



A Journal for Linear Monuments, Frontiers & Borderlands Research Volume 6

Edited by Howard Williams

Aims and Scope

Offa's Dyke Journal is a peer-reviewed venue for the publication of high-quality research on the archaeology, history and heritage of linear monuments, frontiers and borderlands. The editors invite submissions that explore dimensions of Offa's Dyke, Wat's Dyke and the 'short dykes' of western Britain, including their life-histories and landscape contexts. ODJ will also consider comparative studies on the material culture and monumentality of land divisions, boundaries, frontiers and borderlands from elsewhere in Britain, Europe and beyond from prehistory to the present day. We accept:

- 1. Notes and Reviews of up to 3,000 words
- 2. Interim reports on fieldwork of up to 5,000 words
- 3. Original discussions, syntheses and analyses of up to 10,000 words

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Front cover: Photograph of the excavated ditch of Offa's Dyke, Chirk, north-facing section (Ian Grant, CPAT Photo 4565-0134)

Offa's Dyke Journal

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Offa's Dyke and Wat's Dyke: Scientific Dating at Chirk and Erddig

Paul Belford, Ian Grant and Tim Malim

In 2018 and 2019, the Clwyd-Powys Archaeological Trust undertook excavations on Offa's Dyke at Chirk Castle, and on Wat's Dyke at Erddig. The background, circumstances and stratigraphic narrative of these projects were presented in Volume 1 of this journal, but the scientific dating programme was not complete at the time of publication and the results were further delayed due to the COVID-19 pandemic. This paper describes the radiocarbon and OSL dates obtained by 2021 and discusses implications for future research.

Keywords: Dykes, Chirk, Erddig, excavation, radiocarbon dating, OSL dating

Introduction

Offa's and Wat's Dykes are the pre-eminent linear earthworks within the Marches of England and Wales, and are amongst the longest and largest in Britain. They have been subject to detailed study and debate by successive generations of antiquarians and archaeologists, but beyond their geographic location, physical character and topographic positioning in the landscape, most discussion before the twenty-first century has used historical context to attribute a chronological framework for their construction and use. With the exception of Fox's excavation at Ffrith which deliberately targeted a Roman site to help determine whether Offa's Dyke pre- or post-dated the Roman settlement, archaeological investigations largely relied on serendipitous finds of artefacts or charcoal to provide dating evidence. The nature of the dykes, however, is sufficiently different to settlement that the finding of chance artefacts in association with these earthworks is remote. Additionally, the strategy of excavation has almost always comprised narrow trenches across the bank and ditch, rather than a broader excavation of the earthworks and their surroundings, an approach which reduces the chance of finding contextual and artefactual evidence.

Therefore, despite numerous surveys, excavations and speculations (over 80 excavations are together listed in Hill and Worthington's 2003 gazetteer for each dyke), dating evidence for the linear earthworks of Offa's Dyke and Wat's Dyke has been limited. Three excavations provided a broad chronological context using conventional landscape and artefactual evidence. Fox (1927, 1955) found Roman material in the bank of Offa's Dyke at Ffrith (Flintshire); Varley (1976) found a hearth and recovered an Anglo-Saxon loom weight within the infill and near the base of the ditch of Wat's Dyke at Mynydd Isa (Flintshire); and Everson (1991) showed that Offa's Dyke pre-dated surrounding ridge-and-furrow earthworks at Dudston

(Shropshire). A very wide 'early medieval' (i.e. post-Roman but pre-Norman) date has been accepted for these dykes, but not refined.

More recently, however, scientific dating methods have begun to be applied in the context of development-driven fieldwork undertaken within the planning system, or state-funded 'rescue' archaeology. Until now this also comprised just three projects, namely:

- 1. Wat's Dyke at Mile Oak, Oswestry (Shropshire). Radiocarbon date from a hearth which 'predated the construction of the bank by only a very short space of time'; the calibrated date had a range of cal. AD 411–561 (1- σ) or cal. AD 268–630 (2- σ), suggesting a 'most likely' mid-fifth century date for the construction of the Dyke (Hannaford 1997: 5–6).
- 2. Wat's Dyke at Gobowen, Oswestry (Shropshire). Seven OSL dates for the buried soil and ditch infill sequence, of which the four associated with the primary and secondary fill episodes all overlapped within the period AD 792–852, and a contemporaneous one from the buried soil horizon; suggesting a possible construction date in the early-ninth century (Malim and Hayes 2008: 173–175).
- 3. Offa's Dyke at Plas Offa, Chirk (Wrexham). Four radiocarbon dates from turf that had been redeposited at the base of the bank during its construction; three within the range cal. AD 539–635 (1- σ) or cal. AD 430–652 (2- σ), and the fourth in the range cal. AD 897–990 (1- σ) or cal. AD 887–1019 (2- σ). Due to the circumstances of the project further work was not possible (Grant 2014; Belford 2017: 69).

