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Luminescence dating of archaeometallurgical slag from Buriram Province, northeastern Thailand

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ABSTRACT: In this study, the possibility of directly dating archaeometallurgical slags is assessed. The enormous slag heaps distributed in the Ban Kruat district, Buriram Province, Thailand, have been interpreted as one of the most prominent archaeometallurgical sites in Mainland Southeast Asia. Therefore, five slag samples were collected from the topmost level of two heaps. The X-ray diffraction measurement of each slag revealed the existence of quartz minerals, which is conceptually useful to luminescence dating. Based on optically stimulated luminescence (OSL) measurements, two of the five samples showed a weak luminescence signal, which was not suitable for OSL dating, and may reflect the lack of quartz minerals. However, the other three quartz-rich slag samples clearly expressed an OSL signal. Therefore, 40 or 96 aliquots of a single aliquot regenerative measurement were employed to date them.

With the combination of the activated dose rate obtained from environmental radioactive elements (U, Th, and K), two timespans of iron-smelting activity were defined, as approximately 140 y ago and 470–710 y ago. Compared with the radiocarbon dates of the adjacent slag heap in Buriram (560 ± 280 y BP), the 140-y-old slag heap dated in this study is younger and represents the latest (most recently) datable iron-smelting industry in the Angkor highland (Thailand). Meanwhile, the radiocarbon dates (140 ± 20 y BP) of the slag heap in the Angkor lowland (Cambodia) conformed to the date obtained in this study. Therefore, it is concluded that OSL dating is effective for direct dating of the slag-bearing quartz.

KEYWORDS: luminescence dating, archaeometallurgical slag, Ban Kruat archaeological site, iron-smelting activity

INTRODUCTION

In mainland Southeast Asian contexts, iron is known as one of the versatile and dynamic materials used in premodern times, from its initial appearance in the late 6th or early 5th century BC. Metal and its production activities have long been discussed for their role in the socio-economic development of Southeast Asian societies, in particular in Thailand. Iron artifacts in the form of tools and weapons recovered from late prehistoric and historic sites and numerous slag deposits documented across the present-day Thai landscape have shown their close association with the political and socio-economic sectors. This research draws attention to Ban Kruat, Buriram Province, located on the northern part of the Dangrek Mountain Range, which, over a period of three decades of archaeological research in this area, has identified the vestiges of human occupation since early centuries AD to the 12th/13th century. The earlier occupation began some time during the last centuries BC, as evidenced by the settlements/cemeteries at Ban Bueng Noi and Ban Sai Tho 7 South, in which the latter was radiometrically dated to the 4th century AD (cal. AD 253–406) [1].

The main occupation in the area of our interest, however, occurred during the Angkorian Khmer period (9th–14th century AD). In addition to typical Angkorian Khmer structures, in particular temples, Khmer ceramic kiln sites have attracted interest from archaeologists. These kiln sites in Ban Kruat together with Ban Baranae (also in Buriram Province) form the greatest concentrations of Khmer ceramic production locales in Thailand. In addition to the temples and ceramic production, the location of Ban Kruat is closely associated with the Angkorian Khmer northwestern route that connected Angkor with Phimai. This is illustrated by the presence of Dharmasalas or resthouses found in the area (Fig. 1A). These Angkorian Khmer remains give the impression of that the area played an important socio-economic role, especially for local craft production, during this period.

The other evidence that has received attention in only the last few decades is the remains of primary iron production, in the form of slag mounds of various sizes and slag deposits. These slag sites were first identified in the late 1980s and early 1990s by both the Fine Arts Department (FAD) [2, 3] and a Thai-Japanese archaeology team [4, 5], but these mounds had never been extensively documented until recently. In 2007, the Thai-Cambodia joint archaeology team explored Ban Kruat as part of the National Research Council of Thailand (NRCT)-funded Living Angkor Road Project (2007–2010). As a result of extensive survey, at least 50 slag deposits and mounds have been documented within an area of 140 km^2 [1, 6]. Given the number of the identified remains and their association with the Royal route connecting Angkor and Phimai, this was initially interpreted as an Angkorian Khmer iron production locale. Two intact slag mounds, Ban Khao Din Tai and Ban Sai Tho 7 south, were subjected for further excavation and analyses, including chronological determination and archaeometallurgical examination of the production waste [6, 7].

