

SUPPLEMENTAL TEXT

Dating Stone Alignments by Luminescence

James K. Feathers, Maria Nieves Zedeno, Lawrence C. Todd, Stephen Aaberg

Method

Sample collection technique -- Samples were collected by driving a soil sampler vertically into the sediment beneath the approximate center of the rock. The sample was collected in a 5 x 20 cm plastic sleeve insert. A portable light-tight enclosure was placed over the rock to block sunlight from reaching the sediment when the rock was overturned. Aided by a flashlight constrained by the same orange/red filters as used in the laboratory, the rock was removed and the sampler driven in. The sampler was removed and the plastic sleeve extracted, again within the enclosure, capped and wrapped in Al foil and duct tape to prevent light exposure enroute to the laboratory. A dosimeter of CaSO_4Dy , encapsulated in 1 mm of copper, was left on top of the sample hole and the rock replaced. The dosimeter was retrieved one year later. None were left at Whitewater because of planned disturbance from road work. One sample from Kutoyis, UW2440, possibly got exposed to light during collection. The wind blew off the light-blocking enclosure for a brief moment when the rock was overturned and it is possible that some grains got exposed.

The plastic sleeve was unwrapped under laboratory light and sliced into five 4-cm sections, labeled A to E with A being the segment originally directly under the rock. Each section was prepared and dated separately, providing vertical age control. If the bleaching hypothesis is correct, section A should contain the highest number of bleached grains, which should then decrease with depth. While one sample was collected by just sampling the top few centimeters

below the rock with a trowel, the advantage of using the soil sampler is the vertical age data that can be used to support the bleaching model. Preparation procedures are given in Feathers (2012). An important point to mention here is that the K-feldspars were separated using a 2.58 density separation solution. This means the samples consist of all 180-212 μ m grains that are lighter than 2.58 specific gravity – not all of which are K-feldspars.

Fading correction procedures -- Anomalous fading has been attributed to quantum tunneling from electron traps to nearby recombination centers because of overlapping wave functions (Huntley and Lian 2006; Visocekas 2002). One way to deal with fading is to apply a correction. A common method measures the fading rate over a reasonable laboratory time and extrapolating that to longer, archaeological times. Because tunneling would occur earliest for trap/centers that are nearest to each other, the number of trapped electrons would decrease linearly with the logarithm of time (Huntley and Lian 2006). This relationship can be used to determine the correct age through an iterative procedure involving the measured fading rate (Huntley and Lamothe 2001). This procedure has been found valid for younger samples where the dose response curve is in the linear region, which should be the case for these samples. The problem at single-grain scale is achieving good precision, because measurements of fading rate on single grains often carry large error terms. Another way to deal with fading is to avoid it. Over the years, many attempts have been made to isolate a non-fading component in feldspar, with questionable results. However, in the last few years, some success has been reported using a post-IR IR signal measured at elevated temperatures (Buylaert et al. 2012; Buylaert et al. 2009; Jain and Ankjaergaard 2011). The initial IR stimulation (at 50°C) and the higher temperature of the second IR stimulation serve to remove those trap/center pairs most likely to fade, so that the

second IR stimulation mainly samples those pairs that are far enough apart that they do not likely fade. The procedure has mainly been employed for older samples and on multi-grain aliquots. Some evidence suggests the post IR signal bleaches at a slower rate than the conventional IR signal, leaving a residual dose. This appears to be a minor problem for older samples, but is certainly an issue for younger ones. Here we measured fading rates, on individual grains, following Auclair et al. (2003) and applied the Huntley-Lamothe correction to the derived age. For some grains, we applied an elevated stimulation temperature post-IR IR procedure for comparison, particularly to show how the procedure behaves on single-grains and with young samples (Reimann et al. 2012). Fading rates are expressed as g-values, which is the percent signal loss per decade, where a decade is taken as a power of 10, and normalized to two days. For example, a g-value of 4 indicates a loss of 4 percent in two days, another 4 percent in 20 days, another 4 percent in 200 days and so forth.

Additional dose rate methods – Alpha counting, using the pairs technique, was used to calculate U and Th concentrations, while flame photometry was used to measure total K content (which was converted to ^{40}K by natural atomic abundance). While the beta dose rate can be determined indirectly by calculation from flame photometry and alpha counting, assuming secular equilibrium, it can also be determined directly by beta counting. Both methods were used. Where the beta counting produced a different value at one-sigma from the flame photometry and alpha counting, the beta counting result was considered more accurate and was substituted into the age equation for the beta dose rate. This means the concentrations reported in Supplemental Data Table S1 may not be reflected entirely in the dose rates given for some samples.