These results are now joined by radiocarbon and OSL dates from four further excavations: on Offa's Dyke at Chirk Castle (Wrexham), on Wat's Dyke at Erddig (Wrexham), on Wat's Dyke at Greenfield Valley (Flintshire) and Rhosrobin (Wrexham). This article presents the results of the scientific dating programme associated with the first two projects and is effectively an extension to an earlier paper in this journal describing the circumstances, background and stratigraphic results of the excavations (Belford 2019). The projects at Greenfield Valley and Rhosrobin are part of ongoing research and will be reported on following further fieldwork and analysis.

Background

The excavations at Chirk and Erddig were undertaken by the Clwyd-Powys Archaeological Trust (CPAT) in 2018 and 2019, with funding from Cadw, the National Trust and the Dee Valley and Clwydian Range Area of Outstanding National Beauty (AONB). Both projects were located in the landscaped grounds of National Trust properties in Wrexham County Borough, specifically in 'picturesque' parkland designed by William Eames. This landscaping work took place in the 1760s and 1770s at both

properties when Eames retained some earlier landscape features but largely levelled both dykes. Fieldwork took place at Erddig in 2018, and at Chirk in 2018 and 2019 (Grant and Jones 2019a; Grant and Jones 2019b).

Some stratigraphic information is provided here to enable understanding of the context in which the samples were obtained, but this account is best read in conjunction with the earlier one which includes trench locations, plans and photographs (Belford 2019).

Dating methods

A total of 32 samples (17 bulk soil samples which were processed by flotation for retrieving charcoal, and 15 specific sediment samples for OSL dating) were taken from both excavations. Not all of these were subjected to full analysis owing to several factors including the unsuitability of some samples for one or more of the dating methods chosen, and the effects that residual material might have had on the reliability of any dates obtained. Several samples from each site were analysed, with both radiocarbon and OSL dating undertaken at the Scottish Universities Environmental Research Centre (SUERC) in 2019–2021 (Tables 1 and 2). The OSL methodology is summarised in Appendix 1. Figures 1 and 4 show samples that were processed by SUERC and dates achieved, rather than showing all sample locations listed in Tables 1 and 2.

Radiocarbon dating was undertaken on single fragments of charcoal derived from identified species (Table 2) from bulk soil samples processed and assessed by Durham University's Archaeological Services. These were pre-treated by SUERC to remove impurities before analysis using accelerator mass spectrometer (AMS). Detailed descriptions of the methods employed by the SUERC Radiocarbon Laboratory are described in Dunbar *et al.* (2013). The radiocarbon ages were calibrated to the calendar timescale using the Oxford Radiocarbon Unit calibration program OxCal 4, and the date ranges calibrated using the IntCal 20 atmospheric calibration curve (Bronk Ramsey 2009; Reimer *et al.* 2020).

Optical stimulated luminescence (OSL) dating of sediments depends on the accumulation of signals induced by naturally occurring ionising radiation in silicates, which can be stimulated to release measurable luminescence. Daylight effectively 'zeroes' sediment which has been sufficiently exposed to light at the time of deposition, and thus a new signal is developed subsequently. These signals are measured and quantified as equivalent radiation doses using calibrated laboratory sources. In complex sedimentary systems the extent of zeroing may be incomplete, for example in archaeological layers where bulk re-deposition of construction materials takes place with insufficient light exposure. Samples in these cases have residuals and may yield mixed age estimates from different portions (Cresswell et al. 2019). Two samples were dated by these means, one for Offa's Dyke at Chirk, and one for Wat's Dyke at Erddig Hall (Table 3). The methodology applied to this analysis is summarised in Appendix 1, but for both these

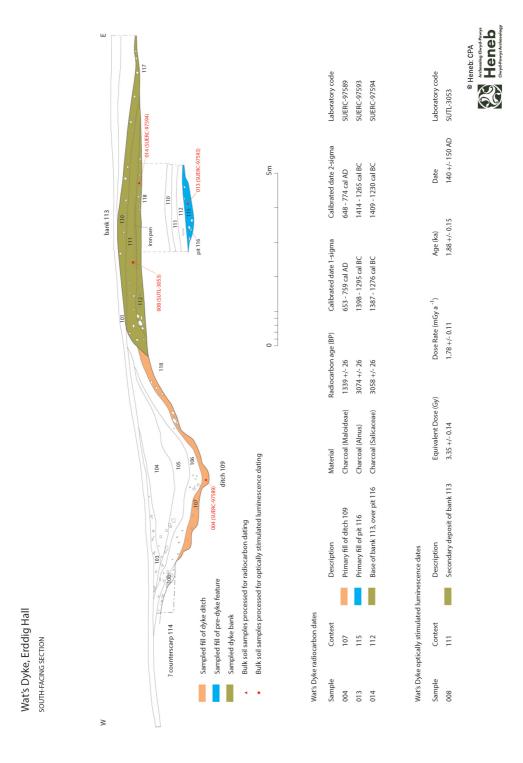


Figure 1: Section drawing of Wat's Dyke, Erddig, showing dyke profile, deposit sequence and sample locations, with resultant C14 and OSL dates tabulated (drawn by Richard Hankinson)