Detailed archaeometallurgical examinations of these finds revealed that both slag mounds were formed by iron smelting activity, based on the direct smelting method using relatively iron-poor but locally ubiquitous lateritic nodules as the ore, but the use of flux was not identified [1, 8–10]. The radiometric dating programs (conventional and accelerator mass spectroscopy; AMS) at both excavated sites of charcoal samples, including four in-slag charcoal samples, in conjunction with the typology of the pottery found, at the sites indicated that the activity was likely to have been carried out during the later phase of the Iron Age (5th century BC–5th century AD) [1, 6], and not during the Angkorian Khmer period as previously thought.

Despite this determination, surface surveys of some slag deposits and mounds recorded pottery remains from later periods, suggesting the possibility that iron-smelting may have been continued into historical periods, possibly during the Angkorian Khmer period together with ceramic production. No post-Angkorian material culture has currently been identified in Ban Kruat suggesting that either occupation was not continued into the post-Angkorian period or evidence for post-Angkorian activities has yet been discovered.

While these remains demonstrate the use of iron over two millennia, an understanding of the role of iron production in ancient societies has been hindered by a lack of systematic dating of the slag remains and so only a few sites have been securely dated. Those dated sites have heavily relied on the relative dating of datable surface finds, in particular ceramic fragments. However, this is not always possible, including at Ban Kruat where ceramic fragments, etc., have rarely been recovered at smelting sites. Establishing the chronology for the other slag deposits and mounds is, therefore, crucial to allow a better understanding of the origins, formation, and development of the iron production activity and how this production has been integrated into the socio-economic development of the Ban Kruat area.

Dating iron production sites

There are three main methods of dating iron production sites: archaeomagnetism, luminescence, and radiocarbon dating. These are considered indirect procedures since they rely on associated datable specimens with secure archaeological contexts, such as *in situ* furnace structures, charcoal, and in-slag charcoal. Of these techniques, luminescence is arguably the only technique that has a great potential for directly dating slag, a by-product from the smelting and smithing processes, that is always available at production sites.

Both luminescence (trapped-charge) dating, i.e. thermoluminescence (TL) and optically stimulated luminescence (OSL) dating, have been explored previously to assess their reliability in calendrical dating. This includes the use of TL to date the ancient slag-bearing quartz collected from Aegean Island, Greece [11]. Although there is a limitation of the annual dose approach, the obtained dates, however, conformed well to the other dates obtained from different materials, including the TL dating of porcelain [12]. Haustein et al [13] successfully dated slag specimens from the archaeometallurgical sites in both Europe and the Mediterranean using TL dating. Moreover, Gautier [14] dated quartz extracted from slag samples from Britain and Greece using OSL dating, and both the OSL and radiocarbon dates were in broad agreement with each other the radiocarbon dates of the charcoal. Therefore, both TL and OSL dating with slag-bearing quartz are one of the possible and powerful techniques to directly define the chronological information of iron-smelting sites.

The reliable chronological technique based on thermoluminescence properties was firstly introduced in 1998 [15]. Then, the recently developed technique using light in stimulation, called OSL combining the single aliquot regeneration (SAR) protocol was carried out in 2000 by [16]. Although both TL and OSL are effective and reliable methods for burnt materials, OSL dating more outweighs TL by following three main reasons. Firstly, a key problem of TL is an inter-aliquot error due to the measurement of multiple aliquots for construction of the growth curve to find equivalent dose (a quantity of given dose after heating to reset zero signal). The SAR allows the user to measure all signals from one aliquot and is a reasonable protocol to avoid TL normalization. The second is a required less amount of quartz in OSL dating combination of SAR, and this is extremely suitable for limited quartz-content samples [14, 17]. Finally, SAR in OSL dating provides more accuracy of equivalent dose statistic evaluation due to more convenient of a vast number of remeasurements.

