Luminescence dating of samples from recent contexts in southern Africa

Dana Drake Rosenstein[a] & James Feathers[b]

[a] School of Anthropology, University of Arizona, Tucson; ddr@email.arizona.edu
[b] Department of Anthropology, University of Washington, Seattle; jimf@u.washington.edu

INTRODUCTION The last 500 years was a formative period of the southern African past, during which hunter-gatherers, agropastoralists and colonists interacted frequently and intensely on the shared landscape. The archaeological and historical data from this era interleave; fitting the material remains into the oral record requires the chronological sequence of archaeological site settlement, use and abandonment to be sound and resolved ideally to a generational scale.

The prominent hill Smelterskop, in Rooiberg, Limpopo Province, South Africa (Figure 1), was at the center of the only major precolonial tin mining and smelting industry in southern Africa. When colonial geologists first surveyed the area at the beginning of the 20th century, it was estimated that earlier African miners extracted 20,000 tons of tin ore with hand tools.[1] The smelted tin was alloyed with copper to produce bronze, a metal traded widely in the region.[2, 3] Systematic archaeological research at Smelterskop began in 2006, and a major goal of this project was to establish a chronology of tin production in southern Africa.[4]

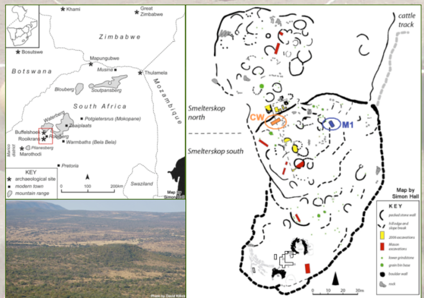


FIGURE 1 Left, photograph of Smelterskop and map of the Rooiberg region. Right, plan of Smelterskop, a major tin production center. The two archaeological features sampled for OSL were the Central Wall midden (CW - orange) and Midden 1 (M1 - blue).

RADIOCARBON DATING For decades, radiocarbon dating has been the preferred chronometric technique for sites in southern Africa; however, this method is inadequate for sites occupied in the last 500 years. Because of anomalies in atmospheric production of radiocarbon in the time range 1650-1950 cal CE, there are acute De Vries effects, or 'wiggles,' in the radiocarbon calibration curve over this period. Any radiocarbon age from this time calibrates to an ambiguous calendar date spanning two or more centuries and thus is not useful for evaluating the archaeology of tin at Smelterskop (Figure 2).

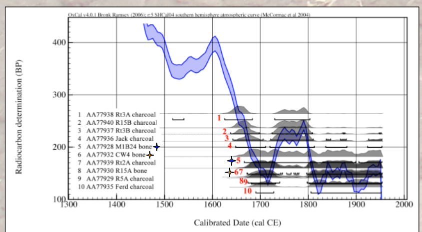


FIGURE 2 OxCal plot[9, 10] of AMS radiocarbon dates (2σ error) for bone from M1 and CW midden and charcoal from tin production areas on Smelterskop. Any radiocarbon determination from 200-100 BP unhelpfully calibrates to a range of calendar dates from 1650-1950 cal CE. Radiocarbon results from 300-200 BP calibrate to the range 1650-1800 cal CE.

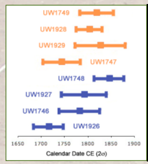


FIGURE 3 Table and plot of OSL ages (1σ error) and calendar dates (2σ error) from Smelterskop. The high overdispersion in Midden 1 is indicative of partial bleaching, and likely is related to incorporation of older grains from the layer below the midden during the sampling of this shallow, upsloping deposit.