Table 1: list of samples taken for OSL dating samples on-site gamma measurements were not taken when the sampling took place.

SUERC code	Site sample number	Depth from ground surface	Description					
Chirk Castle (Offa's Dyke)								
SUTL3047	#107	2.9-3.0m	Basal fill of ditch [023]. Context #021					
SUTL3048	#108	2.4m	Secondary fill of ditch [023]. Context #019					
SUTL3049	#111	1.08m	Secondary fill of pit [032] underlying bank [027]. Context #029					
SUTL3050	#117	0.6m	Basal or secondary construction deposit at central point of bank [027] overlaying a firm pa grey silty clay deposit (034) – probably natura subsoil. Context #024					
SUTL3056			Six profile samples from the ditch [023]					
SUTL3056/1	#101	1.95-2.00m	Upper section of tertiary fill (019) of ditch [023]					
SUTL3056/2	#102	2.1-2.2m	Mid-section of tertiary fill (019) of ditch [023]					
SUTL3056/3	#103	2.3-2.4m	Lower section of tertiary fill (019) of ditch [023]					
SUTL3056/4	#104	2.5-2.7m	Secondary ditch fill (020) of ditch [023].					
SUTL3056/5	#105	2.85-3.00m	Upper section of basal ditch fill (021) of ditch [023].					
SUTL3056/6	#106	3.1m	Lower section of basal ditch fill (021) of ditch [023].					
Erddig (Wat's Dyke)								
SUTL3051	#006	0.9m	Fill of pit [116] underlying Dyke bank [113]. Context #115					
SUTL3052	#007	0.7m	Basal deposit of bank [113]. Context #112					
SUTL3053	#008	0.5m	Secondary deposit of bank [113]. Context #111					
SUTL3054	#009	1.9m	Basal deposit of ditch [109], SW facing section. Context #107					
SUTL3055	#010	1.9m	Basal deposit of ditch [109], NW facing section. Context #107					

Table 2: C14 samples

Sample No	Context type	Identification	Lab No	Age BP	l sigma	2 sigma and probability %	
ODCC 106 Context 21	Ditch primary	Alder	SUERC 97595	1569 <u>+</u> 26	cal. AD 436–465 cal. AD 475–501 cal. AD 508–516 cal. AD 530–548	cal. AD 428–562 95.4%	
ODCC 110 Context 31	Pit primary	Birch	SUERC 97596	2239 <u>+</u> 26	378–353 cal. BC 287–228 cal. BC 219–211 cal. BC	388–346 cal. BC 25.1% 316–204 cal. BC 70.4%	
ODCC 119 Context 24	Bank primary	Alder	SUERC 97597	2506 <u>+</u> 26	768–748 cal. BC 688–666 cal. BC 642–567 cal. BC	776–719 cal. BC 21.4% 709–662 cal. BC 19.2% 653–543 cal. BC 54.8%	
WDEH 004 Context 107	Ditch primary	Maloideae	SUERC 97589	1339 <u>+</u> 26	cal. AD 653–680 cal. AD 748–759	cal. AD 648–703 66.6% cal. AD 741–774 28.9%	
WDEH 013 Context 115	Pit	Alder	SUERC 97593	3074 <u>+</u> 26	1398–1370 cal. BC 1356–1295 cal. BC	1414–1265 cal. BC 95.4%	
WDEH 014 Context 112	Bank primary	Salicaceae (willow)	SUERC 97594	3058 <u>+</u> 26	1387–1339 cal. BC 1318–1276 cal.BC	1409–1258 cal. BC 92.3% 1245–1230 cal. BC 3.2%	

Table 3: OSL sample dates

Sample No.	Lab No.	Description	Equivalent	Dose Rate	Age (ka)	Date
			Dose (Gy)	(mGy a-1)		
108	3048	Chirk Castle:	3.79 ± 0.09	3.18 ± 0.08	1.19 ± 0.05	AD 830
		Secondary fill of				± 50
		ditch				
008	3053	Erddig Hall:	3.35 ± 0.14	1.78 ± 0.11	1.88 ± 0.15	AD 140 ±
		Secondary				150
		deposit of bank				

