# RADIOCARBON DATING AND ZOOMS SPECIES IDENTIFICATION OF FRAGMENTARY BONE AT THE LATE UPPER PALAEOLITHIC AND MESOLITHIC SITE OF KING ARTHUR'S CAVE

## by

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## ABSTRACT

King Arthur's Cave (Wye Valley) contains a late Pleistocene and Holocene sedimentary sequence, with evidence of Late Upper Palaeolithic, Mesolithic and later occupations. It currently provides the earliest dates for a human presence in the British Isles after the Last Glacial Maximum. Here we revisit the faunal material from the University of Bristol Speleological Society 1920s and 1950s excavations to further clarify the chronology of the stratigraphic sequence on the platform outside the cave mouth. The results of six new ultrafiltered radiocarbon dates confirm that fauna date to before the Last Glacial Maximum and to the Late Glacial, and that some post depositional stratigraphic mixing has occurred. We undertook peptide mass fingerprinting (ZooMS) of fragmentary bones from the platform archaeological levels to provide further insights into the fauna during the late Pleistocene and early Holocene. The ZooMS species identification indicate the fragmentary bone assemblage mirrors the species present in the morphologically identifiable bone assemblage. Although dominated by red deer, the presence of "mammoth steppe" fauna such as woolly rhino and spotted hyaena, alongside temperate species and domesticated animals (e.g. sheep) further confirm post depositional stratigraphic mixing. Amongst the fragments identified is a human bone which, based on its provenance, could be Late Glacial or early Holocene in age and relate to the Late Upper Palaeolithic or Mesolithic activity at the site. The specimen is currently being radiocarbon dated.

#### INTRODUCTION

King Arthur's Cave is located on Great Doward Hill, 1 km from Symonds Yat in the Wye Valley, Herefordshire (Figure 1). The site contained an important late Pleistocene and Holocene sedimentary sequence, with evidence of Palaeolithic, Mesolithic and later occupations (ApSimon et al. 1992). Evidence for human activity at the site during the Late Upper Palaeolithic and Mesolithic comes from lithic artefacts and faunal remains bearing traces of anthropogenic activity. The lithics include bi-truncated trapezoidal backed blades ('Cheddar points'), in addition to a single curve-backed blade ('Federmesser') along with straight-backed blades and bladelets, and Mesolithic microliths (ApSimon et al. 1992; Jacobi and Higham, 2011). Faunal remains of horse and red deer bear evidence of cut marks and cultural fractures, indicating exploitation and processing by humans. Radiocarbon dating of these faunal remains demonstrate at least two separate episodes of Palaeolithic human activity, each exploiting a different prey (Jacobi and Higham, 2011). Fractured horse teeth date to 15,515 to 14,315 cal. BP (IntCal20, 95% confidence interval, n=4, OxA-19161 (UBSSM catalogue number W2.21/485), OxA-19166 (W2.21/484), OxA-X-2280-8 (W2.21/559), OxA-X-2280-9(W2.21/560), Table 1) and provide the earliest evidence of humans in the British Isles after the Last Glacial Maximum (LGM) (Jacobi and Higham, 2011), Cut-marked red deer bones date to 14,160 to 13800 cal. BP (IntCal20, 95% confidence interval, n=2, OxA-19159 (W2.20/123), OxA-19160 (W2.20/187), Table 1) (Jacobi and Higham, 2011). Together these dates span the onset and establishment of the Late Glacial Interstadial, a major global climate transition characterised by rapid warming (Jacobi and Higham, 2011). Ancient



Figure 1. Location of King Arthur's Cave.

DNA and archaeological evidence from northern Europe indicate this period also witnessed major а human population turnover. alongside changes in mobility patterns, settlement structure, subsistence economy, technology and social organisation (Holzkämper et al. 2014: Miller. 2012: Pettitt and White, 2012: Maier, 2015: Naudinot et al. 2017: Fu et al. 2016; Posth et al. 2016; Charlton et al. 2022).

Understanding the ecological context of human activity at King Arthur's Cave is particularly important for understanding the subsistence strategies, mobility/settlement patterns, and landscape experiences of these early colonising populations. The

