



pIRIR and IR-RF dating of archaeological deposits at Badahlin and Gu Myaung Caves – First luminescence ages for Myanmar

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ABSTRACT

Reliable chronologies are essential for understanding the timing and routes of human dispersal through Southeast Asia, both of which remain open questions. This study provides luminescence chronologies for two archaeological sites in Myanmar—Badahlin Cave and Gu Myaung Cave—from which Palaeolithic artefacts have been recovered. We applied single-grain post-infrared infrared stimulated luminescence (pIRIR) and multi-grain infrared-radiofluorescence (IR-RF) dating methods to potassium-rich feldspar (K-feldspar) grains extracted from the sedimentary deposits at these two sites. Luminescence characteristics of the K-feldspar extracts showed that the procedures were well suited for dating. The single-grain pIRIR ages suggest human occupation of Badahlin and Gu Myaung Caves by ~30 ka and ~25 ka, respectively, although the age estimates for Gu Myaung Cave are much older than the radiocarbon ages. The ages obtained using the IR-RF signal are even older, which we attribute to insufficient bleaching of this signal—and perhaps also the pIRIR signal—and the probable existence of a substantial residual dose at the time of sediment deposition.

1. Introduction

Badahlin and Gu Myaung Caves both lie at the foot of the Shan Plateau, which stretches eastward from central Myanmar into northern Thailand (Fig. 1). Badahlin Cave was first excavated in 1969 (Aung Thaw, 1971), when stone artefacts and animal bones were recovered. Radiocarbon (¹⁴C) dating indicated a maximum age for the cultural materials of ~13,400 years BP (Aung-Thwin, 2001). In 2013, we conducted a 2.5 m-deep excavation at Badahlin Cave 2, which is separate from the site of Badahlin Cave 1 where the earlier excavations were undertaken. We also conducted a 4 m-deep excavation at Gu Myaung Cave, which had not previously been excavated, and collected three charcoal samples that gave ¹⁴C ages ranging from ~3 to ~15 ka BP (Table S3). The ¹⁴C ages for Badahlin Cave 1 and Gu Myaung Cave provide preliminary—and likely minimum—age constraints for these two sites, which have yielded Palaeolithic artefacts. Additional data are needed to test these chronologies and extend them into the deeper deposits.

Luminescence dating has been used previously to date

archaeological sites in mainland Southeast Asia. Although conventional optically stimulated luminescence (OSL) dating of quartz has been applied to some sites (e.g., Demeter et al., 2015; Forestier et al., 2015; Moncel et al., 2016), the utility of quartz OSL is commonly limited due to signal saturation or low intrinsic grain brightness (Roberts et al., 2005; Jacobs and Roberts, 2007). We encountered these difficulties using the quartz OSL signal during our initial tests of the sediment samples collected from Badahlin and Gu Myaung Caves. Consequently, alternative minerals, luminescence signals and dating procedures were required. In this study, we have used two luminescence signals—single-grain post-infrared infrared stimulated luminescence (pIRIR) and multi-grain infrared radiofluorescence (IR-RF)—to estimate the depositional age of sand-sized grains of potassium-rich feldspar (K-feldspar). Here we report the preliminary results of our investigations to augment the ¹⁴C chronologies for these two human occupation sites. To our knowledge, these are the first luminescence ages for archaeological sites in Myanmar (Aung et al., 2015).

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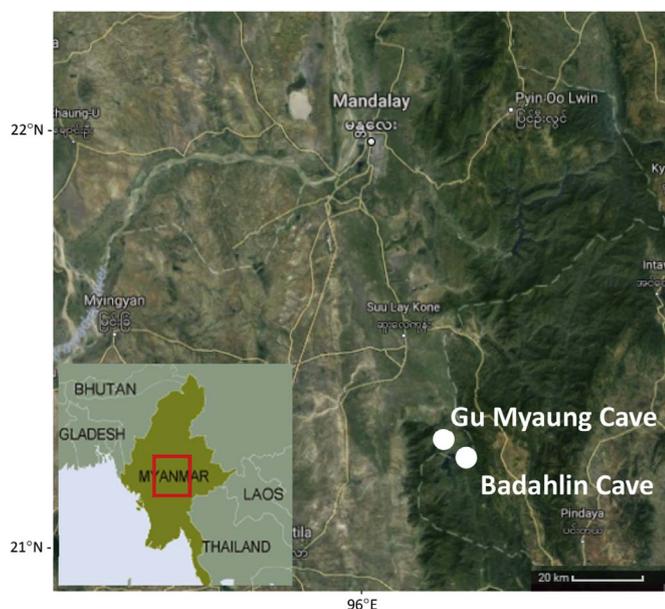


Fig. 1. Map of central Myanmar showing the locations of the two study sites.

2. Site and sample descriptions

2.1. Study sites

Badahlin Cave (21.13274°N, 96.34032°E) and Gu Myaung Cave (21.20156°N, 96.31926°E) are located in central Myanmar (Fig. 1). They are formed in Permian and Permo-Triassic limestone, which contains a number of karstic features, including sinkholes and phreatic tubes.

Badahlin Cave was formed phreatically and consists of several inter-connecting chambers. The cave roof is ~30 m thick and speleothems are abundant, with occasional roof collapses facilitating entry routes for humans into the cave complex. In one of the chambers, we excavated a location close to the wall to a depth of ~2.5 m (Fig. 2a); excavation details will be presented in a forthcoming archaeological report. The deposit is composed mainly of dark sandy silt that becomes enriched in clay with increasing depth. The sediment layers dip at a low angle towards the cave wall and the deposit is moist throughout, with a slight increase in moisture with depth.

In the upper part of the sequence (i.e., the top ~1 m), sediment layers are interspersed by thin flowstones. Calcified sediment and limestone inclusions were also found occasionally, the latter being usually cobble to pebble sized and irregularly shaped. Two excavations were undertaken: squares A, B and E relate to the first of these, and squares C and D to the second.