Wat's Dyke at Erddig

An excavation trench was located at SJ 3258 4799, oriented east—west across the line of Wat's Dyke which at this point ran north—south. Despite Eames' levelling operations, the bank (113) was found to have survived to a height of 0.7m. It had been constructed directly over the natural subsoil, overlying an earlier shallow pit (116). The bank consisted of three layers, the earliest being a gritty silt (112); this was overlain by a sandy silt (111) which was in turn capped with a clay-cobble deposit (110). The ditch (109) had been cut through the undisturbed natural geology and survived to a depth of 1.5m. It had been filled by four deposits in sequence, all probably derived from natural weathering, and eventually sealed by a deposit (possibly levelling) (104) of eighteenth—nineteenth-century origin (Grant and Jones 2019a; Belford 2019).

A total of nine samples, taken from four contexts, were submitted for further analysis ((107), (111), (112) and (115)). Three of these samples were processed for radiocarbon dating and one for OSL dating. The resulting dates, together with the south-facing section of the trench annotated to show context numbers and sample locations, are presented in Figure 1 and a photograph of the excavated trench is shown in Figure 2.

In summary, the radiocarbon dating produced an unambiguous Bronze Age date for the fill of the pit beneath the bank of Wat's Dyke (115), but perhaps surprisingly, it produced a similar date for the lower level of the bank (112). This might be explicable by the samples having been contaminated by disturbance of this pit feature when the later earthwork was constructed. The date for the primary fill of the ditch (107), however, was indicative of the monument having been constructed before the end of the seventh century AD.

A total of five samples were taken for OSL processing, two from basal fills of the ditch, two from bank deposits, and one from the fill of the pit beneath the bank (Table 1). Analysis of three of these samples produced mixed results. The sample from pit fill



Figure 2: Photograph of excavated trench through Wat's Dyke, Erddig, looking north-east (Ian Grant, CPAT Photo 4526-0117)

(115) indicated high residual signals which meant that it was not possible to obtain an accurate date. Although laboratory profile measurements for both bank samples showed low apparent doses (and therefore the samples were probably consistent with archaeological ages), there were high residual signals in the sample from the lower layer (112).

Sample 008 from the secondary deposit of the bank (111) was analysed further. Profile measurements suggested that the quartz in this sample is bright and was zeroed prior to deposition. Equivalent doses were determined for 29 aliquots from this sample, with three aliquots rejected due to low sensitivity leading to very large uncertainties. The distribution of these equivalent doses is shown in Figure 3. This produced a broad dose distribution with a weighted mean age of AD 140 \pm 150. This is clearly earlier than the expected date of construction of the bank. An exploratory evaluation of single-grain analysis confirmed that a small proportion of individual quartz grains (between 1 and 5%) gave measurable OSL signals, with doses that would correspond to mid-first millennium AD ages.

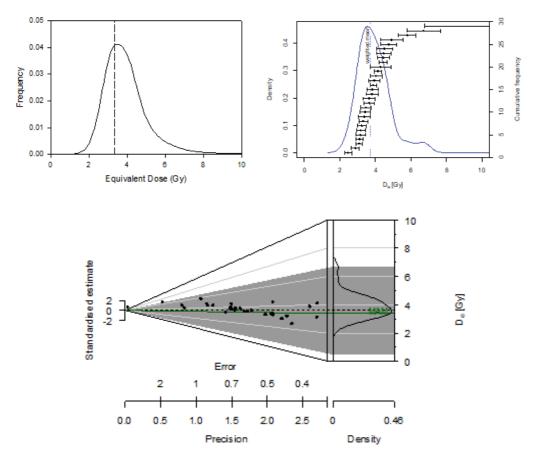


Figure 3: Dose distribution for SUTL3053 displayed as a probability density plot (top left), KDE (top right) and abanico plot (bottom). The dashed line indicates the weighted mean

Offa's Dyke at Chirk Castle

An excavation trench was located at SJ 2694 3871, oriented north-west to south-east across the line of Offa's Dyke. The Dyke had been levelled at this point but was evident as an upstanding earthwork extending to the south-west. The original 2018 trench measured 29.0m by 1.5m in plan, later widened to 3.0m along the north side for most of its length. In 2019 this trench was partly re-excavated and extended to the east and south (Grant and Jones 2019b; Belford 2019).

As with Wat's Dyke at Erddig, the bank (27) had survived Eames' landscaping works and was extant to a height of around 0.40m. It had also been constructed partly over an earlier shallow pit (32) which itself had been cut through the subsoil; this was filled with a series of silty clay deposits. The ditch (23) was up to 2.80m deep including a vertically sided 'ankle-breaker' at the bottom. Most of the ditch fills appear to have been derived from the weathering of the bank, but this took place in distinct stages.