site was first discovered and excavated by Symonds in the 1870's (Symonds, 1871) and further examined by the University of Bristol Spelaeological Society between 1925 and 1929, and then again in 1952 (Hewer, 1924, 1926; Taylor, 1928; ApSimon, et al. 1992). Most recently, excavations were undertaken in the 1990s by Barton (Barton, 1995; 1996; 1997). Some material from the early excavations, which lacked the detailed stratigraphic analysis of modern excavations (Flas, 2011; Hublin, 2015), has so far been largely unstudied; lack of stratigraphic context for this material means it has been assumed to provide limited insight into the archaeology at the site. However, this view is transforming as new biomolecular techniques are developed which can unlock information from old archaeological collections, such as those from King Arthur's Cave. In particular, ZooMS (Zooarchaeology by Mass Spectrometry) is a proteomics approach that can be used to identify morphologically unidentifiable bone fragments. Family/genus/species level information can be gained from protein amino acid sequence variation assessed through peptide mass fingerprinting. Establishing taxonomic identifications on bone fragments previously thought to be 'unidentifiable' provides a more complete picture of faunal assemblage composition, enabling any difference in the species representation between morphologically identifiable and unidentifiable bones to be considered in relation to human subsistence behaviour. In addition, ZooMS analyses may identify additional human remains at an archaeological site. This is of particular value in later Pleistocene and early Holocene contexts in the British Isles, where discoveries of such specimens have so far been extremely infrequent. Here we use ZooMS to explore the taxonomic identification of previously unidentified bone fragments from King Arthur's Cave and undertake further radiocarbon dating to clarify the chronology of the site stratigraphy. Through these analyses we aim to provide further insight into the faunal community and local ecology during the Late Upper Palaeolithic and Mesolithic human occupation of the cave.

#### SAMPLE PROVENANCE

A total of 57 unidentified bone fragments were sampled for ZooMS for this study. Of these, 48 were recorded as being from the "1<sup>st</sup> Hearth and Humus" and 10 were recorded as being from the "Yellow Rubble and Mammoth Layer" from the platform from the University of Bristol Spelaeological Society excavations between 1925 and 1929. The platform supposedly consisted of six discrete Late Pleistocene/Holocene layers (Taylor, 1928). Radiocarbon dates from the platform are given in Table 1. A summary of the archaeology and morphologically identified fauna from the stratigraphic levels of interest is given below and see Figure 2..

The Humus (Unit 1) consisted of a grey-black humic soil which rested on Unit 2b (1<sup>st</sup> Hearth) at the mouth of the cave and further out on the Yellow Rubble (ApSimon, *et al.* 1992). The Humus layer, which is Holocene in origin, was contaminated with Pleistocene material from an overlying old spoil heap from previous excavations. Due to the mixture of Holocene and Pleistocene fauna in this layer, material from this horizon was not included in the zooar-chaeological report for the site (ApSimon, *et al.* 1992).

The 1<sup>st</sup> Hearth (Unit 2b) consisted of a blackish soil with weathered limestone clasts and much ash (ApSimon, et al. 1992). However, the age of the deposit is somewhat unclear. The lithics recovered from the 1<sup>st</sup> Hearth are reported to be typical of Mesolithic industries that include microliths. Numerous cut and chop marks on the bone show undoubted evidence of human activity. The 1<sup>st</sup> Hearth faunal assemblage primarily comprised red deer, aurochs and pig, with roe deer and horse also present, suggesting an early Holocene / Mesolithic age (ApSimon, et al. 1992). The presence of some sheep in the faunal assemblage is suggestive of a Neolithic or later date (ApSimon, et al. 1992). One brown bear carpal was also identified in this unit, which could be of Pleistocene or Holocene age (ApSimon, et al. 1992). The dating of a horse tooth from this unit to 14895 - 14230 cal. BP (IntCal20, 95% confidence interval) (OxA-V-2797-24C, 12,410 ± 50 BP, Reade, et al. 2020), alongside undated reindeer and spotted hyaena bones demonstrate Pleistocene material of both pre- and post-LGM age are also present within Unit 2b, although it is noted that the sample labels on both the reindeer and spotted hyaena specimens suggest they may have come instead from the old spoil heap (ApSimon, et al. 1992). Horse teeth from this unit were found in its lower part and were different in condition compared to the other bones, suggesting they may well have been incorporated into Unit 2b from the underlying Yellow Rubble (Unit 2c) (ApSimon, et al. 1992).

The majority of the faunal remains from the Yellow Rubble (Unit 2c) were red deer, with some horse present. Domestic species present in overlying layers were absent in this unit, except for a single pig tooth which was probably intrusive. Reindeer, red fox, arctic or collared lemming, northern and tundra voles, steppe pika and arctic hare were also present (ApSimon, *et al.* 1992). There are contradictions in reporting the contexts of part of the archaeological material which presently appear irresolvable (Jacobi and Higham, 2011), but it does appear that lithics typical of late Magdalenian (known locally as Creswellian) and Federmessergruppen industries were recovered from the Yellow Rubble, as well as the underlying 2<sup>nd</sup> Hearth (Unit 2d) and Mammoth Layer (Unit 3c). At the time of excavation human remains were reported to have been present in the Yellow Rubble but these have since been lost (ApSimon, *et al.* 1992).

