Thermoluminescence Dating of Early Ceramics from Oman¹

In order to contribute to the chronology of early cultures in Oman, ceramic samples for thermoluminescence (TL) dating were collected during the 5th Oman Expedition of the Deutsches Bergbaumuseum Bochum in February and March 1981 (Weisgerber *et al.* 1981). Most of the samples derive from the archaeological excavations at al-Maysar M1 (locally, the Husn al-Hegli) which shed light on the history of copper production over the ages². In 1982 and 1983, the thermoluminescence laboratory of the Max-Planck-Institut für Kernphysik Heidelberg worked up the data presented below. The publication in which these were to appear was postponed. Since the topic of the radiocarbon chronology recently has come into discussion again (Yule 2005; Yule in press), it seems a good time to publish the existing thermoluminescence data from the Sultanate. Moreover, in the time interval since the pioneer research took place, much has been learned about Oman's early contexts, the different slag types and their dating criteria.

Samples and sampling procedure

Hd TL 7. Rawdah / Bir Kalher, slag heap

coordinates: 58°16'E; 22°54'N

Literature: Weisgerber 1981: 179 Fig. 4

The slag pile of 20x25 m extension and c. 0.5 m thickness consists of small slag fragments lying above clayey soil. It contains also few pieces of furnace ceramics and pebbles of gabbro. Several fragments of furnace ceramics were taken 0.1-0.2 m below the surface.

Hd TL 9. Wadi Salh

coordinates: 58°04'E; 22°48'N

Literature: Weisgerber 1980: 96 Fig. 61

One of the largest archaeometallurgical centres for copper in the western foreland of the mountains exists in the Wads Salh. Samples were collected at three sites here (Weisgerber 1980: 96 fig. 61).

Hd TL 9a. Wadi Salh, slag heap

coordinates: 58°04'E; 22°48'N

Literature: Weisgerber 1980: 96 Fig. 61

The slag heap of 65x15 m extension and 0.3-0.5 m thickness consists of small slag fragments – among them hand-sized pieces – and furnace ceramics. It rests on a magnesitic sinter terrace above water-bearing wadi debris. Various fragments of furnace ceramics were taken from 0.15-0.2 m depth.

Hd TL 9b. Wadi Salh, slag heap coordinates: 58°04'E; 22°48'N

¹ Günther Wagner contributed the thermoluminescence application, Paul Yule the archaeological interpretation. The authors thank Gerd Weisgerber for providing data and commenting on a version of the manuscript. G. Wagner contributed the photos.

² In the original report Weisgerber cites the name "Maysar" for the site, which corresponds to that which appears in the Gazetteer of Oman published by the Defense Mapping Agency in 1983. But in the local villagers refer to "al-Maysar". A few years ago the site name was officially changed, adding a *sheddah* after the *ya*. During our research in 1987 Weisgerber changed some of the site designations around the adjacent Samad oasis as a result of the growing number of sites in Samad/al-Maysar and a shift in the indigenous naming of sites. In 2001 in his *habilitation* Yule added the site abbreviations (*e.g.* instead of "al-Maysar 1", "M1") for Oman. This article is in press in the *Journal of Oman Studies*. Today: 20.08.2008.

Literature: Weisgerber 1980: 96 Fig. 61

The slag heap of 25x25 m extension and 0.3 m thickness consists of small slag fragments and furnace ceramics above water-bearing wadi debris. Furnace ceramics was taken from 0.3 m depth. At a distance of about 2 m from the sampling site lies a medieval pit with slag.

Hd TL 9c. Wadi Salh, slag heap

coordinates: 58°04'E; 22°48'N

Literature: Weisgerber 1980: 96 Fig. 61

The slag heap of 120x70 m extension and more than 1.5 m thickness consists of pit slag. small to medium sized flow slag and common fragments of furnace ceramics. Furnace ceramics was collected from 0.2 m depth within an accumulation of flow slag.

Hd TL 10. al-Maysar

In the region around the present-day village al-Maysar (altitude c. 550 m a.s.l.) samples were collected at six different sites.