Figure 4: Section drawing of Offa's Dyke, Chirk, showing dyke profile, deposit sequence and sample locations, with resultant C14 and OSL dates tabulated (drawn by Richard Hankinson)



Figure 5: Photograph of the excavated ditch of Offa's Dyke, Chirk, north-facing section (Ian Grant, CPAT Photo 4565-0134)

The lower four fills (22, 21, 20 and 19) were sealed by a silty clay which appeared to have been *in situ* for some time before the post-medieval deposition of material from the levelling of the bank (Belford 2019; Grant and Jones 2019b).

A total of fifteen samples were taken from eight contexts. Three of these samples were subjected to radiocarbon dating and one to OSL dating. The resulting dates, together with the north-east-facing section of the trench annotated to show context numbers and sample locations, are presented in Figure 4, and Figure 5 is a photograph across the excavated ditch showing its profile and infill deposits. The C14 date from the primary fill of the ditch suggests construction during the middle of the first millennium AD, however, the sample from the basal bank deposit (24) with an Early Iron Age date could include material from the preceding prehistoric landscape, which had been incorporated as part of the bank construction. Pit (32) with its Middle Iron Age date, was cut into this earlier prehistoric landscape.

A total of two sediment samples were taken from basal ditch fills for OSL dating, and six samples for OSL profiling through the infill sequence. In addition, a single sample for OSL dating was taken from the base of the bank (SUTL3050), and another sample was taken from the fill of the pit beneath the bank (SUTL3049) (Table 1). The OSL, on both quartz and polymineral, yielded archaeological age apparent doses (<10Gy) for the majority of the samples from the ditch with larger doses for the pit and bank. The

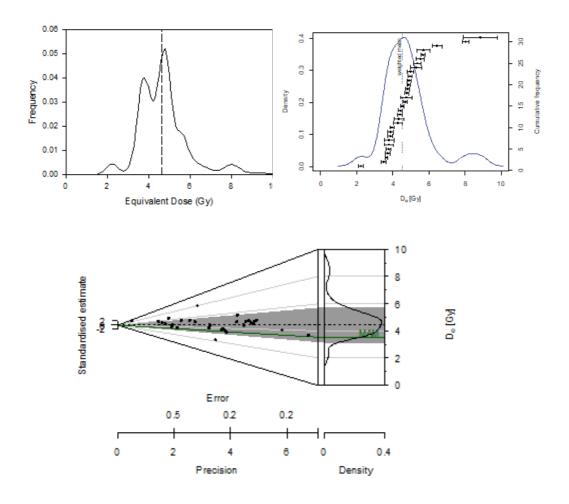


Figure 6: Dose distribution for SUTL3048 displayed as a probability density plot (top left), KDE (top right) and abanico plot (bottom). The dashed line indicates the weighted mean

ditch fill profiles showed that they had been sufficiently exposed to daylight to reset their dose, and the sequence seemed to be consistent with older dates for deeper sample locations. The OSL date for the primary fill (21) sample SUTL3047 (at 2.9-3.0m) has a significantly larger apparent dose and appears to be similar to the bank material. The sample selected for full analysis (SUTL 3048) came from the secondary fill (19) of the ditch and gave a date range of AD 830 ± 50 (Table 6). Profile measurements suggested that the quartz in this sample is bright and well zeroed prior to deposition. The distribution of these equivalent doses is shown in Figure 6.

Discussion

It is clear from these results that there is considerable potential for the deployment of scientific dating techniques on linear earthwork monuments such as Offa's Dyke and Wat's Dyke. Although there are inconsistencies between the results obtained here and elsewhere, and between the outcomes of different approaches and techniques, the results at Chirk and Erddig have further proved the efficacy of OSL dating in helping to understand linear earthworks, as demonstrated previously on Wat's Dyke by Malim and Hayes (2008). OSL dating needs to be deployed with care and understanding in how and where (stratigraphically) samples are taken and processed, as the degree to which residual effects are present can be better determined with careful profiling by specialists in the field. In the present context, the fact that charcoal was found at both excavations at Erddig and Chirk meant that the parallel use of radiocarbon dating has proved extremely valuable.

Comparison between the two techniques helps to correlate their validity, and Bayesian modelling could further refine the dates of specific events represented by the samples. At present these initial results have not been subject to such mathematical modelling, but this might be undertaken when funding becomes available and especially when further data is available to enhance the model, as the existing single OSL dates for each dyke in combination with the C14 determinations, would not provide sufficient results for effective statistical modelling.