The 2nd Hearth (Unit 2d) sits below the Yellow Rubble (Unit 2c) on the platform outside the cave. This is a blackish sediment with weathered limestone clasts contained great quantities of ash but no identifiable charcoal, burnt bone or hearth structures (ApSimon, *et al.* 1992). The unit was very rich in bones, which showed evidence of butchery but were more fragmented than those from the Yellow Rubble (ApSimon, *et al.* 1992). The  $2^{nd}$  Hearth





faunal assemblage comprised horses, red deer, and bovids. A single red deer tooth from this layer dates to 14840- 13790 cal. BP (IntCal20, 95% confidence interval) (OxA-1563, 12,210  $\pm$  120 BP) (Hedges, *et al.* 1989).

The Mammoth Layer (Unit 3c), separated from the Yellow Rubble (Unit 2c) by the 2<sup>nd</sup> Hearth (Unit 2d), was so named due to the discovery of a juvenile mammoth tooth and was initially assumed to date to before LGM (Hedges, *et al.* 1989). However, radiocarbon dating of faunal remains from the layer shows a mixed assemblage containing both pre- and post-LGM material (Hedges, *et al.* 1989; Jacobi and Higham, 2011). The faunal assemblage from this layer comprised horse, spotted hyaena, brown bear, mammoth, woolly rhinoceros, red deer, and large bovid (Bos/Bison). Radiocarbon dates from the Yellow Rubble and Mammoth Layer show some mixing of faunal material between the layers (Table 1). Three groupings can be seen in the radiocarbon dates; horse teeth and red deer bones both date to the Late Glacial, forming two separate groupings, and mammoth dates to at least 36,000 cal. BP (Figures 3 and 4, Table 1). Heavy gnawing of some of the faunal remains along with a lack of evidence of butchery or known pre-LGM lithic technology suggest that the pre-LGM faunal material was accumulated by hyaenas rather than humans (ApSimon, *et al.* 1992). It should be noted that the radiocarbon determinations on the mammoth specimens likely represent minimum ages due to the pre-treatment protocols and radiocarbon procedures used in the 1980s.

#### RADIOCARBON METHODOLOGY

Six new radiocarbon determinations were obtained, four from the Yellow Rubble and two from the Mammoth Layer. The collagen extraction and dating for three of the specimens was undertaken at Oxford Radiocarbon Accelerator Unit (ORAU) using their standard procedures (Brock, *et al.* 2010). For three specimens, collagen was extracted at UCL following the same procedure and the sample was subsequently radiocarbon dated at ORAU. To denote the bone pretreatment at UCL rather than at ORAU, the measured date was given "OxA-V-www-pp" numbers, where "wwww" indicates the wheel number, and "pp" is the position of the sample on the wheel (Brock, *et al.* 2010). A background correction was applied to these dates (OxA V 2754-50C, OxA-V-2797-25C, OxA-V-3058-28C) to account for the collagen extraction being performed at UCL, following the method outlined by Wood *et al.* (2010). Corrected dates are denoted by adding a "C" to the end of the date code assigned by ORAU. Results are reported as uncalibrated radiocarbon dates (14C BP) and discussed as calibrated dates BP (cal. BP, 95% confidence interval). Date calibration was performed using OxCal 4.4 (Bronk Ramsey, 2020) and the IntCal20 dataset (Reimer, *et al.* 2020).

## ZOOMS METHODOLOGY

Collagen peptide fingerprints were obtained following non-destructive collagen extraction methods (Buckley, *et al.* 2009; van Doorn, *et al.* 2011). Between 10 and 20 mg of bone were soaked overnight in 100  $\mu$ l of 50 mM Ammonium Bicarbonate (AmBic). The supernatant was discarded and samples were gelatinised in 100  $\mu$ l 50 mM AmBic for 1h at 65°C. When this protocol failed to provide reliable fingerprints, collagen extraction was performed using an HCl pretreatment (Welker, *et al.* 2016). Samples were demineralised in 0.6 M HCl at 4°C, rinsed with 50 mM AmBic, and incubated in 0.1 M NaOH for 5 min. After another rinse with 50 mM AmBic, gelatinisation was performed as previously described. Samples were then

