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ISA Special Issue Paper

Luminescence dating of mortar and terracotta from a Royal Tomb at Ulaankhermiin Shoroon Bumbagar, Mongolia

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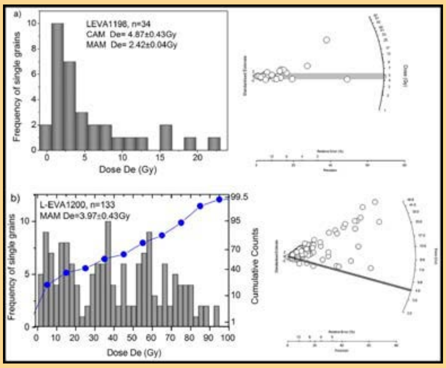
Abstract The spectacular royal tomb "Ulaankhermiin Shoroon Bumbagar" was discovered in Bulgan province, Mongolia, in 2011. Excavation of the site revealed its internal structure; a slope of 42 meters in length leading down to the underground mausoleum at a depth of 7 m below the ground. Archaeological investigations provided the site with an independent age control suggesting the construction date of the Royal tomb to the last quarter of the VII century.

In this study, we directly date different materials from the site, such as terracotta figurines, mortar and host sediment, using infrared-stimulated (IR_{50}) and post-infrared infrared stimulated (pIRIR) luminescence techniques. The most accurate estimate of 670 ± 70AD and 550 ± 110AD was obtained for terracotta figurines using IR_{50} and pIRIR on aliquots of 4–11 μm polymineral grains. By comparison, sand-sized quartz single grain measurements on the embedded sediment yielded normal equivalent dose (D_e) distributions with a few outliers, justifying the use of the central age model (CAM) for age calculation, and yielded construction dating to the 780 ± 140AD. Finally, sand-sized quartz single grain measurements on wall mortar revealed incomplete bleaching of grains, requiring detailed analysis using statistical approaches; from this we identified the most well bleached population age of 740 ± 130AD, using the lowest 5%. The luminescence ages are in general agreement with the historically expected age.



Statement of significance The archaeological site at Ulaankhermiin Shoroon Bumbagar, Mongolia (with an independent age control) offers a great opportunity to apply luminescence dating methods, such as pIRIR on 4–11 μm polymineral fine grains, and single grain measurements on 200–250 μm quartz grains, to various materials, such as terracotta figurines, mortar-like limestone which covered the walls and embedded sediment in order to test the accuracy of optically stimulated luminescence (OSL) dating techniques. Compared to all other datable archaeological materials, mortar has the advantage of being intentionally manufactured at each stage of construction, since it cannot be recycled. However, dating of the well bleached grains of mortar, which provided the greatest challenge in this study, was achieved using the lowest 5% estimate, yielding an age of 740 ± 130AD. pIRIR on heated polymineral (feldspar-bearing) fine grain aliquots from terracotta figurines was tested, yielding IR_{50} and $pIRIR_{180}$ dates in agreement with the CAM estimate from the host sediment.

Keywords Luminescence, Single grain, Fine grains, pIRIR, Mortar

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Data availability The authors confirm that all data underlying the findings are fully available without restriction. All relevant data are contained within the paper.

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Introduction

In 2010–2011, a joint Mongolian–Kazakh archaeological expedition (the Institute of History of the Academy of Sciences of Mongolia and the Eurasian National University Gumilev) excavated the royal tomb "Ulaankhermiin Shoroon Bumbagar" in Maikhan Uul. The tumulus or kurgan of Maikhan Uul is situated in the Bayan–nuur promontory of the Bulgan Province (Fig. 1), at a distance of 210 km to the northwest of Ulaanbaatar, Mongolia. Another kurgan to the south, called "Ulaan Kherem" (The red city) valley has been known for the last 10 years, but its excavations started only after a special non-destructive technique had been developed, which allowed non-destructive exploration of the underground monument.

Excavations of the royal tomb Ulaankhermiin Shoroon Bumbagar revealed an internal structure comprising a downward-sloping ramp leading to the subterranean mausoleum. The ramp length of 42 m, starting from the ground level and slopes toward the underground mausoleum at the depth of 7 m below the ground level (Ochir and Erdenebold 2012a). The walls of the long corridor were covered by mortar and chalk, upon which images were painted (Sartkojauli, 2012, 80). Excavations of the mausoleum and its surroundings (Fig. 1) recovered more than 117 terracotta figurines of warriors on horseback, and 150 gold wares including a golden crown, a gold plate, golden ring and ancient gold coins (presumably Byzantine and Sogdian) (Ochir and Erdenebold 2012a). In 2008–2009, archaeological excavations by Ochir (Ochir 2012b) were conducted on a similar construction located on the left side of the river Tuul in Zaamar, Mongolia, these were dated to the end of VII century based on historical sources. Stylistic dating for Ulaan Khermiin Shoroon Bumbagar, due to its similarity with the tomb of Ochir's excavations, indicates the former to be of similar age and therefore provides an independent age control with suggested construction dating to the last quarter of VII century.