In Thai archaeological contexts, metal production sites have heavily been relied on radiometric dating technique of archaeological charcoal specimens, both excavated charcoal and in-slag charcoal specimens. For luminescence dating, it has been less employed to date metal production sites. In case of Ban Khao Din Tai, TL dates were rejected in favor of more conforming radiocarbon, AMS, and relative dating results. Furthermore, it should also be noted that dating slag using luminescence techniques has never been attempted before in Thai archaeology.

In this paper, in order to better establish the calendrical dates for iron smelting activity in Ban Kruat, OSL measurements of slag samples collected from Ban Sai Tho 7 were performed with the goal of assessing the possibility and reliability of dating slag-bearing quartz by the OSL technique.

MATERIALS AND METHODS

Study area and sample collections

According to a previous archaeological investigation in 2010–2011, Ban Sai Tho 7 was defined by 10–11 slag mounds of various sizes surrounding a flat central area, creating a crater-like shape [18], as shown in Fig. 1B. The excavation at the central area produced evidence for habitational and burial activities (253–406 cal. AD), from which beads, potsherds, and fauna remains were recovered. These early phases were then replaced by metallurgical activity, represented by a hard floor of smithing slag, which was AMS dated to 382–539 cal. AD [1]. Unfortunately, no radiometric dating has been attempted for the slag mounds. In order to define the age of these slag heaps by the OSL dating, five slag samples were collected from the surface of slag heap 1 and 2, i.e., BK 1 and BK 2, respectively (Fig. 1B,C). The samples were macroscopically different in terms of their color, porosity, density, weight, and other metallurgical features, such as flowing texture (Fig. 2). For example, BK 1-1, BK 2-1, and BK 2-2 had a high porosity, low density, and different colors of grey, black, and reddish-brown, respectively. In contrast, BK 2-3 and BK 2-4 were reddish-brown dense slags that demonstrated a flowing structure. These differences are likely caused by differences in the raw material, smelting process, or even different smelting episodes.

X-ray diffraction (XRD) measurement

Theoretically, luminescence dating can date various types of minerals, such as calcite, feldspar, quartz, etc. However, in practice, quartz is the most common mineral that is utilized [19–21]. In this study, all the slag samples were checked for their mineral composition using XRD analysis before dating.

OSL dating

Based on Gautier [14] , the elapsed time since the latest iron smelting and the present is determined from the luminescence dating using Eq. (1):

$$\text{Luminescence date} = \frac{\text{Equivalent dose (ED)}}{\text{Annual dose (AD)}} \qquad (1)$$

where the equivalent dose (ED) in Gy is measured from the luminescence emitted during heating (TL) or optical stimulation (OSL) of the sample. Meanwhile, the AD in Gy/y was evaluated from the concentration of three abundant natural radioisotopes: uranium (U), thorium (Th), and potassium (K), in the environment surrounding the sample. To achieve the OSL dating, the (i) radiometric and (ii) OSL measurements are required.

Radioactive measurement

According to Haustein et al [13] and Puttagan et al [22], the important part in luminescence dating is the AD analyzed from the concentration of the existing radioactive elements. Theoretically, according to the penetrations of the alpha, beta, and gamma radioactivity, the radioactive elements within a 30-cm surrounding should be recognized. Since the heaps contained only dense slag, the concentrations of the radioactive elements can be analyzed directly from the slag specimen.

To archive, the slag was crushed, weighed, and dried out before reweighing to determine the water content. Next, 300 g of dried slag powder, which had a diameter of < 0.85 mm, was sieved, held in a closed plastic box for 1 month at room temperature, and then the U, Th, and K contents were measured using gamma-ray spectrometry as previously reported [23]. Finally, with the complement of U, Th and K, and water contents, the AD was calculated according to Bell [24]. The obtained AD results are expressed in Table 2.

OSL measurement

In this study, the OSL measurement and dating were based on polymineral samples that were purposely selected for dating the slag [11]. Firstly, all samples were eliminated from around the top 5 mm of the outer layer using a steel file. Thereafter, in each sample, the remaining slag was harvested and crushed by a rubber hammer in order to separate individual grains of slag. Those with a grain size of < 250 μm were sieved and collected for the OSL measurement and ED evaluation. SAR was employed using an automated Risø reader (TL/OSL-DA-15) [25]. In order to avoid leakage of the luminescence signal, all stages of the preparation were performed under a subdued red-light environment.