Species	Element	Sample information	Stratigraphic unit	Lab code
Equus ferus	?Left upper M3	W2.21/285	1st Hearth	OxA-V-2797-24C
Equus ferus	Sesamoid	W2.20/147	Yellow Rubble	OxA-V-2797-25C
Cervus elaphus	Innominate, right, cut	W2.20/187	Yellow Rubble	OxA-19160
Cervus elaphus	Bone	KAC 10 W2.20/127.	Yellow Rubble	OxA-21183
Cervus elaphus	Tooth	W.2.21/468	Yellow Rubble	OxA-1562
Cervus elaphus	Dentary, partial right, cut	W2.20/123	Yellow Rubble	OxA-19159
Equus ferus	M3, left upper, fractured	W2.21/451	Yellow Rubble	OxA-19165
Equus ferus	P2, right lower, fractured	W2.21/485	Yellow Rubble	OxA-19161
Equus ferus	oesamoid	W2.20/148	Yellow Rubble	OxA-V-3058-28C
Equus ferus	M1/M2, right lower, fractured	W2.21/484	Yellow Rubble	OxA-19166
Cervus elaphus	Tooth	W.2.21/115	2 <sup>nd</sup> Hearth	OxA-1563
Cervus elaphus	Bone	KAC 11 W2.20/354.	Mammoth Layer	OxA-21184
Equus ferus	Lower left P3/P4, fractured	W2.21/726	Mammoth Layer	OxA-V-2754-50C
Equus ferus	Cheek tooth, left lower, fractured	W2.21/559	Mammoth Layer	OxA-X-2280-8
Equus ferus	M1/M2, right lower, fractured	W2.21/560	Mammoth Layer	OxA-X-2280-9
Mammuthus primigenius	Tooth	W.2.21/169	Mammoth Layer	OxA-1564
Mammuthus primigenius	Tooth	W.2.21/1185	Mammoth Layer	OxA-1565
Mammuthus primigenius	Tooth	W.2.21/954	Mammoth Layer	OxA-1566

Date	Uncertainty	Radiocarbon pre-treatment type	Date reference
12410	50	AF	Reade <i>et al</i> . 2020
10810	50	AF	This paper
12055	55	AF	Jacobi & Higham, 2011
12110	55	AF	This paper
12120	120	AC	Hedges <i>et al</i> . 1989
12140	50	AF	Jacobi & Higham, 2011
12450	60	AF	This paper
12490	60	AF	Jacobi & Higham, 2011
12507	31	AF	This paper
12565	80	AF	Jacobi & Higham, 2011
12,210	120	AC	Hedges et al. 1989
12145	55	AF	This paper
12450	50	AF	This paper
12680	90	AF	Jacobi & Higham, 2011
12720	90	AF	Jacobi & Higham, 2011
34850	1500	AC	Hedges <i>et al</i> . 1989
38500	2300	AC	Hedges <i>et al</i> . 1989
>39500		AC	Hedges et al. 1989

**Table 1.** Radiocarbon dates from the platform area of King Arthur's Cave. Further radiocarbon dates have been obtained from other areas of the site. Details of pre-treatment codes and protocols can be found in Brock, *et al* 2010 and Jacobi, *et al* 2006.

incubated overnight at 37°C with 0.4  $\mu$ g of sequencing grade modified trypsin (Promega). Following trypsin digestion, samples were acidified with 0.5% trifluoroacetic acid (TFA) and purified using PierceTM 100  $\mu$ l C18 resin Tips (Thermo Scientific) using conditioning and eluting solutions composed of 50% acetonitrile and 0.1% TFA. Collagen was eluted in 50  $\mu$ L.



**Figure 3.** Calibrated post-Last Glacial Maximum (LGM) radiocarbon dates from the platform areas of King Arthur's Cave. Blue = Equus ferus, Grey = Cervus elaphus. The radiocarbon ages are compared against the NGRIP  $\delta^{18}O$  ice core record.

For MALDI-TOF-MS, 0.5  $\mu$ L of the trypsin-digested extract was spotted with 0.5  $\mu$ L of a-cyano-hydroxycinnamic acid matrix solution (0.1% TFA in ACN/H2O 1:1 v/v) onto a 48 spot MALDI target plate, and air dried. MALDI-MS analyses were carried out in triplicate on a Shimadzu MALDI 8020 instrument, operating at up to 2000 laser shots per plate spot, over a m/z range of 900-4000. The mass spectra were calibrated against an adjacent MS standard spot containing eight calibrant peptides (TOFMixTM) of 0.8 to 3.7 kiloDalton (kDa) range (Bradyk-inin 1-7, angiotensin II, angiotensin I, Glu1-fibrinopeptide B, N-acetyl Renin substrate, ACTH 1–17 clip, ACTH 18–39 clip and ACTH 7–38 clip) – of which seven were used (1.0 – 3.7 kDa range). The obtained collagen fingerprints were manually inspected for the presence of relevant peptide markers (al 508 – a2 757; Brown, *et al.* 2021) in mMass v. 5.5.0 (Strohalm, *et al.* 

2010), after filtering peaks with a signal-to-noise ratio (S/N) threshold of 3.5, and using previously published collagen peptide markers from reference spectra (Buckley, *et al.* 2009, 2017; Welker, *et al.* 2016).



**Figure 4.** Calibrated pre- and post- Last Glacial Maximum (LGM) radiocarbon dates from the platform areas of King Arthur's Cave. Blue = Equus ferus, Red = Mammuthus primigenius, Grey = Cervus elaphus. The radiocarbon ages are compared against the NGRIP  $\delta^{18}O$  ice core record.