Gu Myaung Cave is also a phreatically formed limestone cave, which overlooks the Panlaung River. We excavated just inside the cave mouth to a depth of ~4 m (Fig. 2b); excavation details will be provided in an archaeological report in preparation. The upper ~1.5 m of deposit consists mostly of horizontally layered grey-brown silt with some clay and gravel, and hearth features with abundant white ash and charcoal. Boundaries between layers are well-defined, probably due to the short time since deposition and the lack of post-depositional disturbance. The number of hearth features declines below this depth, resulting in a less heterogeneous and structured deposit. Below a depth of ~2 m, no stratigraphic features are readily apparent and the deposit is enriched with clay and contains occasional large rocks.

2.2. Sediment samples

Samples were collected using light-safe steel tubes, 22 cm in length

and 5 cm in diameter. *In situ* measurements of the gamma-ray dose rate were made at each sample location by inserting a NaI(Tl) detector into the empty tube holes, and additional bags of sediment were collected from these holes for beta dose rate and moisture content measurements in the laboratory. Samples from Badahlin Cave were collected from relatively moist sediments in square A of the northeast section of the excavation, from 80, 150 and 240 cm depth below surface (Fig. 2a). Samples from Gu Myaung Cave were collected from the sediments in the northeast section of the excavation, from 140 cm and 202 cm depth below surface (Fig. 2b). The uppermost sample at each site coincides with the depth of the lowest artefact found during excavation. The other samples were collected from the underlying archaeologically sterile layers and were analysed to investigate the longer-term history of sediment deposition in these caves.

3. Methods

3.1. Sample preparation and instrumentation

Samples were prepared using standard methods—including wet-sieving, HCl acid and H₂O₂ treatments, and density separations—to isolate sand-sized grains (90–125 μm and 180–212 μm in diameter) of quartz and K-feldspar. Our initial investigations of individual quartz grains (180–212 μm in diameter) revealed that their inherent OSL signals (i.e., their response to a test dose) were very weak or their natural OSL signals were in saturation. Accordingly, we concentrated thereafter on K-feldspar grains of the same or smaller (90–125 μm) grain size, isolating them using a sodium polytungstate solution with a density of 2.58 g/cm³ and then etching them in 10% HF acid for 10 min to clean the surfaces of the grains and to reduce the thickness of the external alpha-irradiated layer.

All pIRIR measurements were carried out on Risø TL-DA-20 readers equipped with bi-alkali photomultiplier tubes (Electron Tubes Ltd 9235QB), blue-transmission filter packs (Schott BG 39 and Corning 7–59), infrared (IR) light-emitting diodes and lasers, and calibrated ⁹⁰Sr/⁹⁰Y beta sources (Bøtter-Jensen et al., 2003). Grains were loaded on to discs drilled with 100 holes, each 300 μm in depth and 300 μm in diameter, and each hole position was calibrated for its beta dose rate. For relevant experiments, samples were bleached using an external solar simulator (Dr Hönle UVACUBE 400). The IR-RF measurements were carried out on aliquots of ~2 mm in diameter (corresponding to a monolayer of between ~70 and ~230 grains, depending on the grain size) using a Freiberg Instruments Lexsyg research reader equipped with a photomultiplier tube sensitive in the near-IR (Hamamatsu H7421-50), a Chroma-D850/40 interference filter, a light source with different wavelengths and a ⁹⁰Sr/⁹⁰Y beta source (Richter et al., 2012, 2013). The Lexsyg reader also has an in-built solar simulator, which we used for all bleaching steps during IR-RF measurements.

3.2. Environmental dose rates

Beta dose rates for dried and powdered sub-samples of the sediments collected in the field were measured using a Risø GM-25-5 beta counter (Bøtter-Jensen and Mejdahl, 1988) and the procedures described in Jacobs and Roberts (2015). The gamma dose rates were measured in the field using an Exploranium GR-320 EnviSpec gamma spectrometer, and the cosmic-ray dose rates were estimated following Prescott and Hutton (1994). Adjustments were made for beta-dose attenuation (Guérin et al., 2012) and for the effect of moisture content on the external beta, gamma and cosmic-ray dose rates (Readhead, 1987; Nathan and Mauz, 2008). The measured (field) water contents were used for each sample and assigned a relative uncertainty of ± 25% (at 1σ) to accommodate any likely variations over the period of sample burial. The internal dose rate can be a significant contributor to the total dose rate for K-feldspar: we assumed an internal potassium content of 10 ± 2% (Smedley et al., 2012) and an internal Rb content of