The C14 and OSL results for Wat's Dyke at Erddig demonstrate the existence of prehistoric and Romano-British activity which include a Bronze Age pit sealed by the bank, and deposits within its make-up that had been disturbed during Iron Age or Roman times (OSL date of AD 140 ± 150). The primary infill episode for the ditch produced a C14 date in the second half of the seventh century AD, whereas the primary bank deposit appears to have included material from the Bronze Age pit which it had sealed, as it produced a very similar date to the pit. There is an alternative interpretation, however, which is that if the bank's basal deposit is correctly dated then its original construction in this location was during the Bronze Age which might explain the Iron Age/Roman date for the secondary deposit as an erosion event of bank material during this period. By analogy other linear earthworks have been dated to the Bronze Age, such as the Devils Mouth Dyke on the Long Mynd (Hankinson and Caseldine 2006), or West Wansdyke at Blackrock Lane, Publow (Erskine 2007). The primary fill for the ditch could then represent an early medieval remodelling of an earlier linear earthwork (also suggested for Wat's Dyke at Oswestry based on its proximity to standing stones and other prehistoric remains beneath, within, or in close vicinity, to the earthwork (Malim 2020), as the primary infill episode can be considered to act as a reasonably accurate terminus ante quem proxy indicator for the date of the ditch cut.

At Offa's Dyke a prehistoric pit was also found sealed by the bank, but the Cl4 date from its primary fill indicates an Iron Age origin, rather than Bronze Age. The Cl4 sample from the base of the bank, however, produced an earlier date in the Iron Age, and it is therefore assumed that this represents a land surface buried by the later earthwork. The Cl4 and OSL dates from the primary fills of the ankle-breaker ditch are from the

fifth—sixth centuries and the early ninth century AD, with their respective stratigraphic sequence aligning with sub-Roman and early medieval dates. The primary fills, however, were recorded in the field as discrete deposits grouped together by a clay sealing layer, before the bank had been deliberately pushed into the ditch. The c. 300–400 years difference between these dates therefore needs some interpretation, unless they reflect a long gradual process of infill, and that the ditch was excavated in Roman or immediately post-Roman times (as suggested for Wat's Dyke by Hannaford 1998, and similar to three or the four dates obtained for Plas Offa (Belford 2019)). Alternatively, the charcoal at the base of the sequence could have derived from material that was on the surface and fell in, soon after the ditch was excavated. It is worth noting that the OSL date corresponds closely with results obtained for Wat's Dyke at Gobowen (Malim and Hayes 2008).

Conclusion

The results do not conclusively confirm that either Offa's Dyke or Wat's Dyke were constructed around the middle of the first millennium AD. What they do confirm is that these linear earthworks were constructed into a landscape which already had evidence for prehistoric and Roman activity. Single C14 dates from the primary fills of each of the monument ditches suggest that both Offa's and Wat's Dykes are post-Roman, and a secondary fill for Offa's Dyke is OSL dated to the early ninth century, but unfortunately these conclusions are not supported by the results of dating from the banks. It is always possible, of course, that these linear earthworks have a more complex history than generally believed, and that the existing monument may be the latest in a sequence of such monuments.

The methodological approach to find the most effective sampling strategy and allied techniques for processing those samples for OSL, continue to be developed through detailed dialogue between archaeologists and scientists. New investigations on the dykes since 2021 have provided opportunities for close collaboration and enhancement of our approach. These have followed the normal strategy of relatively narrow excavation slots across the bank and ditch, as the principal aim has been to retrieve samples for scientific dating, and to assess whether the bank construction and infill sequences largely correspond between geographically different parts of these monuments. Open area excavations as undertaken by Jon Cane at Pentre Wern when the A5 was constructed near Gobowen in 1984-1985 (Cane 1996), and in 2006 further north at Gobowen (Malim and Hayes 2008) is another approach which would justify further adoption, as this allows other elements of the monument and its context to be better appreciated. For example, the extent of turf stripping, kerbstones, evidence for a marking out bank, palisade features, pits and hearths, ploughing or other agricultural activity, so that our understanding of how the linear earthwork was constructed, its impact on the pre-existing landscape and how it has survived later changes within that landscape, can be enhanced.

Further work is required to refine understanding of the chronology of the construction of these monuments, and to produce more sophisticated approaches to the use of scientific dating techniques in similar contexts. The authors would recommend that in future any work on these sorts of monuments – whether undertaken as research projects, or as part of development-driven or 'rescue' archaeology work, or in association with conservation and land management work – should include provision for adequate scientific dating as a matter of course. This should comprise multiple samples with good stratigraphic control, with as many dating techniques as possible applied so that OSL and C14, artefacts and stratigraphy can all be used to correlate the results, and Bayesian modelling adopted to better refine the event dates that the archaeological evidence provides.