Sample	Total dose rate (Gy/ka)	Unlogged age (Gy)	Unlogged age model	% σ_b	Age (ka)	% error (1σ)	Calendar date CE (2σ)
SKOP CW Midden							
UW1747	1.97±0.08	0.52±0.03	MAM	31	0.264±0.020	7.6	1744 ± 40
UW1749	2.01±0.08	0.38±0.03	CAM	0	0.189±0.018	9.4	1819 ± 36
UW1928	2.86±0.09	0.59±0.03	CAM	0	0.206±0.014	6.7	1803 ± 28
UW1929	2.80±0.12	0.51±0.07	CAM	0	0.182±0.027	14.7	1827 ± 54
SKOP Midden 1							
UW1746	2.22±0.11	0.50±0.04	MAM	31	0.225±0.022	9.8	1782 ± 44
UW1748	2.17±0.10	0.35±0.03	CAM	0	0.161±0.016	10.2	1846 ± 32
UW1926	1.71±0.08	0.50±0.04	MAM	19	0.293±0.016	5.5	1716 ± 32
UW1927	2.81±0.12	0.61±0.06	MAM	21	0.217±0.024	11.1	1792 ± 48

LUMINESCENCE DATING Using optically stimulated luminescence (OSL) dating on single quartz grains, chronometric results with good resolution were obtained for multiple samples from two archaeological middens on Smelterskop (Figures 3 and 4). A Venetian white-heart bead was excavated from the CW midden. This type of small glass trade bead appeared in southern Africa in the first decades of the 19th century, and the OSL ages corroborate this *terminus post quem* bead date. The older ages for Midden 1 substantiate archaeological evidence from stone wall architecture and ceramic decoration that suggest the feature relates to an earlier occupation of Smelterskop.

Some challenges in OSL dating of young samples are due to short burial time, yielding a low OSL signal. With low signal, even a small degree of partial bleaching in a sample can be significant. A high percent of overdispersion (σ_b) of individual grain measurements around the equivalent dose (D_e) of the sample is an indicator of a mixed-age sample.[5] This may be due to post-depositional mixing, partial bleaching, or inclusion of grains strata of different ages in the sample during collection. Where there is high overdispersion and evidence for partial bleaching or grains from bordering strata, the sample D_e is calculated with a minimum age model (MAM). Samples with low overdispersion represent a single statistical population, and the sample age is calculated with a central age model (CAM).

We measured hundreds of grains to obtain statistically significant samples of sensitive grains, performed stepped preheat tests and determined the frequency of recuperation to assess if thermal transfer occurred (Figure 5). Recuperation[6] is a term for the transfer of electrons from a light insensitive to a light sensitive trap during the preheat, and it can be a problem for young samples. Recuperation can cause age over-estimation. A significant OSL signal following a bleach and preheat is some indication of its presence (Figure 6). We calculated D_e on sample grains with and without recuperation to determine if it has an effect on age (Figure 7).

In addition, in young samples, a relatively high proportion of measured grains yield a D_e value close to or even less than zero. This is significant, because the common statistical models for calculating sample D_e from the population of accepted single grains are logged models. A logged model does not characterize a D_e population close to zero well and cannot integrate results where $D_e \leq$ zero. Performing a logged analysis, plus systematically deleting grains where $D_e \leq$ zero, will bias the sample D_e and result in an older age. We compared the Smelterskop OSL ages using both logged and unlogged[7, 8] statistical models (Figures 7 and 8).

FIGURE 5 Table of measurement and acceptance data on grains used to determine D_e. OSL measurement on single quartz grains (180-212 μm) was conducted according to the SAR protocol[11] with a 3 Gy test dose, either a stepped preheat test or 200°C preheat for 10s, and either a stepped cut heat or 160°C cut heat. Either 1.0 or 0.5 Gy was administered for the dose recovery tests. We used a Risö DA-TL-15 reader with a 540nm laser. OSL emission was detected through UV 340 filters at 125°C.

Sample	# measured*	Failed recycle test only	Unbleached**	Feldspar	Recuperation†	Accepted‡	% accepted
UW1746	1090	36	15	2	32	149	14
UW1748	1285	34	15	2	46	92	7
UW1926	~1100	~20	~15	0	44	77	7
UW1927	999	46	6	0	32	144	14
UW1747	996	32	29	1	28	145	15
UW1749	985	6	6	1	28	93	9
UW1928	~1500	~40	~25	3	31	104	7
UW1929	500	8	0	0	10	21	4