Hd TL 10a. al-Maysar M42, section 2

coordinates: 58°07'E; 22°49'N

Literature: Weisgerber 1981; 223; Weisgerber/Yule 1999: 105-108

This Early Iron Age site with wall remains is covered with slope debris. In the corner between two rectangular walls (c. 0.4 m thick) the lower part of a pithos was found (Fig. 1). It was filled with dry, yellow-grey silt. A wall fragment of this pithos was collected in c. 0.5 m depth below the top surface (Yule 1999: 133-6).

Hd TL 10c. al-Maysar M1, house 1, loc. 10

coordinates: 58°07'E; 22°48'N

Literature: Weisgerber 1981: 191-192

In yellow-grey, dry silt c. 0.3-0.4 m depth below the top surface occur pot sherds and furnace ceramics incrusted with copper slag (Fig. 2). Samples were collected from a brown-grey. fine-grained sherd of 18 mm wall thickness (*Hd TL 10c1*) and several fragments of furnace ceramics (*Hd TL 10c2*).

Hd TL 10d. al-Maysar M1, house 6, TT27, loc. 36

coordinates: 58°07'E; 22°48'N

Literature: Weisgerber 1981: 193

The interspace of a 0.6 m thick wall with gabbro revetment is filled with yellow-grey loam, slag-incrusted furnace ceramics and slag. Samples were collected from red-burned (*Hd TL 10dl*) as well as black-burned furnace ceramics (*Hd TL 10d2*) at c. 0.3 m depth below the uppermost surface.

Hd TL 10f. al-Maysar M1, house 1, TT8

coordinates: 58°07'E; 22°48'N

Literature: Weisgerber et al. 1981: 192

Remains of a building with yellow loam floor covered by dry yellow silt (Fig. 3). Samples of smelting furnace ceramics were removed from the floor in 0.3-0.4 m depth below the uppermost surface.

Hd TL 10g. al-Maysar M1, house 1, TT21, planum 3. coordinates: 58°07'E; 22°48'N Literature: Weisgerber 1981: 192

Samples of furnace ceramics were taken from a dry yellow silt horizon 0.4-0.5 m depth below the uppermost surface (Fig. 4). This horizon lies about 0.1 m beneath sample *HD TL 10f.* Sample lies above a hard clay layer.

Hd TL 10h. al-Maysar M34, defensive structure, room 6 coordinates: 58°07'E; 22°48'N

Literature: Weisgerber 1981: 233-234

Pot sherds were taken from a dry yellow silt next to a gabbro wall. Sherd Hd TL h1 was brown-grey, coarse-grained and of 1 cm wall-thickness. Sherd Hd TL h11 was grey, fine-grained and of 0.5 cm wall-thickness with fine grooves.

Hd TL 12. Lizq L1 T2, fort

coordinates: 58°11'E; 22°42'N

Literature: Weisgerber et al. 1981: 226-231

Pot sherds were found in trench 2 at the southern wall of the early iron age fort (Fig. 5). The sherds are imbedded in yellow loam. Sherd *Hd TL 12a1* was brown, fine-grained with of 0.8 cm wall-thickness and comes from 0.5 m depth. Sherd *Hd TL 12a2* was brown-grey, coarse-grained with of 2 cm wall-thickness and comes from 0.2 m depth.

Hd TL 14. al-Batin

coordinates: 58°40'E; 22°46'N

Literature: Yule/Weisgerber 1996: 141

There are two heaps of copper slag (up to 10 m in diameter and 0.3 m in height) on both sides of the road south of the oasis al-Batin. Loamy dust fills the spaces between slag lumps. Several fragments of furnace ceramics were collected.

At all sampling locations the environmental external gamma dose-rate was recorded *in situ* with a portable NaI-scintillation counter. Immediately after removing the sample from its archaeological context the probe was inserted into the original find position whereby removed silty material was re-filled. Five minutes are sufficient for the measurement. For porosity determination in the laboratory small specimens of the surrounding sediments were collected. The gamma dose-rates are presented in table 4. They range between 0.22 and 0.42 mGy/a which is relatively low compared to most regions. But such values are compatible with the basic to ultrabasic composition of the regional geology in Oman.