The independently determined late VII century age, and availability of different mineral-bearing materials, provides an opportunity to test the accuracy of optically stimulated luminescence (OSL) dating techniques. At Ulaankhermiin Shoroon Bumbagar there are different materials available which may prove suitable for the luminescence dating, e.g. fired terracotta figurines, mortar-like limestone covering the walls of the underground construction, and sediment embedded within the wall mortar. Various ceramic material (pottery, bricks, cooked clays, clay-cores) can be dated by thermoluminescence (Martini and Sibilia 2001, 241), the OSL approach is less widely used. However, it is a technique worth considering; OSL dating of Mongolian pottery shards (Solongo *et al.*, 2013) provided an approximate age for pottery production, which was consistent with the radiocarbon results.

Although OSL dating of archaeological materials such as mortar and fired terracotta figurines holds promise, limitations to accuracy with respect to the resetting of the OSL signal need to be considered in the experimental framework. A fundamental assumption contained within the OSL approach is that the mineral grains are completely bleached of all residual OSL signal either by sunlight or heat during the event which is being dated – in this case, the construction of the tomb. This assumption appears to be the case with heated sediments such as heat-retainer hearthstones (Rhodes 2008; Fanning *et al.*, 2007) or terracotta figurines during the firing process, and therefore it may be sufficient to undertake dating using aliquots of grains. However, sediment embedded within the wall mortar of the tomb may have been incorporated at various stages of the construction process, and from various sources, and may not necessarily have experienced a complete resetting of the OSL signal. In this case, dating with single grains is advisable in order to identify whether complete bleaching took place, and therefore what form of statistical age modelling will optimize the resulting age accuracy.

Compared to all other datable materials, mortar has, in fact, the advantage of being purposely made at each stage of construction, not being recyclable (Bøtter-Jensen *et al.* 2000, 841). Consequently, the sediments incorporated into the mortar are more likely to have been exposed to sunlight, thereby resetting the OSL signal, at the precise time of tomb construction. Attempts to establish reliable OSL dating protocols based on the assumption of a complete bleaching of quartz grains during the preparation were made by Zacharias, Mauz and Michael (2002) and Goedicke (2003, 409). Attempts were also made using OSL on polymineral fine grain fraction enriched in quartz (Stella *et al.* 2013, 153) of mortar. However, in many cases, insufficient bleaching affected the distribution of individual equivalent doses; the single-grain technique is usually used to extract sub-populations corresponding to the event being dated using specific statistical models.

This paper examines the potential of luminescence dating to test the chronology of a selection of different archaeological materials. We examine and compare quartz single-grain D_e estimates on mortar and host sediments and pIRIR on polymineral (feldspar-bearing) fine grain aliquots from terracotta figurines to test the robustness of the luminescence methods for accurate De determination.

Sample preparation

The internal sections of all samples – which had not been exposed to recent sunlight – were carefully crushed and sieved. For the mortar and embedded sediment samples, the coarse grains (>100 μm) were treated with hydrogen peroxide (H_2O_2, 15 and 30%) to remove any organic material, with hydrochloric acid (HCl, 10%) to dissolve carbonates, with a heavy liquid (lithium heterotungstate) at a density >2.63 g/cm^3 to separate quartz from feldspars, and with hydrofluoric acid (HF, 40% for 60 min) to remove the alpha-radiation affected outer rinds of the quartz