Moisture contents, which affect the dose rate, were estimated at 6 ± 3 percent for all samples, based on average measured contents on samples collected during the summer. This reflects the semi-arid environment. Water content may be more in the winter due to snow, but probably only significant for the high elevation Absarokas. Cosmic ray contribution to the dose rate was calculated taking into account latitude, longitude, altitude and burial depth, following Prescott and Hutton (1994).

Results

Dose rate results – Supplemental Data Table S1 gives the relevant concentrations for segments A and E and for the overlying rock. It also gives the total dose rate for each sample. Some of the variation in U and Th contents can be attributed to use of the pairs technique in alpha counting to separate them. Depending on the alpha dose rate, this can result in large errors in the individual U and Th values, although the effect on the total dose rate may be negligible (Aitken 1985).

On the beta dose rate, of the 82 comparisons between results from beta counting and results from alpha counting/flame photometry, only 10 showed significant difference at one-sigma, none at two sigma. To the extent this provides a measure of disequilibrium in the U decay chain, little evidence is apparent.

Supplemental Data Table S2 gives the average cation concentrations of individual feldspar grains for different samples and for the different sites. One can see that K concentrations are far less than the 14 percent expected on stoichiometric grounds for end-state orthoclase, although for most samples, K is the prominent cation. K has higher concentrations at Whitewater, 48PA1151, and some Kutoyis samples, but it is low for many Kutoyis samples and

for Corral Creek samples. The site average with standard deviation was used for each site. Lower concentration has the advantage of less reliance on dose rates internal to the individual grains. It amounted to only 4-6% of the total dose rate for Kutoyis compared to 12 percent for Whitewater. It is assumed that K-feldspar is the major source of beta heterogeneity at a single grain level for these samples, as carbonates are not present, and that the error from internal K concentrations will account for it.

Anomalous fading. Table S4 shows the median g-value for all grains measured at different stimulation temperatures. Notice the difference in power between the two 50°C categories. Early measurements were stimulated at 70-90 percent power of the IR laser before it was realized that power of that magnitude generated enough heat to raise the actual temperature of stimulation to some unknown amount beyond 50°C, although probably not as high as 200°C. The table shows that median g-values decrease with increasing stimulation temperature, with very little fading noted in the 290°C simulation. Another measure of this is the percentage of grains with fading rates significantly greater than zero (Table S4). The percentage decreases with stimulation temperature, but it is noteworthy that even at 290°C, 38 percent of the grains still showed significant fading. The advantage of a high temperature stimulation is not only better precision, but also a significant savings in machine time required to perform a fading test. But if some grains still fade, one cannot assume no fading and therefore dispense with the test. Supplemental Data Table S4 also shows that the measurements stimulated at 70-90 percent power act like elevated temperature stimulations in that the fading rate is less than for those measurements stimulated at 30 percent power, although still greater than the fading rate at 225°C. The effective temperature at the higher power is likely between 100 and 200°C.

Dose Recovery. Dose recovery tests among different stimulations are given in Table 1. Dose recovery is a method of evaluating the procedures used for determining equivalent dose. The luminescence signal is reduced to zero by exposure to light and then a known dose is applied. The SAR protocol is carried out using the known dose as a proxy for the natural dose. The derived equivalent dose is then compared with the known dose. The ratio between them should be close to one for appropriate procedures. Table 1 shows that increasing the stimulation temperature increasingly over-estimates the given dose. Only the 50°C/30 percent power stimulations shows a ratio close to what it should be. The reason for the over-estimation is not clear, but it could relate to the mode of zeroing, although different modes were used (exposure to IR laser at room temperature, exposure to IR laser at high temperature, exposure to a solar simulator) and no systematic differences were seen. Another possible reason is that the intensity of zeroing was not enough to remove a high-temperature residual, although this would affect the calibrating doses as well as the administered dose.

There is also concern that traps stimulated at high temperatures are less bleachable by sunlight than all traps as a whole. This means that the natural signal may contain a residual, which has been observed in many studies on high temperature stimulation (e.g., Nian et al. 2012; Roberts 2012; Stevens et al. 2011; Thiel et al. 2011). Most work using high temperature stimulations on feldspars has concerned older samples where residuals are less of a problem, but residuals could have a significant effect on Holocene-aged samples (Reimann et al. 2012). Residual levels were not measured directly in this study, but Figure S1 compares the minimum age values for low and high temperature stimulations for several samples from Kutoyis and Whitewater. The high temperature stimulations overestimate the age for all but one of the Kutoyis samples, but the relationship at Whitewater is much less clear, with only two samples

overestimating age but the other four in the same range or even less than the low temperature stimulations. This suggests the presence or size of a residual is sample specific.