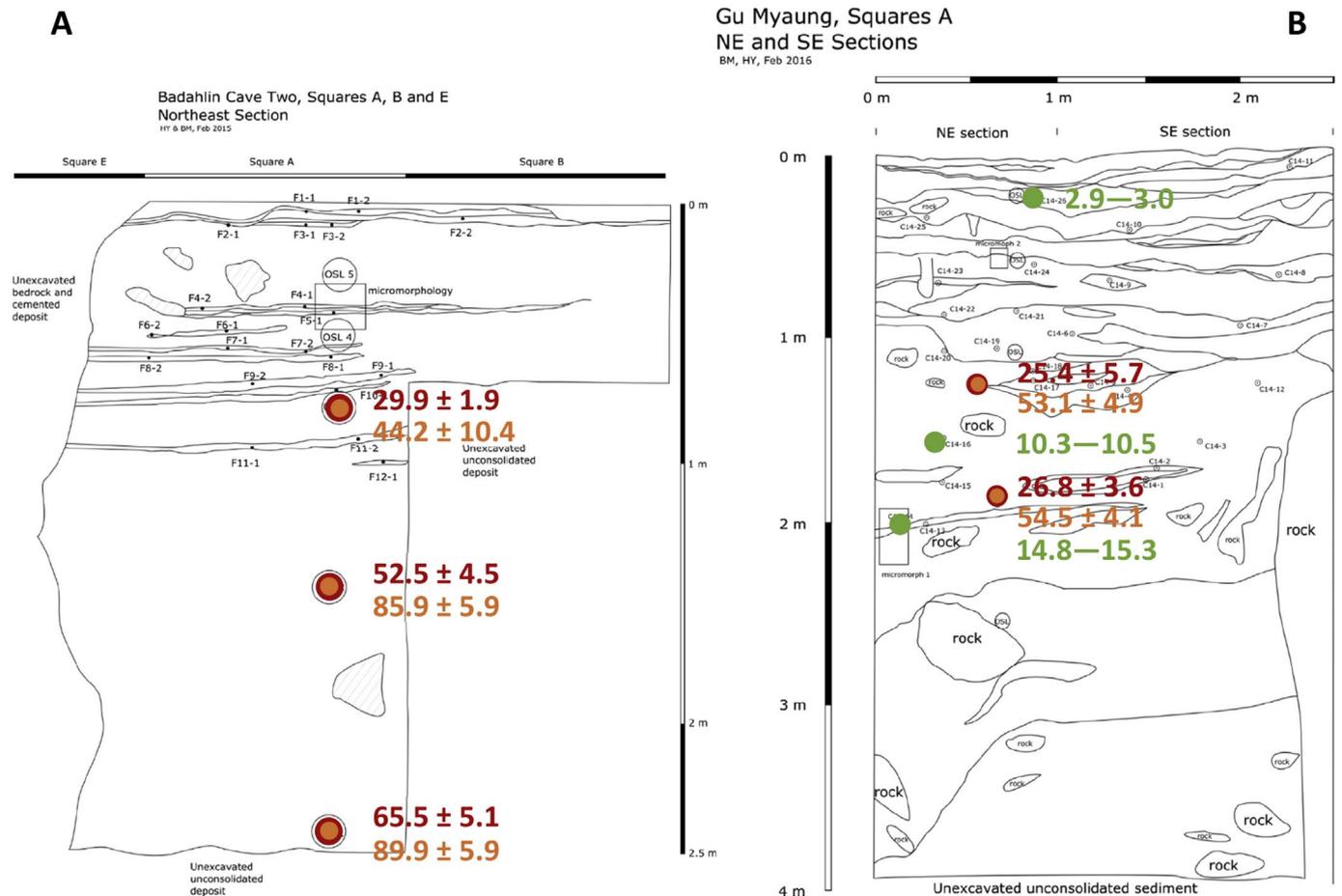


Fig. 2. Section drawings of excavated sections at Badahlin Cave (A) and Gu Myaung Cave (B), showing pIRIR ages (red), IR-RF ages (IRSAR, orange) and calibrated ^{14}C age ranges (green). The pIRIR and IR-RF ages ($\pm 1\sigma$ uncertainties) are in ka, and the ^{14}C age ranges (95.4% confidence interval) are in ka cal BP. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

$400 \pm 100 \mu\text{g/g}$ (Huntley and Hancock, 2001), resulting in estimated internal dose rates of 0.38 ± 0.08 and $0.67 \pm 0.12 \text{ Gy/ka}$ for grains of 90–125 and 180–212 μm diameter, respectively.

3.3. pIRIR measurement procedures

The pIRIR signal measured at elevated temperature has been reported to suffer much less from anomalous fading than the conventional IR stimulated luminescence (IRSL) signal measured at 50°C , and to possibly not fade at all (e.g., Li and Li, 2011; Buylaert et al., 2012b; Li et al., 2014; Roberts et al., 2015). However, it is also harder to bleach in natural sunlight than either the IRSL signal or the quartz OSL signal (e.g., Li and Li, 2011; Colarossi et al., 2015). Single-grain measurements, therefore, provide a useful means of examining the extent of bleaching of individual grains in any particular sample (e.g., Reimann et al., 2012; Trauerstein et al., 2014; Blegen et al., 2015; Rhodes, 2015) and to check if significant post-depositional mixing has occurred.

In this study, individual grains of K-feldspar were dated using a two-step single-grain pIRIR procedure (Blegen et al., 2015). In this procedure (Table S1), the initial IR stimulation is performed at 200°C for 100 s using light-emitting diodes to stimulate all grains simultaneously; the pIRIR dating signals from individual grains are then measured at 275°C using a focused IR laser beam for stimulation. A preheat of 320°C for 60 s was applied to the natural, regenerative and test doses; we used a test dose $\sim 40\%$ of the size of the expected natural dose. These parameters were chosen on the basis of dose recovery tests (Galbraith et al., 1999), as described below. To eliminate grains with luminescence properties unsuitable for the pIRIR procedure, we applied

standard single-grain rejection criteria, following Blegen et al. (2015). Grains were rejected if: 1) the test dose signal measured during the natural dose cycle (T_n) was within 3σ of the background count; 2) the relative error on T_n exceeded 20%; 3) the ‘recycling ratio’ (i.e., the ratio between the sensitivity-corrected signals for a duplicate regenerative dose) was inconsistent with unity at 2σ ; 4) the extent of ‘recuperation’ (i.e., the ratio between the sensitivity-corrected zero dose and natural dose signals) was greater than 5%; 5) the natural signal was equal to or higher than the saturation level of the dose response curve (DRC); and 6) the DRC could not be reliably fitted using a saturating exponential or an exponential-plus-linear function. For all accepted grains (i.e., those remaining after applying these rejection criteria), the equivalent dose (D_e) was estimated by projecting the sensitivity-corrected natural signal of each grain on to its corresponding sensitivity-corrected DRC.