## **RESULTS AND DISCUSSION**

Four new radiocarbon determinations were obtained on bones from the Yellow Rubble. A red deer bone (OxA-21183, W2.20/127) was dated to 14845 to 14140 cal. BP, and a humanly fractured horse tooth (OxA-19165, W2.21/451) to 14975 to 14285 cal. BP. Both dates are consistent with previously published dates for this layer (Jacobi and Higham, 2011). Two further dates on horse bones, showing no evidencing of butchery or other anthropogenic modifications, were also obtained. These dated to 12,835 to 12,715 cal BP (OxA-V-2797-25C,

W2.20/147) and 15,015 to 14,490 cal BP (OxA-V-3058-28C, W2.20/148). The former is significantly younger, while the latter is consistent with the range of other dated horse remains from the site. From the Mammoth Layer fauna, two radiocarbon determinations were obtained. A culturally fractured horse tooth (OxA-V-2754-50C, W2.21/726) was dated to 14,995 to 14,305 cal. BP and a red deer bone (OxA-21184, W2.20/354) to 14,175 to 13,805 cal. BP. The horse date is consistent with the other dated horse remains from both the Mammoth Layer and Yellow Rubble, and the red deer date is consistent with the other dated red deer post-date the LGM; this contrasts with all mammoth, which predate the LGM. The six new radiocarbon determinations confirm the previous observed pattern of an age difference between horse and red deer at the site, related to at least two separate episodes of Late Upper Palaeolithic human activity (Jacobi and Higham, 2011). While we also find that one horse post-dates the red deer material, there is no evidence linking this particular bone to any human activity at the site. Despite some overall stratigraphic structure in the radiocarbon dates, it is clear that there has been a significant amount of mixing of material between stratigraphic units.

ZooMS Identification	No. of identified fragments: Humus / 1 <sup>st</sup> Hearth	No. of identified fragments: Yellow Rubble / Mammoth Layer
Alces, Cervus, Megaloceros, Saiga	29	4
Alces, Cervus, Megaloceros, Saiga, Capreolus	4	5
Bos/Bison	3	
Bovidae (Rupicapra, Ovis)	1	
Carnivora (Crocuta, Panthera)	1	
Equidae	1	1
Failed	3	
Homo	1	
Rhinocerotidae	1	
Suidae	3	
Total	47	10

**Table 2:** Results of ZooMS identification of bone fragments from King Arthur's Cave platform area.

The results of the ZooMS analyses are summarised in Table 2. The detailed identifications are given in Table 3, which is available in supplementary material at http://www.ubss.org.uk/resources/procsupplement/29\_2\_121-135.pdf For some fragments it was possible to use the collagen peptide fingerprints to identify the bone to a single genus/species, but for others it was only possible to restrict the identification to a range of genera. This information can, however, be considered in light of species' known biogeography and the morphologically identified species present at the site. Of the 57 fragments analysed for ZooMS, 27 from the Humus/1st Hearth Layer and 4 from the Yellow Rubble/Platform layer were identified to the genera Alces, Cervus, Megaloceros, or Saiga. While all these genera are possible based on their known biogeography, some of these identifications are more likely than others. The 1<sup>st</sup> Hearth morphologically identifiable faunal assemblage primarily comprised red deer (Cervus elaphus), which were also present in the Yellow Rubble and the Mammoth Layer. Thus, while it is likely that the ZooMS identified specimens are Cervus rather than the other genera, none can be ruled out. Specimens of giant deer (Megaloceros giganticus) were found in the morphologically identifiable material, although they came from the Lower Cave Earth from the Symmond's excavations within the cave and are likely to be older that the Mammoth Laver and pre-LGM in age (ApSimon, et al. 1992). However, as a small number of Megaloceros specimens recovered from Lancashire, Isle of Man, Scotland and Ireland have been dated to the Late Glacial, and from Devon, South Wales and Ireland to 47,000 cal. to 27,000 BP (Lister, et al. 2019), we cannot rule out the presence of the species in the late glacial King Arthur's Cave assemblage. Elk (Alces alces) were not present in the morphologically identifiable faunal assemblage at King Arthur's Cave, but a handful of Alces specimens dated to the Late Glacial / early Holocene are known from Lancashire, Cumbria, Yorkshire and Berkshire (Healy, et al. 1992; Kaagan, 2000; Hedges, et al. 1987; Jacobi and Higham, 2009; Smith, et al. 2013). Like Alces, Saiga was not found in the morphologically identifiable material, however a few specimens of Saiga (Saiga tatarica) dated to the Late Glacial have been identified in the Mendip Hills in Somerset (Currant and Jacobi, 2011; Gillespie, et al. 1985; Hedges, et al. 1989). A further four fragments from the Humus/1st Hearth Layer were identified via ZooMS to the above genera but could also be Caprelous (Roe deer), which was present in the morphologically identifiable fauna from the same contexts. Three fragments from the Humus/1st Hearth Layer were identified as Bos/Bison, and one as Equus, which is consistent with the presence of aurochs and horse in the morphologically identifiable fauna. One fragment from the Mammoth Layer was also identified as Equus, again consistent with the faunal assemblage from this context. Three fragments were identified via ZooMS as Suidae and one as Bovidae, either Rupicapra (chamois) or Ovis (domestic sheep). The Suidae could be wild boar which would likely be Mesolithic/early Holocene in age, or alternatively domestic pig and date to the Neolithic/middle Holocene. As Rupicapra are not known from Late Pleistocene or Holocene contexts in Britain it seems much more likely the latter fragment comes from a domestic sheep. One fragment from the Humus/1<sup>st</sup> Hearth Layer was identified as *Rhinocerotidae* and in the context of Late Pleistocene Britain, this would be a woolly rhino (Coelodonta antiquitatis). This woolly rhino likely dates to the pre-LGM period as this species is not known from any reliable stratigraphic context during or after the LGM (Stuart and Lister, 2012). One fragment from the Humus/1st Hearth Layer was identified as a carnivore, either Crocuta or Panthera; both are present in the morphologically identifiable faunal assemblage from the site (ApSimon, et al. 1992). Finally, one fragment from the Humus/1st Hearth Layer was identified as human (see below for further discussion).