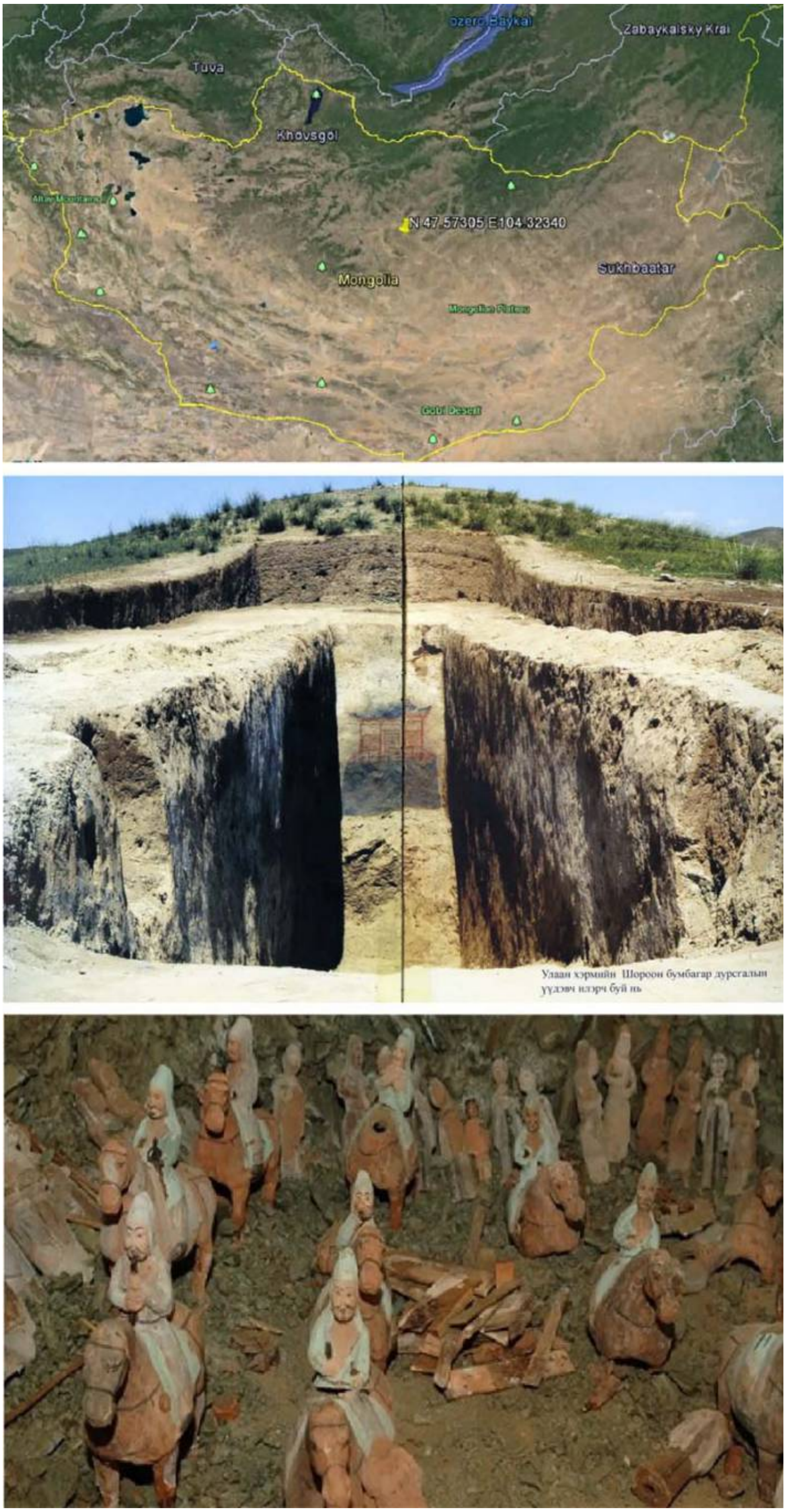

Figure 1 The Royal Tomb at Ulaankhermiin Shoroon Bumbagar: (A) location of the archaeological site; (B) the ramp leading to the underground mausoleum; (C) panorama of warriors. Photos provided by (Ochir and Erdenebold 2012a) and Ulziibayar Sodnom.

Table 1 Summary of dosimetry data.

Lab code	Sample	K (%)	U (ppm)	Th (ppm)	H_2O (%)	D_r (mGy/a), fg	D_r (mGy/a),Q
L-EVA-1198[a]	sediment	2.8 ± 0.1	2.6 ± 0.2	9.8 ± 0.4	16[c]		3.3 ± 0.3
L-EVA-1199[b]	Terracotta	0.6 ± 0.03	3.7 ± 0.2	6.9 ± 0.2	11[c]	4.7 ± 0.2[e] 5.2 ± 0.2[f]	
L-EVA-1200[b]	mortar	1.1 ± 0.1	2.6 ± 0.2	9.6 ± 0.3	0.6[d]		2.6 ± 0.2

[a] High-resolution gamma spectrometry (Felsenkeller, Dresden).
[b] Neutron activation analysis (NAA) (CEZA Mannheim).
[c] Water content as modelled.
[d] Water content as measured.
[e] '*a*-value' of 0.08 ± 0.02.
[f] '*a*-value' of 0.12 ± 0.02.