RESULTS AND DISCUSSION

XRD analysis

As shown in Fig. 3 and Table 1, all the slag samples were composed of similar minerals, including (i) hercynite, (ii) cristobalite, (iii) fayalite, and (iv) quartz, while (v) hematite was found in samples BK 2-1 and BK 2-2. According to the presence of quartz minerals, all five samples collected here were assumed to be suitable for measuring the luminescence signal, and so capable to date using OSL, which is the main aim of this study.

From the classification of [26], the five slag samples can be categorized into two groups according to their mineral composition and content. The first group, comprised of samples BK 1-1, BK 2-3, BK 2-2,

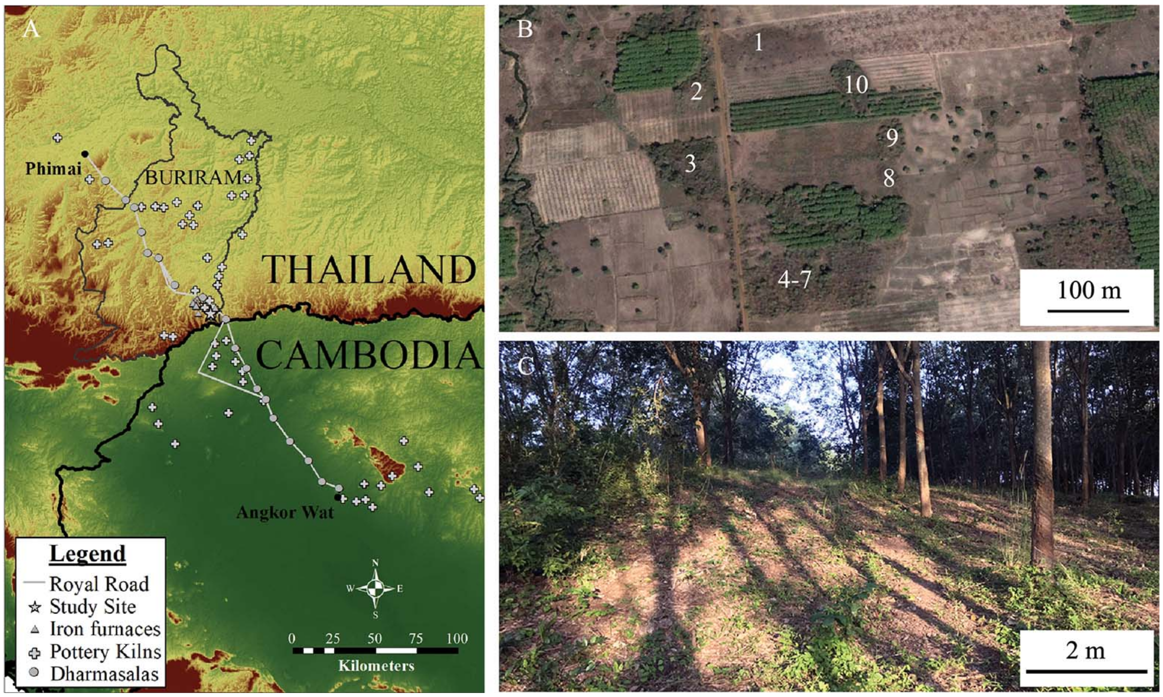

Fig. 1 (A) Map of the Thailand-Cambodia border showing the locations of Dharmasalas (circle), pottery kilns (cross), iron furnaces (triangle), and Ban Sai Tho 7 (star). (B) Satellite image showing the circular distribution of 10–11 slag heaps in Ban Sai Tho 7 [18]. (C) Location of the slag specimens that were collected for the chronological investigation in this study from slag heaps 1 and 2.