Overall, the ZooMS results reinforce the picture of a mixed stratigraphy that includes both Pleistocene and early Holocene fauna. The extent to which this mixing represents more historic post-depositional processes versus more recent contamination from the old spoil heap of previous excavations is unclear. However, additional radiocarbon dating and stable isotope analysis of the assemblage may further resolve the age of individual animals and the ecological setting they inhabited (e.g. Stevens, *et al.* 2021).

The identification of a human amongst the bone fragments from the Humus/1<sup>st</sup> Hearth Layer is of particular interest. It is possible that the human fragment could be early Holocene in age due to the presence of Mesolithic lithics in unit 2b (1<sup>st</sup> Hearth). Alternatively, the human bone could be Late Upper Palaeolithic in age as a horse recovered from the 1<sup>st</sup> hearth has been dated to 14,895 to 14,230 cal. BP (OxA-V-2797-24C). Furthermore, H. Taylor recorded 21 (possibly 22) fragments of human remains from the Yellow Rubble layer (unit 2C) (UBSS catalogue entries, found 23 Sept 1929). Taylor's 1952 faunal list notes them without suggesting disturbance, thus they could have been of Mesolithic or Palaeolithic age (ApSimon, et al. 1992). These specimens are now sadly lost (ApSimon, et al. 1992). This most likely occurred during the Second World War when a bomb landed on the UBSS Museum, and a large amount of the collections were destroyed. Given that Late Upper Palaeolithic human remains have only been recovered from three other sites in the British Isles (Gough's Cave, Sun Hole Cave and Kendrick's Cave), the discovery of a Late Upper Palaeolithic specimen would be an exciting find, particularly because ancient DNA studies at Gough's Cave and Kendrick's Cave have shown that two genetically distinct human populations were present in the British Isles around this time (Charlton, et al. 2022). However, more recent human remains, dating to 5,592 to 5,411 cal. BP (OxA-5863, 4,670± 60 14C BP, Hedges, et al. 1997) have previously been recovered from King Arthur's Cave. Thus, there is the distinct possibility that this human bone fragment is Holocene in age. The specimen is currently being AMS radiocarbon dated and we eagerly await the results.

## ERRATUM

After this paper was accepted for publication, the result of the AMS date for the human bone showed the specimen is from the Early Bronze Age, dating to 3,830 to 3,590 cal. BP (IntCal20, 95% confidence interval) (OxA-V-3138-28C,  $3440 \pm 20$  14C BP).

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## REFERENCES

- ApSimon, A.M., Smart, P.L., Macphail, R., Scott, K., Taylor, H., 1992. King Arthur's Cave, Whitchurch, Herefordshire: reassessment of a Middle and Upper Palaeolithic, Mesolithic and Beaker site. Proceedings of the University of Bristol Spelaeological Society. 19. 2, 183-249.
- Barton, R.N.E. 1995. Third interim report on the survey and excavations in the Wye Valley. *Proceedings* of the University of Bristol Speleological Society. **20.** 2. 153-159.