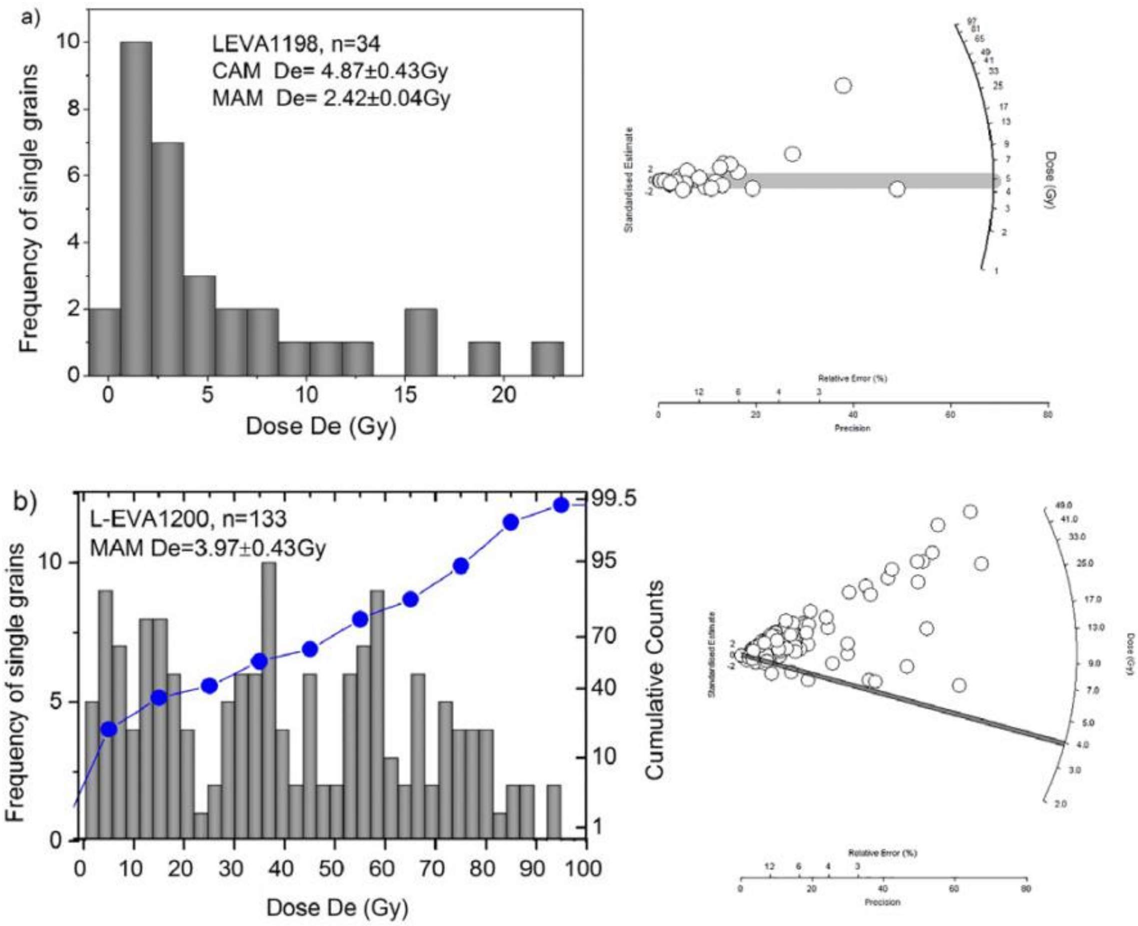

Figure 2 Quartz single grain *De* distributions obtained for a) sediment L-EVA-1199 and b) mortar L-EVA-1200. The corresponding estimates were a) CAM D_e and OD values of 4.87 ± 0.4Gy and 46%, and b) MAM D_e and OD of 3.97 ± 0.4Gy and 87%, respectively. On the right-hand side of each figure *De* values displayed as radial plots are shown for a) well-bleached sediment sample L-EVA-1199, b) an incomplete bleached mortar sample L-EVA-1200. The solid lines indicate the *De* estimates obtained using a) CAM and b) MAM.

grains. Finally, the samples were treated again with hydrochloric acid (HCl, 10%) to remove acid-soluble fluorides and then re-sieved with 180- and 212-mm screens. For the fine-grained terracotta, mortar and embedded sediment samples, polymineral fine grains (4–11 μm) were extracted treated with H_2O_2 (15 and 30%), 10% HCl, dispersed using sodium oxalate and then isolated via Stokes settling.

All luminescence measurements were made using two Risø DA-20 TL/OSL readers. For single-grain measurements, Risø DA-20 reader with a single-grain attachment, equipped with green laser excitation and detection via U340 filter was used. Single grains were measured using the SAR-protocol (Wintle and Murray 2006, 369), employing a preheat temperature of 260°C for 10 s and a cut-heat of 220°C at a heating rate of 5°C/s. Signals for D_e estimation were calculated from the initial 0.1 s of the single-grain OSL intensity less a background based on the sum of the final 0.3 s. Dose estimates were accepted if the relative uncertainty on the natural test dose response and recycling ratio were less 20%.