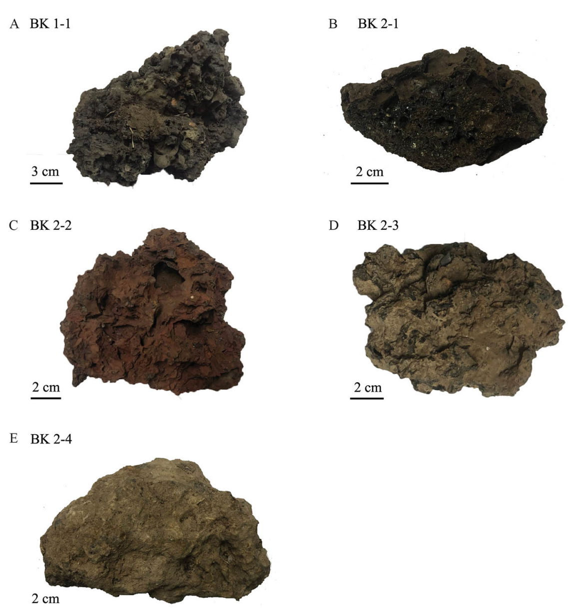

Fig. 2 Macroscopic features of five slag samples collected at the Ban Sai Tho 7, Ban Kruat district, Buriram Province. Samples BK 1 and BK 2 were collected from slag heaps 1 and 2, respectively (as shown in Fig. 1B).

Table 1 Mineral composition of the five slag samples, according to the XRD analysis.

Sample	Mineral content (wt.%)				
	Fayalite	Quartz	Cristobalite	Hercynite	Hematite
BK 1-1	16.63	27.56	48.76	16.63	–
BK 2-1	49.56	14.32	11.41	18.36	6.35
BK 2-2	6.30	27.38	31.69	8.84	25.79
BK 2-3	15.55	78.91	3.00	2.54	–
BK 2-4	3.99	83.76	–	2.54	–

Fayalite: Fe_2SiO_4, quartz and cristobalite: SiO_2, hercynite $FeAl_2O_4$, hematite: Fe_2O_3.

and BK 2-4, were defined as silica-rich slags and were comprised of more than 50% silica minerals (quartz and cristobalite). The second, based on the presence of hematite, fayalite, and hercynite, contained sample BK 2-1 and was defined as an iron-alumina slag.

In addition, according to Tsaimou et al [27], the presence of fayalite implies the commonly used direct smelting method operated at more than 1100 °C [8]. Meanwhile, some archaeometallurgical sites were reported with a composition of kirschsteinite instead of fayalite [8] which implies another modified smelting technique that includes lime fluxing with $CaCO_3$ and/or CaO [8, 26–28]. Since all five slags col-

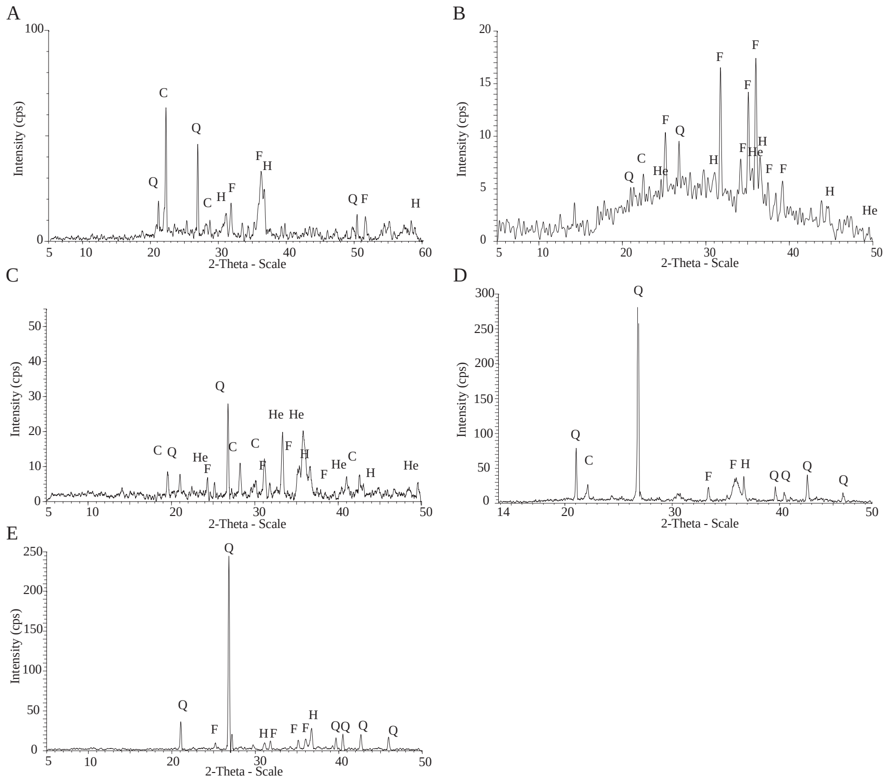

Fig. 3 Representative XRD results showing the mineral compositions of the (A) BK 1-1, (B) BK 2-1, (C) BK 2-2, (D) BK 2-3, and (E) BK 2-4 slag samples.