3.4. IR-RF measurement procedures

Radioluminescence (RL) or radiofluorescence (RF) was first proposed as a dating technique in the late 1990s (Trautmann et al., 1998, 1999a) and recently revisited by Frouin et al. (2017). The method is based on the fluorescence signal emitted during irradiation (Trautmann et al., 1999b), which has been suggested to not fade (Krbetschek et al., 2000; Novothny et al., 2010). If so, then the IR-RF signal would provide a validation test of the pIRIR results. In this study, we applied the original single-aliquot IR-RF procedure of Erfurt and Krbetschek (2003) and the updated procedure of Frouin et al. (2017) to multi-grain aliquots of K-feldspar from our samples.

Erfurt and Krbetschek’s procedure—hereafter referred to as

IRSAR—has four steps: 1) measure the natural IR-RF signal; 2) perform a prolonged solar bleach to reset the IR-RF signal; 3) wait for the phosphorescence signal to decay; and 4) irradiate the disc and measure the regenerated IR-RF signal (Table S1). All measurements made using the IRSAR procedure were carried out at room temperature. The modified IR-RF procedure of Frouin et al. (2017)—hereafter referred to as RF₇₀—has a similar structure as the IRSAR procedure, but samples were measured and irradiated at an elevated temperature (70 °C). To reach this temperature on the Lexsyg system, discs are required to be held at 70 °C for 2 min before making IR-RF measurements. For both procedures, we adopted the solar bleach settings of Frouin et al. (2015), which are listed in the footnote to Table S1.

The D_e value for each aliquot was obtained by sliding the natural IR-RF signal on to the regenerated IR-RF signal horizontally and vertically to minimise the residuals using the R function *analyse_IRSAR.RF()* in the R ‘Luminescence’ package (Kreutzer et al., 2017).

4. Results

4.1. Environmental dose rates

The total dose rates for all samples are summarised in Table 1, with the details given in Table S2. The total dose rates for samples from Badahlin Cave range from ~2.4 to ~3.9 Gy/ka, with much lower dose rates for the samples from Gu Myaung Cave (~1.3 Gy/ka). The dose rates are dominated by the beta dose rates (0.6–2.1 Gy/ka) and the gamma dose rates (0.3–1.3 Gy/ka), with internal dose rates of 0.4–0.7 Gy/ka (depending on grain size) and cosmic-ray dose rates of 0.02 Gy/ka or less (the latter at Badahlin Cave because of rock shielding). The total dose rates for Badahlin Cave are two- or three-fold higher than for Gu Myaung Cave, which we attribute primarily to the greater amount of low-radioactivity carbonate in the latter samples. This is reflected in the U and Th concentrations measured by field gamma spectrometry, which differ significantly between the two sites: ~5 µg/g U and ~7 µg/g Th at Badahlin Cave, compared to ~0.4 µg/g U and ~3.3 µg/g Th at Gu Myaung Cave.

4.2. Single-grain pIRIR

A representative single-grain pIRIR decay curve and the corresponding DRC for an accepted K-feldspar grain from sample BDL2-OSL 2 are shown in Fig. S1. We conducted single-grain dose recovery tests on 3–5 discs of one sample from each site (BDL2-OSL 2 and GUMY-OSL 3) to validate the pIRIR experimental conditions used here for dating. Grains were bleached for 4 h under the solar simulator to empty them of their natural doses, and then given a beta dose (as a surrogate natural dose) of 236 Gy or 30 Gy, respectively, before being measured using the pIRIR procedure in Table S1. The respective weighted-mean dose recovery ratios (i.e., the ratio of measured dose to given dose) are 1.06 ± 0.04 and 0.97 ± 0.07, with dose overdispersion (OD) values of 27 ± 3% and 31 ± 6% (Fig. S2). To examine the anomalous fading rate of the pIRIR signal, we estimated g-values for each sample using single (multi-grain) aliquots, following Auclair et al. (2003). The results obtained for storage periods of up to 2 weeks (Fig. S3) indicate that the g-values vary from ~0 to 0.3% per decade which suggests that the pIRIR signals in these samples do not fade significantly—at least not on laboratory timescales. This finding is consistent with previous studies that used an initial IR stimulation at 200 °C to effectively remove the fading component (e.g., Li and Li, 2012; Fu, 2014; Blegen et al., 2015; Yi et al., 2016).

The pIRIR signal has been shown to be relatively hard to bleach, with a residual dose of several Gy remaining even after prolonged bleaching in sunlight (e.g., Li and Li, 2011; Buylaert et al., 2012b). To estimate the size of the ‘non-bleachable’ residual dose, and correct for it, we measured the dose values for 100–300 natural grains of each sample after bleaching them for 4 h in the solar simulator. The

Table 1
D_e values, dose rates and calculated pIRIR and IR-RF ages for the samples from Badahlin Cave (BDL2) and Gu Myaung Cave (GUMY).

Sample	Depth [cm]	Grain size [µm]	Number of grains ^a	Dose rate [Gy/ka]	Equivalent dose [Gy] ^b		Overdispersion [%]	Age [ka] ^c		
					pIRIR	IR-RF (IRSAR)		IR-RF (RF ₇₀)	pIRIR	IR-RF (IRSAR)
BDL2-OSL 3	80	90–125	1200/326	2.38 ± 0.13	71 ± 2	105 ± 24	38 ± 2	29.9 ± 1.9	44.2 ± 10.4	54.3 ± 4.6
		180–212	300/47	2.60 ± 0.16	80 ± 3	–	35 ± 4	30.8 ± 2.3	–	–
BDL2-OSL 2	150	180–212	300/42	3.86 ± 0.20	203 ± 13	332 ± 13	38 ± 5	52.5 ± 4.5	85.9 ± 5.9	88.8 ± 8.4
BDL2-OSL 1	240	180–212	300/80	3.85 ± 0.23	252 ± 12	346 ± 8	39 ± 3	65.5 ± 5.1	89.9 ± 5.9	73.0 ± 5.6
GUMY-OSL 3	140	90–125	600/14	1.31 ± 0.10	33 ± 7	69 ± 4	70 ± 15	25.4 ± 5.7	53.1 ± 4.9	59.9 ± 19.4
GUMY-OSL 2	202	90–125	600/37	1.31 ± 0.09	35 ± 4	71 ± 2	61 ± 8	26.8 ± 3.6	54.5 ± 4.1	–

^a Number of grains measured/number of grains accepted for estimation of pIRIR D_e values.