Measurement of the polymineral fine grain aliquots was undertaken using infrared (IR) LEDs (870 nm,

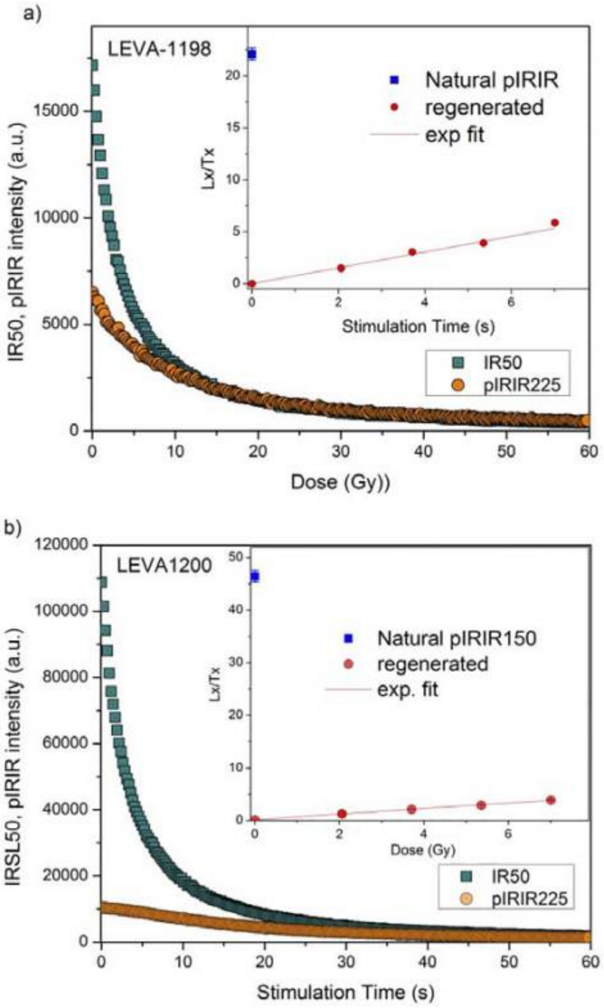

Figure 3 IR_{50} and $pIRIR_{225}$ measurements using fine grains of a) sediment sample L-EVA-1198 and b) mortar sample L-EVA-1200. Inset shows corresponding DRC indicating that the natural signal intercepts the dose response curve at doses about 120Gy, confirming the suggestion that the fine grain samples were incompletely bleached.

130 mW/cm^2) and the IRSL signal was detected through a blue filter combination (Schott BG-39, Corning 7–59). These samples were measured using the post-IR infrared luminescence (pIRIR) approach (Buylaert *et al.* 2009, 560), (Buylaert *et al.* 2012, 759). The integrated luminescence intensity of the initial 4 seconds (1–10 channels) of the IR_{50} and 7.6 seconds (1–20channels) of the pIRIR stimulation for 60 seconds (less a background based on the sum of the final 10 seconds for the IR_{50} and final 20 seconds for the pIRIR) was used.

The environmental radiation dose rates were calculated from analyses of subsamples collected from the external layer of the ceramic and mortar samples. Neutron activation analysis (NAA) was undertaken on all samples. In addition, one sediment sample was measured using high resolution gamma-spectrometry at the Felsenkeller, RFI Dresden, Germany. For polymineral fine-grains an '*a*-value' of 0.08 ± 0.02 and 0.12 ± 0.02 were used for IR_{50} and $pIRIR_{180}$ measurements, respectively. The conversion factors of (Guérin, Mercier and Adamiec 2011, 5) were used to calculate the total dose rates. The concentrations of K, U and Th together with the measured water content, total dose rates for fine grains (fg) and quartz (Q, 200 μm) are given in Table 1. The uncertainties mentioned with the radionuclide activities and calculated total dose rates are random; all uncertainties represent 1σ.

Luminescence results

Mortar dating by single-grain measurements on quartz

Dating of the mortars from Ulaankhermiin Shoroon Bumbagar is assumed to represent the event whereby the mortar was prepared; in this case, when grains of sand (including quartz and feldspar) were extracted, possibly sieved, added to and mixed with the lime. Mortar dating by OSL is still challenging (Goedicke 2011, 42): previously published results obtained from coarse-grained quartz using different analytical methods showed wide distributions indicative of incomplete bleaching. The determination of dose distribution on a single grain level might reveal the presence of an incomplete bleaching or mixed grain populations, allowing to extract sub-population of interest by using statistical tools such as the MAM (Minimum Age Model) or the CAM (Central Age Model) (Galbraith and Roberts 2012, 1).