Table 2 The OSL dating results of three slag samples from the Ban Kruat district, Buriram Province. CAM and MAM represent the central and minimum age models, respectively, while W depicts the content of moisture used in the AD estimation.

Sample	U (ppm)	Th (ppm)	K (%)	W (%)	AD (Gy/ka)	ED (Gy)	Age (y)	Age (model)
BK 1-1	16.32	24.31	0.56	0.414	12.90 ± 0.15	1.9 ± 0.12	140 ± 10	CAM
BK 2-3	14.19	20.23	0.93	0.937	11.46 ± 0.03	5.4 ± 0.08	470 ± 7	MAM
BK 2-4	19.08	23.94	1.09	1.242	14.65 ± 0.03	10.5 ± 0.10	710 ± 9	MAM

lected in this study were composed of fayalite without kirschsteinite, the smelting method utilized at Ban Sai Tho 7 was direct smelting without any supplementary lime flux. This conforms to the smelting production method proposed at the nearby Ban Khao Din Tai slag heap [1, 8].

Dating result

Based on the OSL signal as shown in Fig. 4, although the XRD analysis indicated the presence of quartz mineral, the OSL signals of BK 2-1 and BK 2-2 (Fig. 4B,C) were quite low and fluctuated. In addition, even though an artificial additive dose for both samples (1.41 Gy) was demonstrated, the OSL signal was still lacking. There are several cases of low OSL sensitivity from various causes [29]. Puttagan [22] points out that the burnt brick that absented sensitivity may come from an insufficient heating part of a kiln. Contrary, slag is a by-product of the smelting process that melting raw material in a high-temperature condition; therefore, slag had been in a furnace with sufficient temperature. As a result, we summated that both the BK 2-1 and BK 2-2 had no potential for dating using the OSL approach. In contrast, samples BK 1-1, BK 2-3, and BK 2-4 clearly showed OSL decay curves. With respect to the quartz mineral composition, it was notable that BK 2-1 and BK 2-2 were composed of less than 60% quartz. In contrast, the three datable specimens (i.e., BK 1-1, BK 2-3, and BK 2-4) that illustrated an OSL signal were composed of at least 75% quartz. Thus, besides the existence of quartz, the content or quantity of quartz is important to be concerned as well.

According to the SAR procedure, 40 or 96 aliquots were analyzed systematically. As shown in Fig. 5, the