Experimental Results

Ideally for luminescence dating of ceramics several samples should be collected for each archaeological context and each ceramic sample should be analysed by the fine-grain as well as the quartz-inclusion techniques. In the present project this was hardly ever possible due to small sample size (< 30 g), deficient quantity of samples per context, insufficient TL-properties or too little quartz temper. Of all collected samples finally only those which are suited are shown in table 1.

In natural condition all samples were dry and their porosity was determined (table 4). After crushing the fine-grain fraction (0.002-0.008 mm) and the quartz inclusion fraction (0.125-0.200 mm) were separated. Merely in six samples sufficient amounts of quartz (> 80 mg) were found. The fine-grain fractions were examined for the following TL-properties (cf Aitken 1985. Wagner 1998): plateau behaviour in the glow-curve range above 360°C, TL-signal growth versus applied dose, reproducibility and anomalous fading of the TL-signal. Due to insufficient properties, c. 25% of the fine-grain fractions had to be discarded. The

quartz-inclusion fraction was etched for 40 minutes in 40% hydrofluoric acid at room temperature. The quartz fractions were tested for the shape of the glow-curve, signal growth versus applied dose and the reproducibility of the TL-signal. Six fractions had to be discarded. The TL-sensitivity was determined with a calibrated ⁹⁰Sr- ⁹⁰Y beta source (1.5 GBq) and six calibrated ²⁴¹Am-alpha sources (each 1.85 MBq). The resulting values for the equivalent dose ED, the intercept correction I₀ and the archaeodose AD (= ED + I₀) appear in table 2.

We subjected uranium and thorium to neutron activation analysis for internal dose-rate determination, uranium also by fission tracks and potassium by atomic absorption spectroscopy. The analytical results appear in table 3, whereby the uranium content is the mean of both applied techniques. The alpha and beta dose-rates were calculated from these contents using the conversion factors by Aitken (1985) and are shown in table 4. Although the ceramic samples were found in dry condition, the dos-rate calculation takes into account an assumed natural moisture of 10 % (0.1 > porosity) as average for the whole past.

TL Dating Results

Table 5 presents the TL ages as well as the corresponding calendar dates. For two samples (Hd TL 9c1, HD TL 10d1), that allowed fine-grain as well as quartz inclusion dating, also the error-weighted mean age is given. The given 1 sigma probability is the total error combining statistical and systematic components (Aitken 1985). It includes also an uncertainty for the assumed moisture 0.1 (<0.1). Any uncertainty due to possible radioactive disequilibrium is also considered according to Mangini *et al.* (1983).

Archaeological Interpretation

The TL determinations of the archaeological contexts have the advantage of providing a corrective to other dating methods, the most important of which is radiocarbon dating. TL itself has been subject to considerable study since samples were taken in Oman in 1981. While TL is generally not as precise a determinant of age as is carbon dating, given recent doubts cast on the validity of the ¹⁴C determinations for the Samad period (Haerinck 2003), the former method takes on an important, even decisive role in the matter of chronology. Since the TL plus-minus values of the assays are unmistakably stated, there is no doubt as to the exactness of a given assay. Turning to the Table 5, which shows the TL age of the conventional chronology. Some of the archaeological context dates have been corrected since the fieldwork took place in 1981. In what follows, TL and selected carbon determinations are compared in ascending chronological order.

Different archaeological sites are known at al-Batin, but the one which was selected for TL testing is one is the earliest smelting site known in Oman, perhaps of the **Hafit** period. The determination of 2500 BCE \pm 1000 is low in terms of the conventional chronology for the Hafit period, but is all the more valuable owing to the rarity of dating means for this age. The slag deposit is contemporary with another rare example at Mullaq (Weisgerber *et al.* 1981: 209 fig. 41).