We first measured single grains of embedded sediment and mortar from the Ulaankhermiin Shoroon Bumbagar (Samples L-EVA-1198 and L-EVA-1200). About 300 grains from L-EVA1198 were measured, n=34 of which passed the rejection criteria, the doses range from 0.5 ± 0.1 to 92.0 ± 2.4 Gy giving a CAM dose and over-dispersion (OD) of 4.9 ± 0.4 Gy and 46%, respectively. Single-grain D_e distributions of the sediment showed a normal distribution with a few outliers, justifying the use of the CAM. Figure 2A shows single-grain dose distributions from the host sediment L-EVA1198; in the corresponding radial plot the solid line indicates the CAM estimate.

In contrast, altogether 1100 grains from mortar sample L-EVA1200 were measured, of which n=133 were accepted. The D_e estimates range from 1.4 ± 0.2 Gy to 120.0 ± 9.5 Gy giving a CAM dose and OD of 30.7 ± 2.4 Gy and 87%, respectively (see Fig. 2B). The shape of the dose distribution indicates that the mortar sample is affected by incomplete bleaching, and therefore justifies the use of the Minimum Age Model (MAM). Applying the MAM to the dose distribution, we obtained 4.0 ± 0.4 Gy. Given the OD valued >50%, the well bleached population most likely comprises the youngest 5%. This latter leading-edge approach results in a dose of 3.3 ± 0.2 Gy. Figure 2B shows single-grain De distributions from mortar sample L-EVA1200. The corresponding radial plot is also shown; the solid line indicates the MAM estimate.

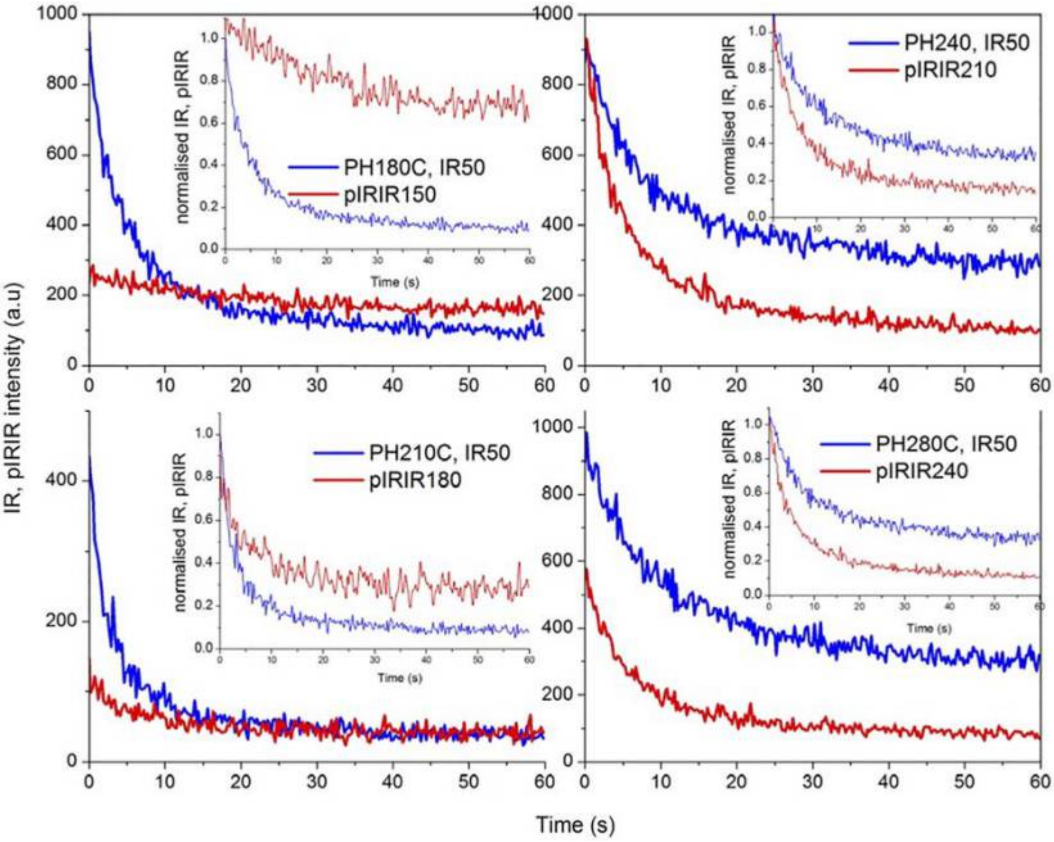

Figure 4 IR_{50} and pIRIR natural decay curves from fine grain aliquot L-EVA1199 measured at different preheat temperatures and stimulation temperatures as indicated. Inset showed normalised natural IR_{50} and pIRIR decay curves. Two aliquots were used for each preheat and stimulation combinations.