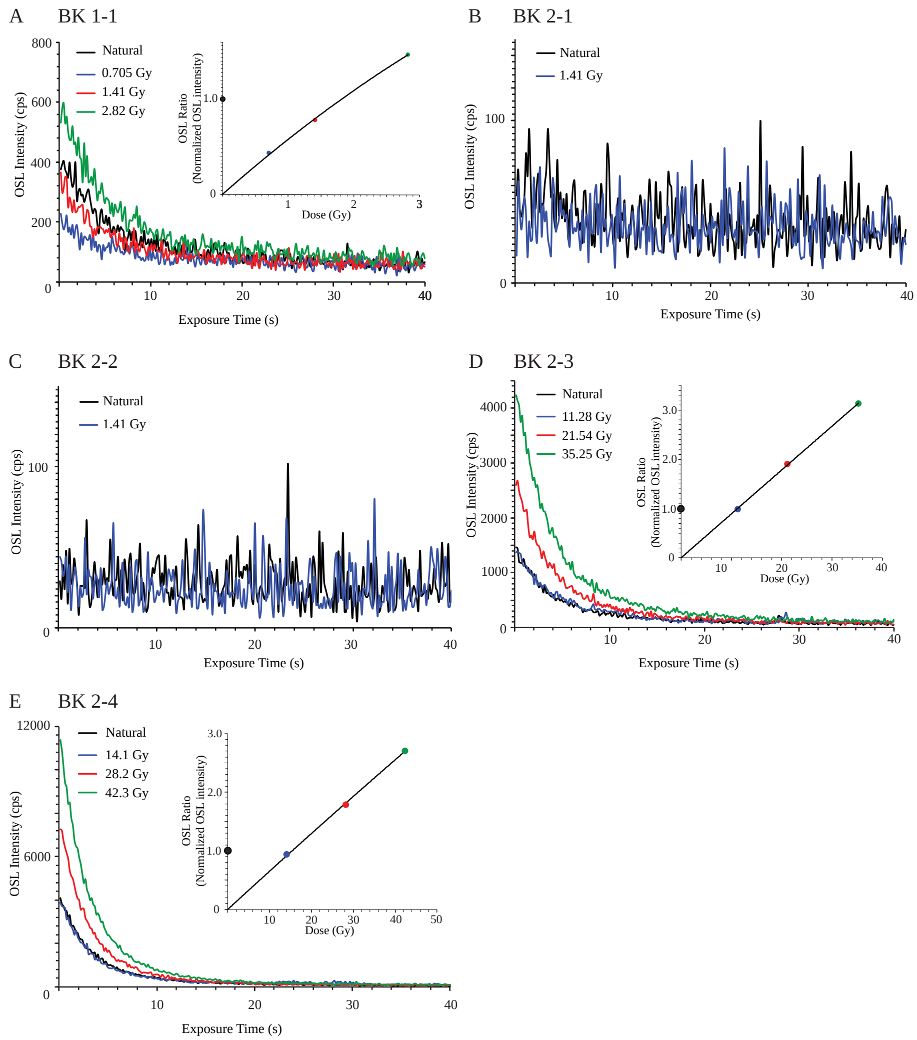

Fig. 4 The OSL decay curves and growth curves (inset) of the five slag samples analyzed. The natural (black line); the additive beta doses (blue, red, and green lines).

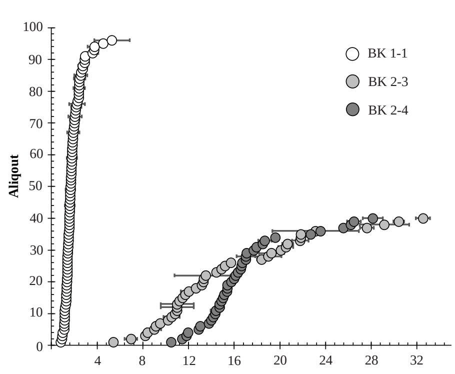

Fig. 5 Typical ED distributions of the three slag samples.

obtained ED of each sample expressed a different degree of variation. The BK 1-1 illustrated a low variation in the ED when analyzed from 96 aliquots. The net ED of BK 1-1, therefore, was defined at 1.9 ± 0.12 Gy according to the central age model [30]. Meanwhile, the BK 2-3 and BK 2-4 samples had a comparatively high variation in the ED from 40 aliquots with estimated ED values for BK 2-3 and BK 2-4 of 5.4 ± 0.08 Gy and 10.5 ± 0.10 Gy, respectively, based on the minimum age model [31]. With the complementary data of the AD, the dates of the three datable slag samples are expressed in Table 2.

As mentioned earlier, all five slags dated in this study were collected from the topmost surface layer of the heaps. The OSL dates, therefore, potentially represent the latest smelting activity at both individual heaps; although, it is also needed to take into account