^b Equivalent dose (D_e) values were estimated using the central age model. The pIRIR D_e values were corrected for a small residual dose (see text). For IR-RF D_e estimation, 2–5 single aliquots were measured for each sample.

^c Age uncertainties are expressed at 1σ and include a 2% systematic error to allow for possible bias associated with calibration of the laboratory beta sources.

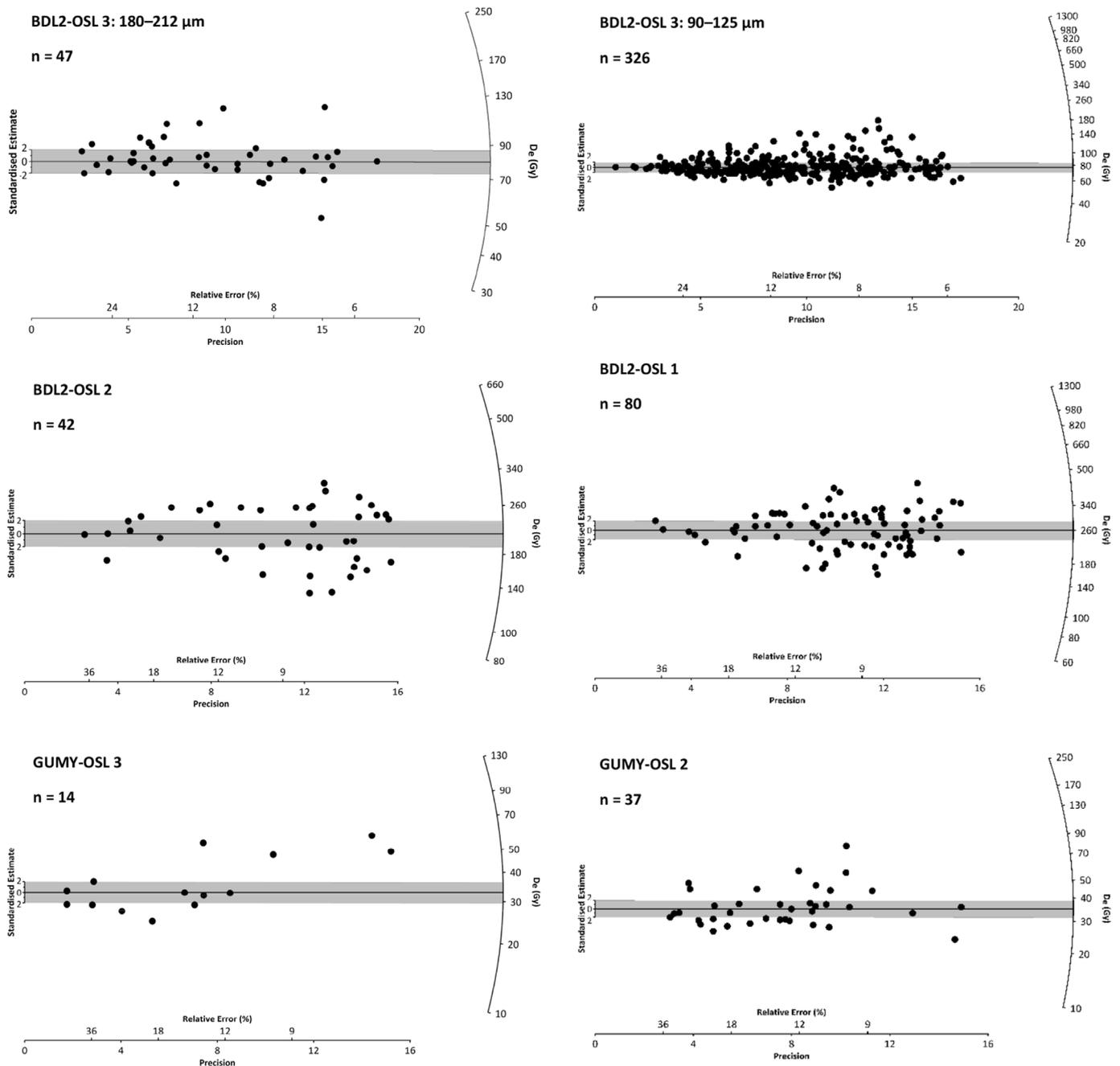


Fig. 3. Single-grain pIRIR D_e distributions for all samples. The grey bands are centred on the weighted-mean D_e values estimated using the central age model; n = number of individual D_e values.

weighted-mean residual dose for the Badahlin Cave samples is 7 ± 2 Gy, and 0.01 ± 6 Gy for samples from Gu Myaung Cave. These residual doses are small to negligible compared with the measured D_e values of these samples, but we nonetheless subtracted them to obtain the final D_e values for age determination. In summary, the factors discussed above indicate that the pIRIR procedure is well-suited to our samples.

D_e values were determined using 180–212 μm grains for two of the Badahlin Cave samples and 90–125 μm grains for both samples from Gu Myaung Cave, owing to the scarcity of larger grains; both grain-size fractions were measured for sample BDL2-OSL 3, as a methodological cross-check. The number of grains—or pseudo-single-grain measurements for the Gu Myaung Cave samples (~ 5 grains per hole)—that passed the rejection criteria corresponds to between 2% and 27% of the total number of grains measured (Table 1). Most of the grains were

rejected based on the first criterion (low intrinsic brightness).