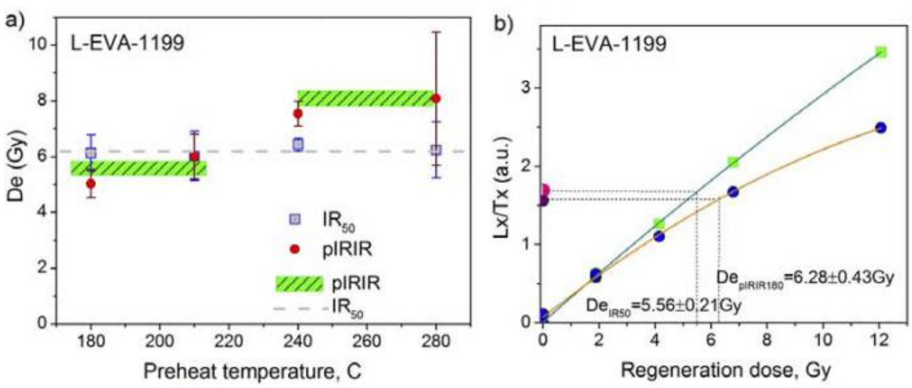

Figure 5 (A) D_e – preheat plot of the IR_{50} and pIRIR measurements for a fine grain sample LEVA1199. The overall average for IR_{50} is 6.2 ± 0.2 Gy and represented as dashed line, a green band represents pIRIR plateau. Squares represent the mean data collected using IR_{50}; diamonds represent the mean value of pIRIR measurements, which are made at stimulation temperatures 30°C higher than the preceding preheat. Three aliquots were measured for each preheat temperature and error bars represent one standard deviation. No fading-corrections were made, no residual doses were measured. (B) Dose-response curve Lx/Tx as a function of regeneration dose for IR and pIRIR measurements.

Table 2 Luminescence results

Sample ID	Mineral	Method	Model	*n*	De(Gy)	Age (*a*)	Date (AD)
L-EVA-1198, sediment	Q	SG	CAM	34	**4.9 ± 0.4**	1230 ± 140	780 ± 140
			MAM		2.4 ± 0.04	600 ± 50	1400 ± 50
L-EVA-1200, mortar	Q	SG	CAM	133	30.7 ± 2.4	–	
			MAM		**4.0 ± 0.4**	1540 ± 200	480 ± 200
			5%	7	**3.3 ± 0.2**	1270 ± 130	740 ± 130
L-EVA-1199, terracotta	Fg	IR_{50}	CAM	23	**6.3 ± 0.15**	1340 ± 70	670 ± 70
		$pIRIR_{180}$	CAM	20	7.6 ± 0.44	1460 ± 110	550 ± 110

Terracotta dating using pIRIR measurements on fine grains

We applied the recently introduced post-IR IRSL method (Buylaert *et al.* 2012, 759) to the polymineral fine grains (Buylaert *et al.* 2009, 560) from the terracotta sample L-EVA-1199, as well as to the two mortar and embedded sediment samples L-EVA-1198 and L-EVA-1200. Using stimulation energies of about 1.4 eV, i.e. the approximate IR excitation resonance of feldspars, for the polymineral fine grain fraction it is assumed that the measured emission in the violet-blue band is predominantly caused by feldspar minerals. The corresponding IR and $pIRIR_{225}$ measurements using fine grains of sediment and mortar samples are shown in Fig. 3; the insets show the dose-response curve indicating that the natural signal exceeds the respective dose-response curves, confirming the suggestion that the samples were incompletely bleached.

Thereafter, pIRIR was applied to fine grains of terracotta sample, since the fired sample is expected to be well bleached. The behaviour of pIRIR signals from fine grains of terracotta L-EVA-1199 were investigated under various preheat and stimulation conditions in order to choose the appropriate experimental conditions for young sample under the study. Fine grain aliquots were preheated to temperatures between 180°C and 280°C and their natural IR_{50} and pIRIR arising from stimulation temperatures ranging from 150°C to 240°C were measured. The corresponding experimental IR_{50} and pIRIR decay curves are shown in Fig. 4A, and the comparison of these curves revealed an increase in the pIRIR luminescence intensity with the temperature. Following preheating at 180°C, the pIRIR natural signal intensities are around $\sim$200 counts, increasing to the maximum intensity following a preheat at 240°C. The inset of Fig. 4 shows the normalised natural decay curves, it displays clearly that the decay rates of IR and pIRIR signals differ from each other. The $pIRIR_{150}$ and $pIRIR_{180}$ decayed slower than the preceding IR_{50}. By comparison, beyond the preheating temperature of 240°C, the $pIRIR_{210}$ and $pIRIR_{240}$ decay more rapidly than the preceding IR_{50}. The similar observation have been made by (Solongo and Tengis 2015) who used fitting procedure to decay curves. Nevertheless, it is beyond the scope of this study to address the question of the IR and pIRIR signal analysis.