All grains measured according to the SAR protocol:[10] 3 Gy test dose, seven regeneration doses, one zero dose, one repeat, two doses followed by IR exposure before OSL.
* approximate values for UW1926 and UW1928
** signal from N larger than signal from highest regeneration dose (Figure 6)
† recuperation = Lx/Tx from zero dose > 10% of natural signal and > 0.1 (Figure 6)
‡ includes grains with recuperation

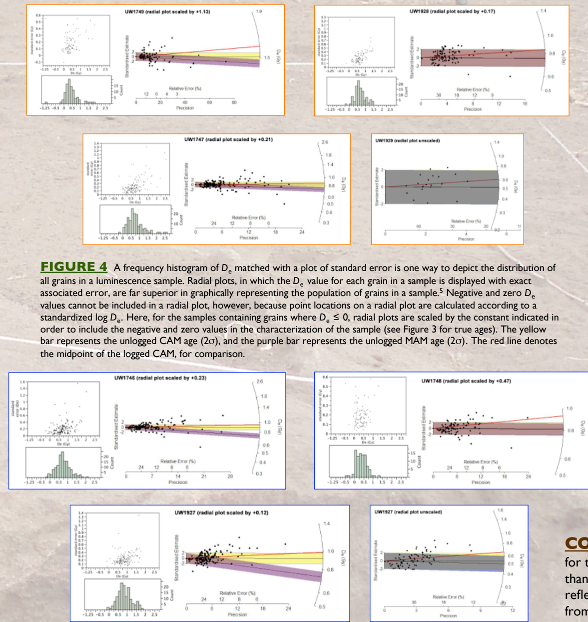
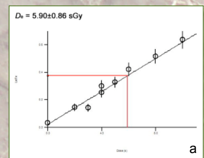


FIGURE 4 A frequency histogram of D_e matched with a plot of standard error is one way to depict the distribution of all grains in a luminescence sample. Radial plots, in which the D_e value for each grain in a sample is displayed with exact associated error, are far superior in graphically representing the population of grains in a sample.[5] Negative and zero D_e values cannot be included in a radial plot, however, because point locations on a radial plot are calculated according to a standardized log D_e. Here, for the samples containing grains where $D_e \leq 0$, radial plots are scaled by the constant indicated in order to include the negative and zero values in the characterization of the sample (see Figure 3 for true ages). The yellow bar represents the unlogged CAM age (2σ), and the purple bar represents the unlogged MAM age (2σ). The red line denotes the midpoint of the logged CAM, for comparison.

FIGURE 6 Dose response curves of grains from sample UW1748: (a) typical dose response curve; (b) dose response curve where the OSL signal from the natural is higher than the signal from the highest regeneration dose, indicating an unbleached or partially bleached grain or misestimation of the age range of the sample; (c) example of recuperation in a dose response curve, where Lx/Tx from the zero dose > 10% of the natural signal > 0.1 (Lx/Tx 0 sGy = 0.137; N = 0.450).



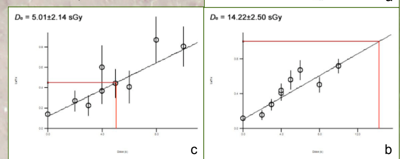


FIGURE 7 Comparison of Smelterskop OSL results analyzed with the unlogged CAM and MAM according to occurrence of recuperation. There is no significant difference between age values for grain populations of high versus low recuperation within 1σ (except UW1926-CAM, which is better characterized by the MAM due to high overdispersion).