- Barton, R.N.E. 1996. Fourth interim report on the survey and excavations in the Wye Valley. *Proceedings* of the University of Bristol Speleological Society. **20.** 3, 263-273.
- Barton, R.N.E. 1997. Fifth interim report on the survey and excavations in the Wye Valley, and new AMS radiocarbon dating results from Madawg Rockshelter. *Proceedings of the University of Bristol Speleological Society.* 21. 1. 99-108.
- Brock, F., Higham, T., Ditchfield, P., Bronk Ramsey, C. 2010. Current pretreatment methods for AMS radiocarbon dating at the Oxford Radiocarbon Accelerator Unit (ORAU). *Radiocarbon.* 52. 1, 103-112.
- Bronk Ramsey, C. 2020. OxCal Version 4.4. http://c14.arch.ox.ac.uk/oxcal.html .
- Brown, S., Douka, K., Collins, M., Richter, K.K. 2021. On the Standardization of ZooMS Nomenclature. *Journal of Proteomics.* 235, 104041.
- Buckley, M., Harvey, V.L., Chamberlain, A.T. 2017. Species identification and decay assessment of Late Pleistocene fragmentary vertebrate remains from Pin Hole Cave (Creswell Crags, UK) using collagen fingerprinting. *Boreas*. 46. 402-411.
- Buckley, M., Collins, M., Thomas-Oates, J., Wilson, J.C., 2009. Species identification by analysis of bone collagen using matrix-assisted laser desorption/ionisation time-of-flight mass spectrometry. *Rapid Communications in Mass Spectrometry*. 23. 23. 3843-3854.
- Charlton, S., Brace, S., Hajdinjak, M., Kearney, R., Booth, T., Reade, H., Tripp, J., Sayle, K.L., Grimm, S.B., Bello, S.M., Walker, E.A., Gilardet, A., East, P., Glocke, I., Larson, G., Higham, T.F.G., Stringer, C., Skoglund, P., Barnes, I. Stevens, R.E. (2022). Dual ancestries and ecologies of the Late Glacial Palaeolithic in Britain. *Nature Ecology and Evolution*. **6**, 1658-1668.
- Currant, A., Jacobi, R. 2001. A formal mammalian biostratigraphy for the Late Pleistocene of Britain. *Quaternary Science Reviews.* **20**, 1707-1716.
- Flas, D. 2011. The Middle to Upper Paleolithic transition in Northern Europe: the Lincombian-Ranisian-Jerzmanowician and the issue of acculturation of the last Neanderthals. *World Archaeology*. 43. 4, 605-627.
- Fu Q, Posth C, Hajdinjak M, Petr M, Mallick S, Fernandes D, et al. 2016. The genetic history of Ice Age Europe. Nature. 534: 200-205. pmid:27135931
- Gillespie, R., Gowlett, J.A.J., Hall, E.T., Hedges, R.E.M., Perry, C. 1985. Radiocarbon dates from the Oxford AMS System: Archaeometry Datelist 2. *Archaeometry*. **27**, 237-246.
- Healy, F., Heaton, M., Lobb, S.J., Allen, M.J., Fenwick, I.M., Grace, R., Scaife, R.G. 1992. Excavations of a Mesolithic Site at Thatcham, Berkshire. *Proceedings of the Prehistoric Society*. 58, 41-76.
- Hedges, R.E.M., Housley, R.A., Law, I.A., Perry, C., Gowlett, J.A.J. 1987. Radiocarbon dates from the Oxford AMS system: Archaeometry datelist 6. *Archaeometry*. 29, 289-306.
- Hedges, R.E.M., Housley, R.A., Law, I.A., Bronk, C.R. 1989. Radiocarbon dates from the Oxford AMS system: Archaeometry datelist 9. Archaeometry. 31. 207-234
- Hewer, T.F. 1924. First report on excavations in the Wye Valley. Proceedings of the University of Bristol Spelaeological Society, 2. 2. 147-155.