Appendix 1: OSL dating methodology

A.J. Cresswell and D.C.W. Sanderson (SUERC)

Laboratory Profile Measurements

All sample handling and preparation was conducted under safelight conditions in the SUERC luminescence dating laboratories. Each sample was wet sieved to extract the 90–250 µm grain size fraction. This was subjected to an acid treatment of 1M HCl for 10 minutes, 15% HF for 10 mins and 1M HCl for 10 mins, with the sample washed thoroughly with deionised water between each treatment. Approximately half of the material was retained, washed in acetone to displace water and dried as a polymineral sample. The remaining material was subjected to a further acid treatment of 40% HF for 40 mins and 1M HCl for 10 mins, with the sample washed thoroughly with deionised water between each treatment. This fraction was washed in acetone to displace water and dried as a nominal quartz sample.

Clean 10mm diameter stainless steel discs were prepared with one side sprayed with silicone grease as an adhesive layer, with sample material dispensed as a monolayer onto the central ~5mm of the disc. For each sample, a pair of polymineral and a pair of quartz discs were dispensed.

Luminescence sensitivities (Photon Counts per Gy), sensitivity changes and stored doses (Gy) were evaluated from the paired aliquots of the polymineral and HF-etched quartz fractions, using Risø DA-15/DA-20 automatic readers equipped with a 90Sr/90Y β-source for irradiation, using blue LEDs emitting around 470nm (OSL) and infrared (laser) diodes emitting around 830nm (IRSL) for optical stimulation, and a U340 detection filter pack to detect in the region 270–380nm. For quartz, each measurement was preceded by a pre-heat at 200°C for 10s, with a 30s OSL measurement at 125°C. Measurements were conducted for the natural signal, and following nominal 5 Gy and 50 Gy irradiations, with all measurements accompanied by a nominal 1 Gy test dose. For the polymineral samples, each measurement was preceded by a pre-heat at

 200° C for 10s, with a 30s IRSL measurement at 50° C and a TL measurement to 500° C. Measurements were conducted for the natural signal, and following nominal 5 Gy and 50 Gy irradiations. No test dose measurements were included.

Quartz SAR measurements

Approximately 50 g of material was removed for each tube and processed to obtain sand-sized quartz grains for luminescence measurements. Each sample was wet sieved to obtain the 90–150 and 150–250 μm fractions. The 150–250 μm fractions were treated with 1 M hydrochloric acid (HCl) for 10 minutes, 15% hydrofluoric acid (HF) for 15 minutes, and 1 M HCl for a further 10 minutes. The HF-etched sub-samples were then centrifuged in sodium polytungstate solutions of ~2.58, 2.62, and 2.74 g cm-3, to obtain concentrates of potassium-rich feldspars (<2.58 g cm-3), sodium feldspars (2.58–2.62 g cm-3) and quartz plus plagioclase (2.62–2.74 g cm-3). The selected quartz fraction was then subjected to further HF and HCl washes (40% HF for 40 mins, followed by 1M HCl for 10 mins).

All materials were dried at 50°C and transferred to Eppendorf tubes. The 40% HF-etched, 2.62-2.74 g cm-3 'quartz' 150-250 µm fractions were dispensed to 10 mm stainless steel discs for measurement. Initially, 16 aliquots were dispensed for each sample with further aliquots dispensed as required to improve the determination of dose distributions. The purity of which was checked using a Hitachi S-3400N scanning electron microscope (SEM), coupled with an Oxfords Instruments INCA EDX system, to determine approximate elemental concentrations for each sample.

Equivalent dose determinations were made on sets of 16 aliquots per sample, using a single aliquot regeneration (SAR) sequence (cf. Murray and Wintle 2000). Using this procedure, the OSL signal levels from each individual disc were calibrated to provide an absorbed dose estimate (the equivalent dose) using an interpolated dose-response curve, constructed by regenerating OSL signals by beta irradiation in the laboratory. Sensitivity changes which may occur as a result of readout, irradiation and preheating (to remove unstable radiation-induced signals) were monitored using small test doses after each regenerative dose. Each measurement was standardised to the test dose response determined immediately after its readout, to compensate for changes in sensitivity during the laboratory measurement sequence. The regenerative doses were chosen to encompass the likely value of the equivalent (natural) dose. A repeat dose point was included to check the ability of the SAR procedure to correct for laboratoryinduced sensitivity changes (the 'recycling test'), a zero dose point is included late in the sequence to check for thermally induced charge transfer during the irradiation and preheating cycle (the 'zero cycle'), and an IR response check included to assess the magnitude of non-quartz signals. Regenerative dose response curves were constructed using doses of 1, 3, 6, 9, 12 and 20 Gy, with test doses of 1.0 Gy. The 16 aliquot sets were sub-divided into four subsets of four aliquots, such that four preheating regimes were explored (200°C, 220°C, 240°C and 260°C). All measurements were conducted using a

Risø DA-15 automatic reader equipped with a 90Sr/90Y β -source for irradiation, blue LEDs emitting around 470 nm and infrared (laser) diodes emitting around 830 nm for optical stimulation, and a U340 detection filter pack to detect in the region 270-380 nm, while cutting out stimulating light (Bøtter-Jensen et al., 2000).