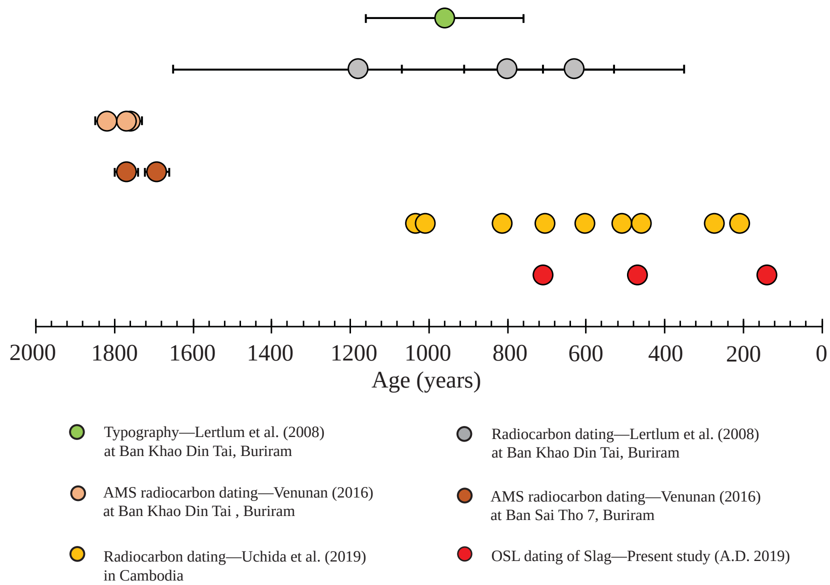

Fig. 6 Distribution of the preliminary ages for the slag samples and reference ages.

modern disturbance of each site. However, at the studied slag heaps, there was no sign of modern heavy disturbance on the surface. The results of slag dating from two slag heaps suggested three different periods of iron-smelting production: early 14th (BK 2-4), mid 16th (BK 2-3) and late 19th centuries (BK 1-1) as shown in Table 2. This chronological difference is also observed in the studies by Hendrickson et al [32] and Uchida et al [33] using AMS dating of in-slag-charcoal which illustrated slag heaps at Preah Khan of Kompong Svay lasted in later periods of activities. The most recent date of slag from heap no. 1 was the youngest date in the lower northeastern Thailand; therefore, comparing the three dates for the two heaps, it can interpret that the iron-smelting activity at heap 2 (Fig. 1B) was terminated around 470 ± 7 y ago, whereas, at heap 1, it continued to 140 ± 10 y ago.

Comparison between the previously reported ages of the slag heaps (Fig. 6) and the OSL dates derived in this study revealed that the smelting activity at heap 2 was in the same time span as at the nearby Ban Khao Din Tai (1100 ± 470 to 560 ± 280 y BP) [6]. Whereas, for heap 1, the OSL date of 140 ± 10 y ago makes it the youngest iron smelting industry in Ban Kruat, if not northeast Thailand. Indeed, this date for heap 1 conformed well with the radiocarbon dates (140 ± 20 y BP) of a slag site in the Angkorian lowland (Cambodia) [33], suggesting that iron smelting in Ban Kruat might have taken place during the late 19th century; although, it is unsettled if the activity was continued from the Angkorian period.

The dates of heap no. 2 from the two slags illustrate that the older slag was in the 14th century during a decline of the Khmer Empire (1431 CE) and the younger one (16th century) was during the early Ayutthaya period [34]. According to the hypothesis that the iron smelting sites in Thailand had been continuity conducted by direct smelting technique since the late prehistoric [1, 35] until Late Ayutthaya [6, 34] before the transition to indirect smelting technique derived from China in late the 17th century AD [36], these date results in this study are in good agreement and shed new light of the post Angkorian activities in this site and Ban Kruat, where it was previously unknown.

More significantly, combining the XRD result presenting contemporary of the direct iron-smelting technique and the youngest age of slag by OSL dating confirms the assumption that iron-smelting in this site had possibly produced in the initial site extend to the post-Angkorian period.

CONCLUSION

The main aim of this study is to provide new knowledge of the calendrical dates for iron smelting activity in Ban Kruat and assessing the possibility and reliability of dating slag-bearing quartz by the OSL technique. This study shows the most recent datable iron-smelting activity in the Angkor highland. Therefore, it can be concluded that the iron-smelting industry at Ban Sai Tho 7 had a long dynamic history that at least between the late Iron age (the 3rd–the 6th centuries) and the post-Angkorian (the 17th century). Moreover, iron-smelting can be produced in the initial location in several centuries. In addition, OSL dating is seen to be an effective and meaningful method for the direct dating of slag-bearing quartz using polyminerals.

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