- Hewer, T.F. 1926. Second report on excavations in the Wye Valley. Proceedings of the University of Bristol Spelaeological Society. 2. 3. 216-228.
- Holzkämper, J., Kretschmer, I., Maier, A., Baales, M., von Berg, A., Bos, J.A.A., Bradtmöller, M., Edinborough, K., Flohr, S., Giemsch, L., Grimm, S.B., Hilpert, J., Kalis, A.J., Kerig, T., Langley, M.C., Leesch, D., Meurers-Balke, J., Mevel, L., Orschiedt, J., Otte, M., Pastoors, A., Pettitt, P., Rensink, E., Richter, J., Riede, F., Schmidt, I., Schmitz, R.W., Shennan, S., Street, M., Tafelmaier, Y., Weber, M.-J., Wendt, K.P. Weniger, G.-C. & Zimmermann, A. 2014. The Upper–Late Palaeolithic transition in western Central Europe. Typology, technology, environment and demography. Report on the workshop held in Rösrath, 21st–24th June 2012. Archäologische Informationen. 36. 161-86.
- Hublin, J.J. 2015. The modern human colonization of western Eurasia: when and where? *Quaternary Science Reviews.* **118.** 194-210.
- Jacobi, R.M., Higham, T.F.G., Bronk Ramsey, C. 2006. AMS radiocarbon dating of Middle and Upper Palaeolithic bone in the British Isles: improved reliability using ultrafiltration. *Journal of Quaternary Science.* 21. 557-573.
- Jacobi, R.M., Higham, T.F.G. 2009. The early Lateglacial re-colonization of Britain: new radiocarbon evidence from Gough's Cave, southwest England. *Quaternary Science Reviews.* 28, 1895-1913.
- Jacobi, R. and Higham, T. 2011. The Later Upper Palaeolithic recolonisation of Britain: new results from AMS radiocarbon dating. In: Ashton, N., Lewis, S, Stringer, C., (Eds.), The ancient human occupation of Britain. *Developments in Quaternary Science*. 14. 223-247.
- Kaagan, L. M. 2000. *The horse in Late Pleistocene and Holocene Britain*. Unpublished PhD thesis, University College London.
- Lister, A.M., Stuart, A.J. 2019. The extinction of the giant deer Megaloceros giganteus (Blumenbach): new radiocarbon evidence. *Quaternary International*. **500.** 185-203.
- Maier, A. 2015. The Central European Magdalenian: regional diversity and internal variability. Dordrecht: Springer.
- Miller, R. 2012. Mapping the expansion of the Northwest Magdalenian. *Quaternary International.* 272-273. 209-230.
- Naudinot, N., Tomasso, A., Messager, E., Finsinger, W., Ruffaldi, P. and Langlais, M. 2017. Between Atlantic and Mediterranean: changes in technology during the Late Glacial in Western Europe and the climate hypothesis. *Quaternary International.* 428(B). 33–49.
- Pettitt, P.B. and White, M.J. 2012. The British Palaeolithic. Human Societies at the Edge of the Pleistocene World. London. Routledge.
- Posth, C., Renaud, G., Mittnik, A., Drucker, D.G., Rougier, H., Cupillard, C., Valentin, F., et al. 2016. Pleistocene Mitochondrial Genomes Suggest a Single Major Dispersal of Non-Africans and a Late Glacial Population Turnover in Europe. Current Biology. 26. 6. 827-33.
- Reade, H., Holloran, F., Tripp, J., Charlton, S., Jourdan, A.-.L., Stevens, R.E. 2020. Late glacial palaeoclimate investigations at King Arthur's Cave and Sun Hole Cave. *Proceedings of the University of Bristol Spelaeological Society*, 28. 2. 221-238.

- Reimer, P.J., Austin, W.E.N., Bard, E. Bayliss, A., Blackwell, P.G., Ramsey, C.B. *et al.* 2020. The IntCal20 Northern Hemisphere radiocarbon age calibration curve (0–55 cal kBP). *Radiocarbon.* 62. 4. 725-757.
- Smith I.R., Wilkinson D.M., and O'Regan H.J. 2013. New Lateglacial fauna and early Mesolithic human remains from northern England. *Journal of Quaternary Science*. 28. 542-544.
- Stevens, R.E., Reade, H., Tripp, J.A., Sayle, K.L., Walker, E.A. 2021. Changing environment at the Late Upper Palaeolithic site of Lynx Cave, North Wales. Festschrift in honour of Martin Street and Elaine Turner. *Monography-Series of the Römisch-Germanisches Zentralmuseum, Leibniz-Research-Institute for Archaeology.*
- Strohalm, M., Kavan, D., Novak, P., Volny, M., and Havlicek, V. 2010. mMass 3: a crossplatform software environment for precise analysis of mass spectrometric data. *Analytical Chemistry*. 82(11). 4648-4651.
- Stuart, A.J., and Lister, A.M. 2012. Extinction chronology of the woolly rhinoceros Coelodonta antiquitatis in the context of late Quaternary megafaunal extinctions in northern Eurasia. Quaternary Science Reviews. 51. 1-17.
- Symonds, W.S. 1871. On the contents of a hyena's den on the Great Doward, Whitchurch, Ross. Geological Magazine. 8. 433-438.
- Taylor, H. 1928. King Arthur's Cave, near Whitchurch, Ross-on-Wye. Second Report: Excavations in 1926-1927. Proceedings of the University of Bristol Spelaeological Society, 3. 2. 59-83.
- van Doorn, N.L., Hollund, H., Collins, M.J. 2011. A novel and non-destructive approach for ZooMS analysis: ammonium bicarbonate buffer extraction. *Archaeological and Anthropological Sciences.* **3**, 281-289.
- Welker F, Hajdinjak M, Talamo S, et al. 2016. Palaeoproteomic evidence identifies archaic hominins associated with the Chatelperronian at the Grotte du Renne. *Proceedings of the National Academy of Sciences of the United States of America*. **113**, 11162–11167.
- Wood, R,E., Bronk Ramsey, C., and Higham, T.F.G. 2010. Refining background corrections for radiocarbon dating of bone collagen at ORAU. *Radiocarbon.* 52. 2. 600-611.

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