The D_e distributions (Fig. 3) show some overdispersion in individual values, with OD values of $35 \pm 4\%$ to $39 \pm 3\%$ for the Badahlin Cave samples and $61 \pm 8\%$ and $70 \pm 15\%$ for GUMY-OSL 2 and GUMY-OSL 3, respectively. Most of the D_e overdispersion in the Badahlin Cave samples, and a large fraction of that observed in the Gu Myaung Cave samples, can be explained by intrinsic factors, as suggested by the high OD values ($\sim 27\%$ and $\sim 31\%$) obtained in the dose recovery tests (Fig. S2). The additional spread in the Gu Myaung Cave distributions could reflect incomplete bleaching during the last sediment transport event, beta microdosimetry variations, post-depositional mixing (although the stratigraphic sequence suggests a lack of disturbance) and/or the effects of non-identical bleaching and irradiation conditions in the laboratory and field (Galbraith et al., 2005). Both Gu Myaung Cave samples have small D_e datasets, however, so the OD values will be more

sensitive to outliers in the D_e distributions.

The D_e distributions are broadly symmetrical, with values spread more or less equally above and below the central estimate. There are no indications of post-depositional mixing or partial bleaching—that is, no discrete populations of D_e values are evident, nor do the D_e values cluster at low doses with a ‘tail’ extending to high doses. The sole exception is the 90–125 μm D_e distribution of BDL2-OSL 3, which has a tail of D_e values extending to > 300 Gy that could be interpreted as evidence of partial bleaching. This dataset is by far the largest of any of our samples, so such grains (which represent fewer than 10% of the total number) could also be present in the other samples, but are less conspicuous due to their low abundance. However, while the 180–212 μm D_e distribution of BDL2-OSL 3 contains a few grains with D_e values > 100 Gy, it does not exhibit the same pattern of spread as observed for the 90–125 μm fraction and the weighted-mean ages for these two grain-size fractions are statistically indistinguishable (Table 1). This suggests that, even if some of the grains had been partially bleached, the net effect is not significant.

Based on these observations and interpretations, we calculated the D_e values for our samples using the central age model (CAM), which gives an estimate of the weighted geometric mean (Galbraith et al., 1999; Galbraith and Roberts, 2012). The final ages for the three samples from Badahlin Cave are in stratigraphic order and range from 30 ± 2 ka to 66 ± 5 ka, while both samples from Gu Myaung Cave are ~ 26 ka (Table 1).

4.3. Multi-grain IR-RF

Before determining D_e values using the IRSAR IR-RF procedure, we conducted several tests to determine the most appropriate measurement conditions. Previous studies have suggested that prolonged solar bleaching (step 2 in Table S1) is required to reset the IR-RF signal (e.g., 3 h: Frouin et al., 2015, 2017). For our samples, however, we found that solar simulator bleaches of > 1 h did not cause a further increase in the IR-RF signal—that is, the IR-RF source traps were fully emptied by a solar simulator bleach of 1 h (Fig. 4, left panel)—so we used a solar bleach time of 1 h in this study. We also performed a pause duration test to determine the minimum delay time needed to eliminate phosphorescence before measuring the regenerated IR-RF signal (step 3 in Table S1). We found that, similar to Buylaert et al. (2012a), the phosphorescence signals of our samples are very weak compared to the IR-RF signal (Fig. S4), and we could not distinguish between the regenerated IR-RF signals measured after pauses of 15–60 min (Fig. 4, right panel). We concluded, therefore, that the effects of phosphorescence on the IR-RF signals of our samples were negligible after a pause time of 15 min, which we adopted to save machine time. We caution, however, that these settings are sample- and instrument-specific, and may not be appropriate for samples from other sites.

To validate our revised procedures, we conducted dose recovery

tests on natural aliquots of samples BDL2-OSL 3 (Fig. S5) and GUMY-OSL 3. We first bleached them for 4 h in the solar simulator and then gave them beta doses similar in size to the expected D_e values. The weighted-mean dose recovery ratios are 1.06 ± 0.03 and 1.31 ± 0.13 , respectively. These results suggest that the given dose can be recovered with sufficient accuracy using the experimental conditions in our IRSAR procedure (Table S1) for samples from Badahlin Cave, but that the D_e values for the Gu Myaung Cave samples may be slightly overestimated.

We also tested the RF₇₀ procedure (Frouin et al., 2017) on our samples. Preliminary tests showed that IR-RF measurements made at 70 °C did not improve the shapes of the decay curves for our samples, in contrast to the findings of Huot et al. (2015) and Frouin et al. (2017). Nevertheless, the RF₇₀ procedure yields slightly different results to those obtained using the IRSAR procedure: the RF₇₀ signal intensity is higher and the calculated D_e values differ by 11–65 Gy, with the RF₇₀ values being larger in some instances and smaller in others (Table 1); two examples are shown in Fig. 5. The cause(s) of these differences are under investigation, including tests of the suitability of the RF₇₀ measurement conditions for our samples.

We estimated the IR-RF ages from the weighted-mean (CAM) D_e values for 2–5 aliquots of each sample, without making any allowance for a residual dose (Table 1). The calculated ages for the Badahlin Cave samples range from 44 ± 10 ka to 90 ± 6 ka (IRSAR) and from 54 ± 5 ka to 89 ± 8 ka (RF₇₀). The Gu Myaung Cave samples yielded IRSAR ages of ~ 54 ka and, for GUMY-OSL 3, a RF₇₀ age of 60 ± 19 ka. These ages are much older than the pIRIR and ¹⁴C chronologies, which is possibly due to insufficient bleaching of the IR-RF signal. Subtraction of an appropriate residual dose would likely reduce these age discrepancies considerably, and we are in the process of assessing its likely size for these samples.