Three of the TL assays were taken from slag heaps in the Wadi Salh, which date to different periods. The TL assay 9a1 (2590 BCE \pm 320) can only derive from an archaeologically proven **Umm an Nar** deposit here. Moreover, G. Weisgerber, the excavator of al-Maysar, dates the settlement M1 to this same period, a dating which is established in the literature (*e.g.* Prange 2001: 14). By comparison, calibrated carbon datings (Weisgerber *et al.* 1981: 251 table 2) from house 1 at M1 yield the following results:

lab no.	find-spot	¹⁴ C age BP	cal. age W1 BCE
Hd-5736-5427	M1 H1 Loc 6	3840 <u>+</u> 50	2456-2205
Hd-5737-5294	M1 H1 Loc 1	3460+50	1881-1705
Hd-5738-5295	M1 H1 Loc 2	3780 <u>+</u> 60	2317-2137
HAM 1171	M1 H1 Loc 2	3560 <u>+</u> 70	1950-1920
Hd-8522-8740	M1 H1 Loc 43	3865 <u>+</u> 65	2470-2230

Selected radiocarbon determinations from Umm an Nar period, M1.

The TL assays (1560 BCE \pm 260, 1380 BCE \pm 290, 1600 BCE \pm 270, 1540 BCE \pm 300, 2080 BCE \pm 330, 1940 BCE \pm 310) are in line with the carbon results.

Further bronze age sites include Bil Kalher (TL: 1310 BCE ± 250), which independent of the assay Weisgerber dates archaeologically to the **Wadi Suq period** (nominally 1800-1300 BCE). An initial classification of this site in the Umm an Nar period rested on the strength of the mineralogical attributes of the slag. The slags of the two periods are very similar in appearance and the TL dating points to a more probable later dating for the deposit.

Early iron age sites with TL determinations include the Lizq/Rumaylah period mountain fort, Lizq L1, and the al-Maysar settlement site, M42. The term "Lizq/Rumaylah" emphasises the essential artefactual unity of this archaeological assemblage over its area of distribution. What is excavated of the first of these two eponymous sites shows a shallow/mixed stratification. P. Magee established a three-part chronology for the EIA (1996), which fits well with the TL results. In it he dated the Lizq fort in the early iron II phase by virtue of two ¹⁴C determinations (1996: 247 table 2, 1100-600 BCE)³.

The TL assay of 280 BCE \pm 170 from M42 belongs to a context at the very end of the early iron age, by virtue of the dating the mechanics of the associated *falaj* M46, and its relative dating with regard to the associated sites M42, M34, M43, the latter two of the subsequent Samad period (Weisgerber *et al.* 1981: 223 fig. 58; Weisgerber/Yule 1999: 98-100 Fig. 2). In light of the inferential nature of the absolute dating of the end of the early iron age (and the beginning of the late iron age), this TL datum is most welcome. A. Hauptmann mentions a site in the Wadi Salh as dating to the early Islamic period by virtue of the characteristics of the slag found there (Hauptmann 1985: 12 fig. 1). Our TL assays 9b1 and 9c1 of 620 \pm 260 and 670 BCE \pm 250 can only derive from early iron age deposits which were proven archaeologically after Hauptmann's work took place (Weisgerber personal communication).

In Magee's EIA chronology, one difficulty is that at the time he wrote the only site essentially available to him in the Sultanate in terms of published pottery was the Lizq fort, known from a limited number of drawings and photos (Weisgerber 1981: 224 Abb. 60, 228-232 Abb. 67-70). Magee's chronology rests on finds from Rumaylah, Tell Abraq and Shimal, sites all in

³ Magee does not cite this particular ¹⁴C data as published (source: Yule/Weisgerber 1988: 32; Yule 2001: 153 Tab. 6.8). In fact BLN 2747 derived from grave of LIA type (S101130 in cemetery S10) which paradoxically contained an EIA stone bowl (artefact class Sg27, Yule 2001: 153 table 6.8). This problematic dating is of little use. A second ¹⁴C assay escaped Magee's attention from a context in Saruj, the EIA finds of which are unpublished (Yule 2001: 149 table 6.4 citing Hannß 1991: 55, 103 Profil 2151 Tab. 4). Given the fewness of the relevant determinations, it seems appropriate to point out the above-mentioned examples, whatever problems they might have.

lab no.	find-spot	¹⁴ C age BP	cal. age W1 BCE
Kn 3499	L1	2770 <u>+</u> 160	1210-810
BLN 2747	S101130	2730 <u>+</u> 60	915-822
Hv 14589	Saruj	3035 <u>+</u> 85	1429-1114

Selected radiocarbon determination from the early iron age contexts.

the U.A.E. But after 1999, stylistically EIA pottery was catalogued from sites such as the Bawshar honeycomb cemetery (Yule 1999b), al-Maysar M34 (Yule 1999a), M42, Samad S10, S21, S22 and other sites (Yule 2002) in the Sultanate. It would be desirable one day for Emirates specialists to integrate these sites into the discussion on chronology on early "Oman".