Sample	Recuperation	n	Central age (Gy)	σ_b	Minimum age (Gy)
	low	117	0.69±0.03	35	0.52±0.05
	high	32	0.60±0.07	30	0.46±0.10
UW1746	*total grains*	*149*	*0.67±0.03*	*31*	*0.50±0.04*
	low	46	0.39±0.04	0	0.38±0.04
	high	46	0.31±0.04	0	0.30±0.04
UW1748	*total grains*	*92*	*0.35±0.03*	*0*	*0.34±0.03*
	low	33	0.62±0.06	0	0.56±0.09
	high	44	0.47±0.05	26	0.45±0.05
UW1926	*total grains*	*77*	*0.54±0.04*	*19*	*0.50±0.04*
	low	112	0.79±0.03	24	0.62±0.07
	high	32	0.85±0.07	22	0.65±0.20
UW1927	*total grains*	*144*	*0.80±0.03*	*21*	*0.61±0.06*
	low	117	0.63±0.04	36	0.53±0.03
	high	28	0.56±0.07	29	0.38±0.15
UW1747	*total grains*	*145*	*0.62±0.03*	*31*	*0.52±0.03*
	low	65	0.37±0.03	28	0.33±0.04
	high	58	0.40±0.07	0	0.36±0.07
UW1749	*total grains*	*93*	*0.38±0.03*	*0*	*0.33±0.03*
	low	73	0.61±0.04	0	0.59±0.04
	high	31	0.56±0.06	0	0.54±0.06
UW1928	*total grains*	*104*	*0.59±0.03*	*0*	*0.58±0.03*
	low	11	0.51±0.10	0	0.51±0.10
	high	10	0.52±0.10	0	0.51±0.12
UW1929	*total grains*	*21*	*0.51±0.07*	*0*	*0.51±0.08*

CONCLUSION The Smelterskop OSL dates demonstrate that for the recent past, luminescence dating is significantly more accurate than is possible with radiocarbon. Unlogged statistical models better reflect the range of D_e values typical in a young OSL sample. The results from Smelterskop indicate that use of the hill as a center for tin production postdates the earliest exploitation of tin from the Rooiberg valley by 500 years or more.[2] Refined chronologies using luminescence dating will enable us to map out ancient trade networks, identify centers of political economic power, explore the nature of relationships between communities and identify technological innovations in metallurgical and agropastoral production over the last 500 years - a major contribution to the historical record of South Africa.

FIGURE 8 Comparison of Smelterskop OSL results analyzed with the logged and unlogged CAM. The logged data are systematically higher, which would produce older ages. For young samples, which contain many grains with near zero and negative De values, logged statistical models do not characterize the population well.

Sample	Logged central age (Gy)	Unlogged central age (Gy)
UW1746	0.72±0.03	0.67±0.03
UW1748	0.46±0.02	0.35±0.03
UW1926	0.66±0.04	0.54±0.04
UW1927	0.87±0.03	0.80±0.03
UW1747	0.66±0.03	0.62±0.03
UW1749	0.46±0.03	0.38±0.03
UW1928	0.67±0.03	0.59±0.03
UW1929	0.61±0.06	0.52±0.07

ACKNOWLEDGEMENTS
David Killick, Simon Hall, and Shadreck Chirikure
National Science Foundation
National Research Foundation (South Africa)
UA-NSF IGERT Program in Archaeological Sciences
University of Cape Town Smelterskop field school students

REFERENCES

1. M. Baumann, *Journal of the Chemical, Metallurgical and Mining Society of South Africa* 19, 120 (1919).
2. L. J. Molofsky, J. T. Chesley, D. J. Killick, J. Ruiz, *Archaeometry* (in revision).
3. S. Chirikure, R. Heimann, D. Killick, *Journal of Archaeological Science* (in press).
4. S. Chirikure, S. Hall, D. Miller, in *Metals and mines: studies in archaeolometallurgy* S. La Niece, D. Hook, P. Craddock, Eds. (British Museum Press, London, 2007) pp. 112-119.
5. R. F. Galbraith, R. G. Roberts, G. M. Laslett, H. Yoshida, J. M. Olley, *Archaeometry* 41, 339 (1999).
6. M. J. Aitken, B. W. Smith, *Quaternary Science Reviews* 7, 387 (1988).
7. L. J. Arnold, R. G. Roberts, R. F. Galbraith, S. B. DeLong, *Quaternary Geochronology* 4, 306 (2009).
8. T. J. Pietsch, *Quaternary Geochronology* 4, 406 (2009).
9. F. G. McCormac *et al.*, *Radiocarbon* 46, 1087 (2004).
10. C. Bronk Ramsey. (Available at http://c14.arch.ox.ac.uk/, 2006).
11. A. G. Wintle, A. S. Murray, *Radiation Measurements* 41, 369 (2006).