5. Discussion

In this preliminary study of K-feldspar grains from two archaeological sites in central Myanmar, we have shown that the single-grain pIRIR procedure is suitable for dating, with acceptable results from dose recovery tests and no evidence for fading of the pIRIR signal over laboratory timescales. There is potential, therefore, to extend the applicability of pIRIR dating to other Palaeolithic sites in the region.

A comparison with independent (¹⁴C) age control at Gu Myaung Cave has revealed, however, that both pIRIR ages (~ 26 ka) significantly overestimate the ¹⁴C ages (~ 10 and ~ 15 ka cal BP; Table S3) obtained for charcoal fragments collected from stratigraphic layers between and below these two sediment samples (Fig. 2b). Further evidence is required to validate which, if either, of these two chronologies is the more reliable. Our residual dose test of K-feldspar grains from Badahlin and Gu Myaung Caves suggests that the pIRIR signal can be (nearly) fully reset by solar bleaching in the laboratory, but this does

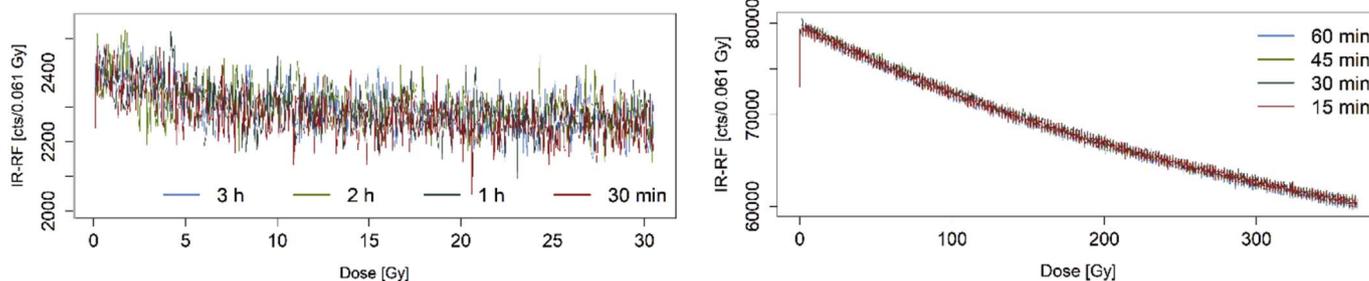


Fig. 4. Test of solar bleaching time (left) and pause time (right) for IR-RF measurements of sample BDL2-OSL 3 using the IRSAR procedure (Table S1). In the first experiment (left panel), a used single aliquot was given a regenerative dose of ~ 305 Gy, bleached for duration t_b using the solar simulator in the Lexsyg reader, and paused for 1 h before measuring the IR-RF signal. This cycle was repeated four times with t_b varied from 30 min to 3 h. The left panel shows the early portion (0–30 Gy) of the IR-RF signal. In the second experiment (right panel), the same disc was first bleached for 1 h using the solar simulator and paused for duration t_p before measuring the IR-RF signal. This cycle was repeated four times with t_p varied from 15 min to 60 min. Additional data are presented in Fig. S4.