The data were processed to determine quality parameters for the SAR procedure, with any aliquot which failed these tests rejected from further analysis, as follows:

- the sensitivity (c Gy-1) was determined from the response to the first test dose;
- the sensitivity change is determined from the difference between the last and first test dose responses divided by the number of measurement cycles, as a percentage of the first test dose;
- the recycling ratio is the ratio of the normalised OSL measurement for the repeat of the first regenerative dose divided by the normalised OSL measurement for the first regenerative dose. This should be unity;
- the zero cycle response is the normalised OSL measurement following the zero dose cycle. This should be zero;
- the IR response is the ratio of the response to IR stimulation following a IGy dose to the response to blue stimulation following a IGy dose. This should be zero;
- the dose recovery test uses the response to the first test dose normalised using the response to the first regenerative dose to confirm that the curve fitting returns the test dose value. This should be IGy.

For each regenerative dose, the OSL counts normalised using the corresponding test dose are plotted against dose and an exponential rise to maximum curve fitted through the data. These are plotted for the average of each of the four pre-heating groups and for all samples, and any differences between the pre-heating groups noted. Any aliquots showing significantly different dose responses compared to the other aliquots are removed from the analysis. The equivalent dose for each aliquot is determined by interpolation of the normalised natural OSL counts to the fitted curve.

Dose rate measurements

Field gamma spectrometry (FGS) measurements were not made at the time of sampling, therefore dose rates have been determined exclusively from the sampled material. Locally averaged gamma dose rates have been determined from all the relevant samples, and used in place of FGS measurements. For the ditch of Offa's Dyke these are the two samples from the ditch (SUTL3047 and 3048) and the sample from the bank (SUTL3050), on the assumption that the bank material is representative of the soil layers the ditch is excavated into. For the bank of Wat's Dyke these are the two samples from the bank (SUTL3052 and 3053).

Laboratory measurements of dose rate were conducted using dried bulk material from the surrounding of the sample tubes for High Resolution Gamma Spectrometry (HRGS) and from materials from the tubes for Thick Source Beta Counting (TSBC). Dating materials were weighed, saturated with water and re-weighed. Following oven drying at 50 °C to constant weight, the actual and saturated water contents were determined as fractions of dry weight. These data were used, together with information on field conditions to determine water contents and an associated water content uncertainty for use in dose rate determination.

HRGS measurements were performed using a 50% relative efficiency 'n' type hyperpure Ge detector (EG&G Ortec Gamma-X) operated in a low background lead shield with a copper liner. Gamma ray spectra were recorded over the 30 keV to 3 MeV range from each sample, interleaved with background measurements and measurements from SUERC Shap Granite standard in the same geometries. Sample counts were for 80 ks. The spectra were analysed to determine count rates from the major line emissions from 40K (1461 keV), and from selected nuclides in the U decay series (234Th, 226Ra + 235U, 214Pb, 214Bi and 210Pb) and the Th decay series (228Ac, 212Pb, 208Tl) and their statistical counting uncertainties. Net rates and activity concentrations for each of these nuclides were determined relative to Shap Granite by weighted combination of the individual lines for each nuclide. The internal consistency of nuclide specific estimates for U and Th decay series nuclides was assessed relative to measurement precision, and weighted combinations used to estimate mean activity concentrations (Bq kg-1) and elemental concentrations (% K and ppm U, Th) for the parent activity. These data were used to determine infinite matrix dose rates for alpha, beta and gamma radiation.

Beta dose rates were also measured directly using the SUERC TSBC system (Sanderson, 1988). Count rates were determined with six replicate 600 s counts on each sample, bracketed by background measurements and sensitivity determinations using the Shap Granite secondary reference material. Infinite-matrix dose rates were calculated by scaling the net count rates of samples and reference material to the working beta dose rate of the Shap Granite $(6.25 \pm 0.03 \text{ mGy a-1})$. The estimated errors combine counting statistics, observed variance and the uncertainty on the reference value.

The dose rate measurements were used in combination with the assumed burial water contents, to determine the overall effective dose rates for age estimation. Cosmic dose rates were evaluated by combining latitude and altitude specific dose rates (0.185 \pm 0.01 mGy a-1) for the site with corrections for estimated depth of overburden using the method of Prescott and Hutton (1994).

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