To check which preheat and stimulation combinations are the most appropriate for the fine-grained terracotta sample L-EVA1199, a preheat temperature plateau test was measured using pIRIR approach. We varied the preheat temperatures from 180°C to 280°C and the pIRIR stimulation temperature from 150°C to 240°C respectively; the results are shown in Fig. 5A. The *De* values obtained from the IR_{50} (squares) measurements represent the average of three measurements, and yield a plateau at 6.2 ± 0.2 Gy for the whole range of the preheat temperature.

However, the pIRIR *De* (filled circles) values gradually increase towards the higher preheat and stimulation temperatures. In our experiment, *De* obtained using the $pIRIR_{180}$ (following a preheat of 210°C) is 6.0 ± 0.3 Gy ($pIRIR_{180}$) while the $pIRIR_{240}$ signal (following a preheat of 280°C) gives 8.1 ± 2.4 Gy (n=3). A plateau, measured between 180°C and 210°C, shows a rising trend towards higher preheat and stimulation temperatures. This increase in plateau is in accordance with observations made by Solongo and Tengis (2014) and Reimann and Tsukamoto (2012, 180) for young samples who proposed the use of a much lower preheat (200°C) and pIRIR stimulation (180°C) temperatures in order to reduce the residual dose of pIRIR to $\sim$1 Gy in their $pIRIR_{180}$ protocol. Based on these measurements, it can be concluded that the preheat temperature below than 240°C (e.g. stimulation temperature 210°C) would be appropriate for sample LEVA1199.

For the dose evaluation, altogether 24 aliquots were measured using $pIRIR_{180}$, while n=20 were accepted, which yield *De* = 7.6 ± 0.4 Gy and an OD = 16%. It is worth to note that from IR_{50} measurements n=23 were accepted which yield *De*=6.3 ± 0.2 Gy and OD = 6%. Figure 5B shows a dose-response curve from fine grains L-EVA1199 measured using $pIRIR_{180}$.

Luminescence ages

The luminescence dating results are summarised in Table 2. The minerals and measurement methods are listed, the applied model and the number of aliquots are given. *De* values, which are accepted for age evaluation and are consistent with the expected age are shown bold.

The single-grain dose distribution of an incompletely bleached mortar LEVA-1200 is still challenging: only 133 grains from measured altogether n=1100 were accepted. The MAM model yields an OSL age (4.0 ± 0.4 Gy), indicating that the coating the walls with mortar took place 1540 ± 200 years ago. However, using the lowest 5%, which corresponds to seven single grains only, we obtained an age 1270 ± 130 years as the best estimate of the dose distribution. It should be noted that both MAM 480 ± 200 AD and lowest dose (740 ± 130 AD) OSL dates are consistent within 1σ of the independent historical date, e.g. the end of VII century.

Single-grain data from the host sediment LEVA-1198 provided an OSL age of 1230 ± 140 (CAM, n=34, OD=46%) , which corresponds to the date 780 ± 140 AD. This is consistent with the expected result. For the terracotta sample LEVA1199, both IR_{50} and $pIRIR_{180}$ ages of 1340 ± 70a and 1460 ± 110a provided dates 670 ± 70AD and 550 ± 110AD, respectively. These are in agreement within the 1σ of the historically expected date.

Conclusions

The archaeological site at Ulaankhermiin Shoroon Bumbagar (Mongolia) offers a great opportunity to apply different luminescence dating methods, such as post-IR IR on 4–11 µm fine grains, and single grain measurements on 200–250 µm quartz grains, to various materials, such as terracotta figurines, mortar-like limestone, which covered the walls and embedded sediment in order to reconstruct the geo-archaeological chronologies.

Notably IR_{50} date (670 ± 70AD) is regarded as the best estimate for fine grains of terracotta which is in agreement with the CAM estimate of the host sediment. $pIRIR_{180}$ dating of fired fine grains of terracotta appears problematic due to low sensitivity. Dating of the well bleached grains of mortar, which provided the greatest challenge in this study, was achieved using the lowest 5% estimate, yielding date of construction 740 ± 130AD.

Conflicts of interest

The authors confirm there are no conflicts of interest

Author biographies

At the time that this work was carried out, SaS was a guest scientist at the Luminescence laboratory, MPI Leipzig working on luminescence dating and Principal investigator at the Institute of Physics and Technology, Mongolian Academy of Sciences. AO is Professor of History at the Mongolian Academy of Sciences. KF was a leader of the Luminescence laboratory at the MPI, Leipzig. ST is researcher at the IPT, Mongolian Academy of Sciences. JJH is Professor at the Max Planck Institute for Evolutionary Anthropology in Leipzig (Germany).

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