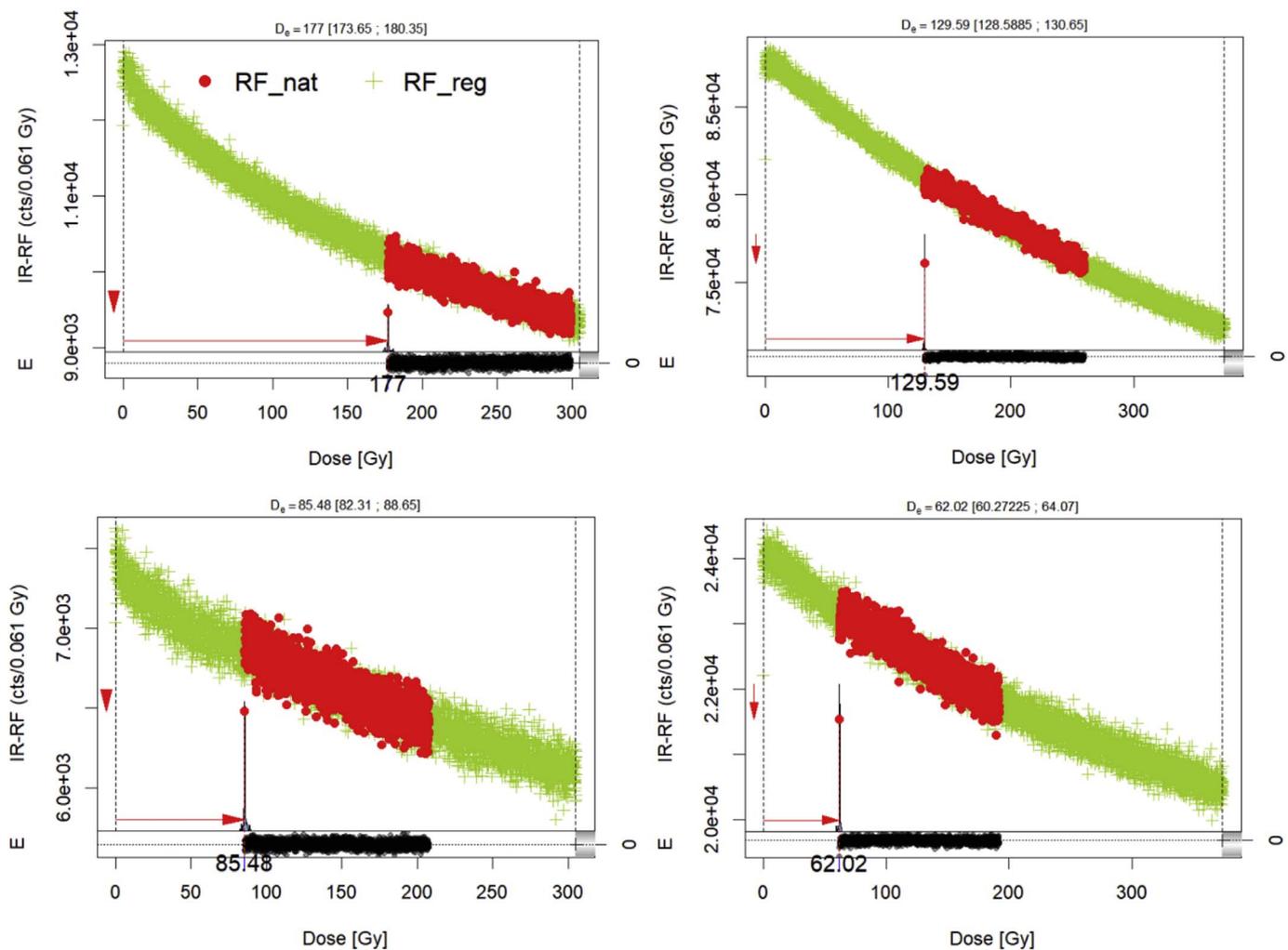


Fig. 5. Typical IRSAR and RF_{70} data for BDL2-OSL 3 from Badahlin Cave (top panels) and GUMY-OSL 3 from Gu Myaung Cave (bottom panels). Each panel shows the data for a single disc.

not imply that the sediments were, in fact, exposed to sufficient sunlight when deposited at these sites. On the other hand, the pIRIR D_e distribution patterns provide no support for heterogeneous bleaching (Fig. 3), so the causes of the age discrepancy at Gu Myaung Cave remain elusive and are currently under investigation.

Duller (1994) proposed two types of poorly bleached sediments in nature: 1) sediments containing a mixture of grains that have experienced differing degrees of bleaching—some grains being completely bleached at deposition and others receiving little or no light exposure; and 2) sediments in which all grains have been equally poorly bleached at deposition. If the discrepant pIRIR and ^{14}C ages at Gu Myaung Cave are due to insufficient bleaching of the pIRIR signal, then these samples may represent the second type of sediment, which could yield symmetric D_e distributions. We note, however, that this type of sediment is expected to be relatively rare in natural environments (Wallinga, 2002).

In the case of Gu Myaung Cave, we consider it unlikely that all of the grains were poorly bleached, since the sediment samples were collected from an excavation located close to the cave entrance. To obtain further insights into the bleaching of the sediments and whether the residual doses measured in the laboratory are representative of the sediments when deposited in the cave, we will conduct residual dose tests and examine the D_e distributions of recently deposited samples from the upper part of the stratigraphy. The reliability of the ^{14}C ages also warrants further investigation, and comparisons with chronologies constructed from complementary techniques (e.g., U-series dating of suitable flowstones) would be instructive.

The IR-RF ages obtained in this study consistently overestimate their pIRIR counterparts by 8–36 ka (Table 1), which we attribute to the different bleachabilities of these two signals. The use of multi-grain aliquots for IR-RF dating will exacerbate problems of D_e overestimation if partial bleaching is an issue. We note that the pIRIR and IRSAR IR-RF ages systematically increase with depth at Badahlin Cave, while the two pIRIR ages for Gu Myaung Cave are statistically indistinguishable, as are the two IR-RF ages at this site. This pattern suggests a common underlying cause for the age offset, which we are currently investigating. Other future lines of enquiry include further testing of the RF_{70} procedure of Frouin et al. (2017), including dose recovery tests and other variations to readout temperature, and processing of younger samples from both sites to obtain an improved estimate of the residual dose at deposition.

If our pIRIR ages for Badahlin Cave are accurate, then human occupation started at least ~30 ka ago (Fig. 2a). The underlying, culturally sterile deposits dated to ~53 ka and ~66 ka might be interpreted as evidence that people had not arrived in this region by 53 ka, but we consider such inferences as premature given the many chambers of Badahlin Cave that have yet to be excavated. Sediments appear to have been accumulating in parts of the large cave complex for more than 50 millennia and further excavations may reveal earlier traces of human habitation. Based on our current pIRIR ages, occupation of Gu Myaung Cave appears to have commenced ~25 ka ago, whereas the ^{14}C chronology suggests human arrival during the early Holocene (Fig. 2b). In addition to resolving these discordant chronologies, future work may

yet reveal a longer record of human occupation of this site, which cannot be ruled out on the basis of the limited excavations conducted thus far.

6. Conclusions

We have applied single-grain pIRIR and multi-grain IR-RF dating procedures to K-feldspar extracts from archaeological deposits at Badahlin and Gu Myaung Caves in central Myanmar. Luminescence investigations of the pIRIR and IR-RF signals, and the corresponding D_e distributions, indicates that the pIRIR procedure, at least, may be suitable for sediment dating. The IR-RF ages overestimate the pIRIR D_e values and ages by a considerable margin, which may be related to the poorer bleachability and higher residual doses associated with the IR-RF signal. Even the pIRIR ages are substantially older than the ^{14}C ages at Gu Myaung Cave, despite the lack of support for heterogeneous bleaching in the single-grain D_e distributions. Future investigations could involve pIRIR and IR-RF measurements of recently deposited sediments to determine the residual doses at deposition in the natural environment, and IR-RF measurements of individual K-feldspar grains (e.g., Trautmann et al., 2000) to avoid the shortcomings of using multi-grain aliquots to date partially bleached sediments. Our preliminary pIRIR chronologies indicate that human occupation of Badahlin and Gu Myaung Caves started at least ~30 ka and ~25 ka ago, respectively, thereby adding to the sparse chronological record supporting Palaeolithic settlement of the region during the Late Pleistocene (Aung et al., 2015).

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Appendix A. Supplementary data

Supplementary data related to this article can be found at <http://dx.doi.org/10.1016/j.quageo.2018.01.001>.

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