 180-212µm grains from four samples, using an administered dose of 200s (~24 Gy) of beta irradiation. A total of 201 grains produced an acceptable signal. The weighted average, using the central age model, was 205 ± 3.4 s, but with an over-dispersion of 18.5%. These results suggest the procedures are generally appropriate, but the grains show wide variation in behavior. The 18.5% over-dispersion indicates that scatter of at least that magnitude can be expected in the natural samples even if all grains are the same age.

Fading rates were also highly variable. A weighted average of all g-values from the 180-212 µm grains yielded 3.9 ± 0.3% (normalized to two days), which is in the 3-5% range reported for southern British Columbia and northern Washington by Huntley and

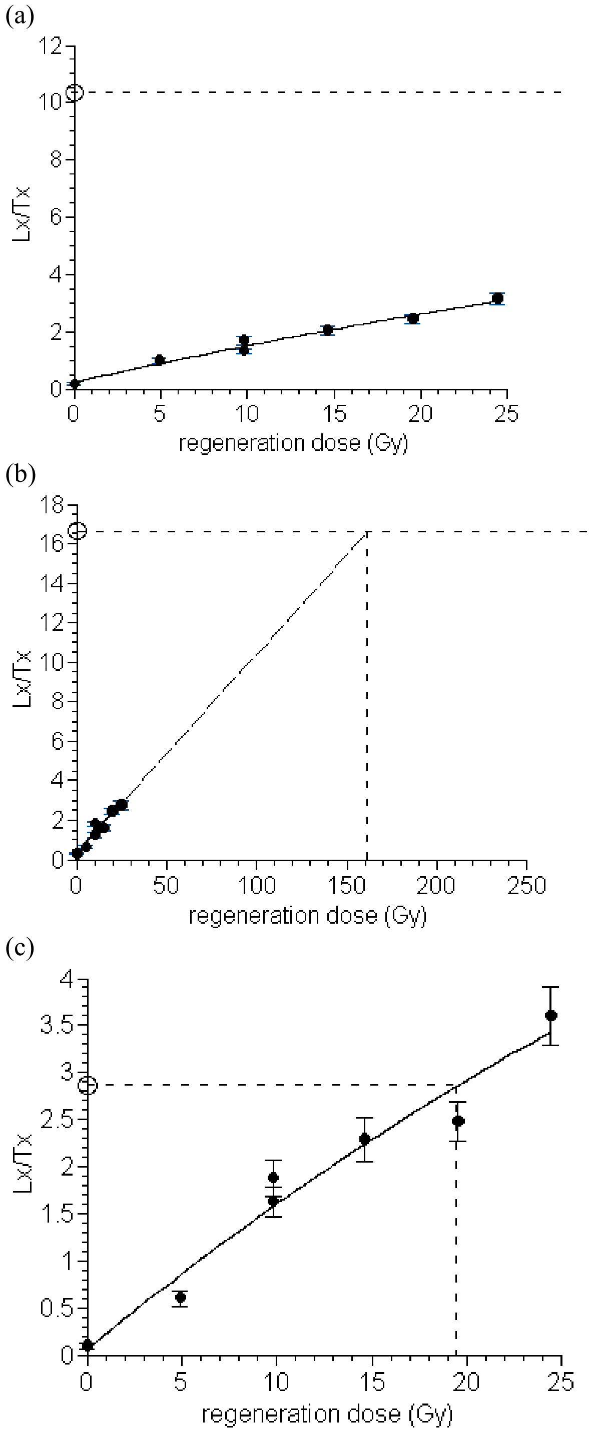


Figure 1: *Growth curves for three grains from UW1326, 180-212µm grains. a) grain where the regeneration curve saturates below the natural level, b) grain where an extrapolated saturating exponential curve is able to reach the natural level, c) grain where the natural level is within the limits of the regeneration growth curve.*

Sample UW	Age (ka) from MAM for maximum regeneration doses					
		300		185		25
	N	Gy	N	Gy	N	Gy
1863	122	2.9±0.8	109	2.9±0.8	72	3.2±0.8
1864	131	3.0±0.5	124	3.0±0.5	58	2.9±0.6
1865	214	4.4±0.6	198	4.3±0.6	74	3.3±0.6
1866	226	5.0±0.9	193	4.6±0.9	84	5.0±1.4

Table 2 : *Minimum age values for different maximum regeneration doses. In each case, N, is the number of grains used in the MAM.*

Lamothe (2001). Leaving off negative values (17% of them), over-dispersion of the g-values was calculated at 43%.