Excavated in 1981, the **late iron age** (Samad period) hill fort M34 (Yule 1999a: 129 Fig. 7; 137) contained some handmade pottery of a kind associated with the early iron age, but the majority are classified as "Samad ware(s)" of the subsequent period (Weisgerber 1982: 86-91 figs. 4-9; Yule 1999: 139 fig. 17, 141 Fig. 18). Such are also usually hand-made; but they evidence different forms, unfortunately, few of which are readily datable by means of typological comparison (Yule 2005: 307-311). The TL assay of 130 CE \pm 150 for M34 places it early in the Samad sequence, and tacks down nicely this part of the LIA. The TL determination fits the archaeologically early nature of the LIA finds.

Owing to their limited \pm factors, TL data firm up the ¹⁴C chronology and provide informational points where assays are defective or unavailable, both in the early and late pre-Islamic chronology. Especially in the EIA they offer an anchoring point, during which the carbon data suffer from isotope diffraction and calibrate erratically. They also form a useful corrective to the ¹⁴C assays of the succeeding LIA age, which range down to 1000 CE, some of which now seem to be too low, and are unsupported archaeologically.

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Hd TL	Site	type
number		
7a1	Bil Kaher	furnace ceramics
9a1	Wadi Salh	furnace ceramics
9b1	Wadi Salh	furnace ceramics
9c1	Wadi Salh	furnace ceramics
10a1	al-Maysar M42 section 2	pithos
10c1	al-Maysar M1 house 1. loc. 10	pot sherd
10c2	al-Maysar M1 house 1. loc 10	furnace ceramics
10d1	al-Maysar M1 house 6. TT. 27. loc 36	red furnace ceramics
10d2	al-Maysar M1 house 6. TT. 27. loc 36	black furnace ceramics
10f1	al-Maysar M1 house 1. TT. 21. pl 3	furnace ceramics
10g1	al-Maysar M1 house 1. TT. 21. pl 3	furnace ceramics
10h11	al-Maysar M34 room 6	pot sherd
12a1	Lizq L1 trench 2, 40 cm deep	pot sherd
12a2	Lizq L1 trench 2, 40 cm deep	pot sherd
14a1	al-Batin	furnace ceramics

Table 1: Ceramic samples used for TL dating

Hd TL	fraction	equivalent dose ED	intercept I _o	archaeodose AD
number		(Gy)	(Gy)	(Gy)
7a1	fg	9.83	3.62	13.45
9a1	fg	8.44	2.69	11.13
9b1	qu	5.42	0.27	5.69
9c1	fg	9.89	0.83	10.72
	qu	5.04	0.26	5.30
10a1	fg	7.29	2.24	9.53
10c1	fg	8.72	4.88	13.60
10c2	fg	11.23	2.92	14.15
10d1	fg	7.25	2.89	10.14
	qu	4.46	1.71	6.17
10d2	fg	13.55	1.92	15.47
10f1	fg	12.40	5.03	17.43
10g1	fg	8.82	5.29	14.11
10h1	fg	6.03	1.96	7.99
12a1	fg	6.62	1.09	7.71
12a2	fg	6.91	3.19	10.10
14a1	qu	8.58	2.35	10.93

Table 2: Archaeodose data for fine-grain (fg) and quartz (qu) fractions

Hd TL	uranium	thorium	potassium
number	$(\mu g/g)$	(µg/g)	(wt%)
7a1	2.22	13.0	2.07
9a1	1.38	9.2	1.71
9b1	1.66	11.9	1.82
9c1	1.70	11.6	1.68
10a1	1.61	15.1	1.68
10c1	1.66	7.3	1.35
10c2	2.22	11.7	1.71
10d1	1.68	7.8	1.37
10d2	2.07	10.2	1.90
10f1	2.33	12.8	1.77
10g1	2.00	11.5	1.68
10h11	1.94	13.2	1.67
12a1	2.15	9.7	1.32
12a2	2.44	9.9	1.46
14a1	3.50	11.0	1.77