Modern samples. The modern samples yield ages of from 40 to 80 years. The size of the residual is a function of the intensity of turbation. For example, if it takes 100 years to cycle a significant number of grains to the surface, then residuals of 50 years or so might be expected. The more intense turbation evident at Whitewater, as compared to Kutoyis, may explain in part the smaller residual at Whitewater.

Another issue not explored is thermal transfer, which is the transfer of trapped charge from a light insensitive trap to a sensitive one during the preheat. The preheat proceeds each measurement to remove unstable charge. Although some thermal transfer can be expected and be responsible for some of the age offset, the correlation of the size of residual with intensity of turbation suggests the latter is more important.

Ages. In compiling the ages, if low temperature and high temperature stimulations yielded age distributions that were in agreement, then both were considered together. In all cases where there was a high temperature stimulation, there was also a low temperature stimulation for the same grain. The value with the lowest error term, most commonly the high temperature stimulation because of reduced fading, was used for that grain. Where high and low temperature stimulations were not in agreement, only the low stimulation values were used.

Supplemental References

Auclair, Marie, Michel Lamothe, and Sebastien Huot

2003 Measurement of Anomalous Fading of Feldspar IRSL using SAR. *Radiation Measurements* 37:487-492.

Buylaert, Jan-Pieter, Mayank Jain, Andrew S. Murray, Kristina J. Thomsen, Christine Thiel, and Reza Sohbat

2012 A robust feldspar luminescence dating method for Middle and Late Pleistocene sediments. *Boreas* 41:435-451.

Buylaert, Jan-Pieter, Andrew S. Murray, Kristina J. Thomsen, and Mayank Jain

2009 Testing the potential of an elevated temperature IRSL signal from K-feldspar. *Radiation Measurements* 44:560-565.

Feathers, James K.

2012 Luminescence dating of anthropogenic rock structures in the northern Rockies and adjacent High Plains, North America: a progress report. *Quaternary Geochronology* 10:399-405.

Huntley, David. J., and Michel Lamothe,

2001 Ubiquity of anomalous fading in K-feldspars and measurement and correction for it in optical dating. *Canadian Journal of Earth Science* 38:1093-1106.

Huntley, David J., and Olav B. Lian

2006 Some observations on tunneling of trapped electrons in feldspars and their implications for optical dating. *Quaternary Science Reviews* 25:2503-2512.

Jain, Mayank, and Christina Ankjaergaard

2011 Towards a non-fading signal in feldspar: insight into charge transport and tunneling from time-resolved optically stimulated luminescence. *Radiation Measurements* 46:292-309.

Nian, Xiao-Mei, Richard. M. Bailey, and Li-Ping Zhou

2012 Investigations of the post-IR IRSL protocol applied to single K-feldspar grains from fluvial sediments samples. *Radiation Measurements* 47:703-709.

Prescott, John R., and John T. Hutton

1994 Cosmic ray contributions to dose rates for luminescence and ESR dating: large depths and long time durations. *Radiation Measurements* 23:497-500.

Reimann, Tony, Kristina J.Thomsen, Mayank Jain, Andrew S. Murray, and Manfred Frechen

2012 Single-grain dating of young sediments using the pIRIR signal from feldspar. *Quaternary Geochronology* 11:28-41.

Roberts, Helen M.

2012 Testing post-IR IRSL protocols for minimizing fading in feldspars, using Alaska loess with independent chronological control. *Radiation Measurements* 47:716-724.

Stevens, T, S. B. Markovi, M. Zech, U. Hambach,, and P. Sümegi

2011 Dust deposition and climate in the Carpathian Basin over an independently dated last glacial interglacial cycle. *Quaternary Science Reviews*. 30:662-681.

Thiel, Christine, Jan-Pieter Buylaert, Andrew S.Murray, B. Terhorst, I. Hofer, Sumiko

Tsukamoto, and Manfred Frechen

2011 Luminescence dating of the Strazing loess profile (Austria) – testing the potential of an elevated temperature post-IR IRSL protocol. *Quaternary International* 234:23-31.

Visocekas, Rafael

2002 Tunneling in afterglow, its coexistence and interweaving with thermally stimulated luminescence, *Radiation Protection Dosimetry* 100:45-54.