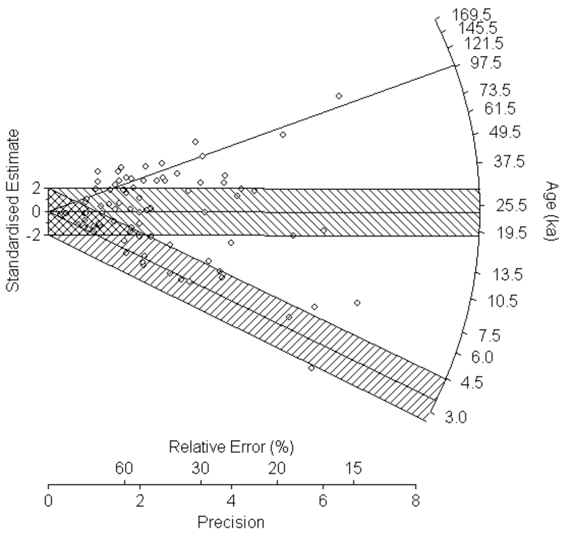
Table 1 gives the ages determined by the central age model as well as the over-dispersion. Except for the single-grains of UW1326, the over-dispersion is high, over 100% for all other single-grain samples. The central age is higher and over-dispersion lower for the small aliquots, on a sample to sample comparison. The lack of over-dispersion for single-grains of UW1326 can be explained by the use of a maximum regeneration dose of 25 Gy. The older grains for this sample were simply not accepted because the natural signals were too large. A total of 43% of grains from this sample were rejected for this reason. The accepted grains form an effective single-age sample. It is possible the age for this sample is underestimated because the regeneration doses were not carried out far enough, but the determined age is consistent with independent evidence. We also reanalyzed this sample accepting all the grains where the natural signal was larger than the highest regeneration dose, using extrapolation, either linear or saturating exponential, of unknown reliability. This resulted in 183 acceptable values (after the fading correction). No longer consistent with a single age, the minimum age for this sample was 15.0 ± 1.51, somewhat older but within error terms of the single grain age determination from the clipped data set.

The most likely cause of high over-dispersion for the other samples is partial bleaching. The central age model produces ages that are Pleistocene or very early Holocene, which is not in accordance with the geological evidence that the terrace sedimentation is post-glacial. Radiocarbon evidence strongly points to deposition on the uppermost terrace ending some time after 11,400 ^{14}C yrs. B.P. (Saunders et al., 1987). Post-depositional mixing is not likely, because it is not clear where the older grains would be coming from. The older ages more likely represent grains whose signal was not fully reset at the time of deposition. Partial bleaching is not uncommon for high energy streams. Another possible cause of over-dispersion is differential dose rate caused by variation in K content within individual grains. For the 33 grains for which both K content and age were determined, there is no significant correlation between K and age ($R^2 = 0.01$). Reducing the internal dose rate by a third (which would be appropriate for lower K grains) only reduces the total dose rate by 10%, which cannot account for the differences by a factor of 5 to 10 between the central age and minimum age for these samples.

If partial bleaching is the cause of over-dispersion, then the minimum age model is appropriate. We used the model of Galbraith et al. (1999), with three unknown variables, and adding 18.5% error in quadrature to the error on each age (the 18.5% value coming from dose recovery). Minimum ages are given in Table 1, with 125-150 µm results in bold. The minimum ages for the 180-212 µm grains are in accordance with the post-glacial expectations from the geology, and agree with some independent dating evidence, including radiocarbon analyses from the outwash surface of Terrace 1. They also roughly agree with the chronological order of the terraces, although errors are high. The ages of the 180-212 µm fraction from UW1327-1328 also agree with counterparts UW1864-1865 from the same terraces, even though the latter were measured with higher regeneration doses. The minimum ages of the 125-150 µm small aliquots, on the other hand, are much older for every sample, two of them Pleistocene in age.

Figure 2 shows radial graphs of the age distributions for both grain sizes for UW1327. The two shaded areas represent the 2σ range for the minimum age and central age for the 180-212 µm fraction. The third line represents a possible higher component. Both graphs show the older components, but the younger ages are mostly absent with single-aliquots using smaller grain sizes. This can be readily explained by averaging effects, particularly considering the high acceptance rate for these samples and the high number of partially bleached grains. The smaller signals associated with the younger grains are simply swamped by the larger signals from the more plentiful older, partially bleached grains. Perhaps if enough measurements were made, at considerable cost in machine time, a large enough population of younger ages could be measured so that they would show up statistically as a minimum age. The larger the aliquot the less likely this would be possible (Fig 4. in Olley et al. 1999).

(a)



(b)

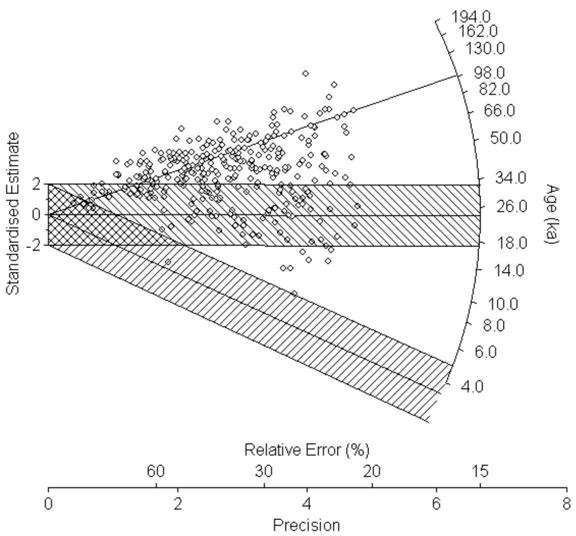


Figure 2. *Radial graphs showing central age and minimum age for UW1327 using (a) 180-212µm single grains and (b) 125-150µm multi-grain aliquots. The reference lines in both graphs are the minimum age and central age results from the 180-212µm fraction plus a possible older component around 100ka. The shaded areas around the minimum age and central age reference lines represent 2σ range.*

The data presented here suggest that for partially bleached sediments, particularly involving highly sensitive feldspars, accurate minimum age determinations will require single-grain analysis.

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Reviewer
M. Lamothe

Reviewers Comment:
Feldspar single grains are back! This is a nice demonstration that unless the most simple sedimentary object is used, that is a single grain, luminescence dating will not provide an accurate evaluation of burial time for partially-bleached sediments. More research in feldspar single grain laser technology is timely, to increase precision, a malign collateral “damage” of anomalous fading.