Table 3: Uranium, thorium und potassium contents in ceramic samples

Hd TL	porosity	fraction	a-value	alpha	beta	gamma	total
number	(%)			(mGy/a)	(mGy/a)	(mGy/a)	(mGy/a)
7a1	28.3	fg	0.118	1.494	2.332	0.226	4.092
9a1	26.1	fg	0.157	1.344	1.824	0.228	3.396
9b1	26.3	qu	-	-	1.844	0.342	2.186
9c1	22.6	fg	0.160	1.721	1.919	0.304	3.943
		qu	-	-	1.746	0.304	2.050
10a1	16.9	fg	0.142	1.824	2.019	0.380	4.223
10c1	12.4	fg	0.118	1.697	1.830	0.319	3.846
10c2	29.6	fg	0.160	1.894	2.003	0.319	4.216
10d1	30.1	fg	0.127	1.050	1.545	0.289	2.884
		qu	-	-	1.406	0.289	1.695
10d2	22.7	fg	0.186	1.993	2.112	0.289	4.394
10f1	27.4	fg	0.142	1.810	2.102	0.380	4.292
10g1	26.9	fg	0.115	1.305	1.950	0.342	3.598
10h11	10.6	fg	0.164	2.054	2.020	0.243	4.317
12a1	13.2	fg	0.127	1.371	1.661	0.228	3.259
12a2	17.2	fg	0.101	1.157	1.813	0.228	3.198
14a1	27.5	Qu	-	-	2.021	0.418	2.439

Table 4: Dose rate data.

fg=fine-grain and qu=quartz

Table 5: TL age data of ceramics

Site	Hd TL	technique	TL age	TL date	1σ total (a)	archaeological
	no.		(a)			phase
Rawdah/Bil Kalher	7a1	fg	3290	1310 BCE	<u>+</u> 250	BA
Wadi Salh	9a1	fg	3280	2590 BCE	<u>+</u> 320	EBA
Wadi Salh	9b1	qu	2600	620 BCE	<u>+</u> 260	EIA
Wadi Salh	9c1	fg	2720) 2650*	670 BCE	<u>+</u> 250	EIA
		qu	2590)			
al-Maysar M42 section 2	10a1	fg	2260	280 BCE	<u>+</u> 170	EIA
al-Maysar M1 house 1 loc 10	10c1	fg	3540	1560 BCE	<u>+</u> 260	EBA
al-Maysar M1 house 6 TT 27 loc 36	10c2	fg	3360	1380 BCE	<u>+</u> 290	EBA
al-Maysar M1 house 6 TT 27 loc 36	10d1	fg	3520) 3580*	1600 BCE	<u>+</u> 270	EBA
		qu	3640)			
al-Maysar M1 house 6 TT 27 loc 36	10d2	fg	3520	1540 BCE	<u>+</u> 300	EBA
al-Maysar M1 house 1 TT 21 pl 3	10f1	fg	4060	2080 BCE	<u>+</u> 330	EBA
al-Maysar M1 house 1 TT 21 pl 3	10g1	fg	3920	1940 BCE	<u>+</u> 310	EBA
al-Maysar M34 room 6	10h11	fg	1850	130 CE	<u>+</u> 150	LIA
Lizq L1 trench 2. 40 cm deep	12a1	fg	2370	390 BCE	<u>+</u> 190	EIA
Lizq L1 trench 2. 40 cm deep	12a2	fg	3160	1180 BCE	<u>+</u> 250	EIA
al-Batin	14a1	qu	4480	2500 BCE	<u>+</u> 1000	Hafit

* age combined of fine-grain and quartz ages, fg=fine-grain and qu=quartz

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Fig. 1. TL recording at al-Maysar settlement, M42.



Fig. 2. TL recording at al-Maysar settlement, M1.

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Fig. 3. TL recording at al-Maysar settlement, M1.



Fig. 4. TL recording at al-Maysar settlement, M1.

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Fig. 5. TL recording at Lizq fortress, L1.