



A luminescence-derived cryptostratigraphy from the Lake Suigetsu sedimentary profile, Japan: 45,000–30,200 IntCal20 yr BP

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ABSTRACT

The luminescence characteristics of sediments are affected by a variety of environmental factors, reflecting both local and broader regional influences. If seeking to apply stimulated luminescence as a ‘pure’ dating technique, variability in these external variables needs to be controlled for, involving, *inter alia*, lengthy pretreatment procedures and complex dose rate corrections. However, in so doing, a lot of potentially valuable palaeoenvironmental information is lost.

Instead, in the present study, we explicitly analysed raw, non-pretreated sediment that preserves this wealth of contributory environmental influence. Using a SUERC portable luminescence (POSL) reader, we performed rapid profiling across a 14,800 year interval of the annually laminated (varved) Lake Suigetsu sedimentary profile, central Japan (i.e., 45,000 to 30,200 IntCal20 yr BP), producing 303 contiguous measurements with a mean sampling resolution of 49 years. To further inform our understanding of this dataset, additional follow-up laboratory dosing was performed to provide sensitivity estimates.

The ‘cryptostratigraphy’ (‘hidden stratigraphy’) revealed by our data includes the identification of a step-change in luminescence parameters circa 39,200 IntCal20 yr BP, which we attribute to a major earthquake that resulted in re-routing of inflow to the lake. Further variability in the derived luminescence signals is compared with supporting high resolution x-ray fluorescence (μXRF) data and palynological data from Lake Suigetsu. A correlation between the luminescence profile (both net infra-red-stimulated and net blue light-stimulated signals) and mean annual temperature is revealed, mediated through subtle differences in sediment characteristics under warmer or cooler climatic conditions.

1. Introduction

Luminescence dating, including optically-stimulated luminescence (OSL) and infra-red-stimulated luminescence (IRSL), represents a well-established chronometric technique that can be applied across a range of depositional environments, including loess (e.g., Fenn et al., 2020),

sand dunes (e.g., Stone and Thomas, 2008) and, more recently, marine (e.g., Sanderson and Kinnaird, 2019) and lacustrine sedimentary archives (e.g., Roberts et al., 2018; Píšková et al., 2019; for a more comprehensive list of references see, e.g., Preusser et al., 2008 or Bateman, 2019). If the mineral grains (typically, quartz and feldspar) contained within such archives were fully bleached (i.e., ‘zeroed’) by

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their last exposure to light (or heat) then measurement of the accumulated luminescence signal within the grains can provide an estimate of the time elapsed since that zeroing event. Depending on the specific depositional context, bleaching (from sunlight) may cease at the point of deposition and consequent burial of said mineral grains and, hence, a stimulated luminescence measurement in such circumstances can provide an estimated age of that deposition.

As with other trapped charge dating methods (i.e., thermoluminescence, TL, and electron-spin resonance, ESR), OSL and IRSL utilise the accumulating signal within mineral grains that results from exposure to naturally-occurring ionising radiation, principally from uranium, thorium and potassium within the sedimentary matrix, as well as receipt from cosmic rays (e.g., Aitken, 1998). Accordingly, the luminescence signal conveyed by mineral grains is affected by the amount of ionising radiation (the ‘dose rate’) received within the burial environment. Another key variable affecting the luminescence signal is the sensitivity of the grains themselves to this ionising radiation (i.e., accumulated luminescence response per unit radiation dose), which is fundamentally related to their mineralogy.

Therefore, if seeking to apply luminescence for dating purposes, both the environmental dose rate and mineral sensitivity need to be accounted for in order to calculate the age of deposition from the measured luminescence response. This can involve time consuming chemical pretreatment procedures (involving hazardous chemicals, such as hydrofluoric acid, HF, under red light conditions) to extract the optimal grains to elicit the most reliable dating information possible. Furthermore, the environmental dose rate must be ascertained for the specific mineral phases examined, and this too can involve lengthy laboratory processes and/or involve the incorporation of much uncertainty, for example, through the estimation of palaeo-moisture content within the depositional regime.

Of course, mineral sensitivity and environmental dose rate are not random variables; rather, these characteristics are affected by a variety of environmental factors, including those acting at a very localised scale as well as those reflecting broader regional influences. Through application of the aforementioned pre-treatment procedures, an attempt is made to ‘correct’ for the totality of these environmental variables, allowing the derivation of an estimated age since deposition. However, if instead dating is *not* the primary goal, including situations where robust chronology is provided by some other independent technique, such ‘corrections’ for environmental variability need not be performed, and luminescence might instead be used as a palaeoenvironmental proxy, rather than its customary role as a chronometer (e.g., Munyikwa et al., 2021). The present study seeks to achieve just this, taking advantage of the uniquely well-dated sedimentary archive underlying Lake Suigetsu, central Japan, to assess the utility of OSL and IRSL as a complementary proxy for palaeoenvironmental reconstruction.

2. Study site: Lake Suigetsu, Japan

Lake Suigetsu (‘Suigetsu-ko’) is a 34 m deep tectonic lake situated on the western side of the active Mikata fault in Fukui Prefecture, central Japan (35° 35' N, 135° 53' E). It covers a surface area of ~4.3 km², and forms part of a five lake system, the ‘Mikata-goko’, such that, in the present day, the majority of water flowing into Suigetsu first passes through the shallower adjacent Lake Mikata (Fig. 1). This hydrological situation, in combination with the protection from winds afforded by the surrounding Palaeozoic hills (maximum elevation 400 m), results in bottom water anoxia that enables the preservation of laminations composed of seasonally distinctive material in the sediments underlying the lake, collectively comprising varves (Schlolut et al., 2012, Fig. 2).

It is these varves that make Suigetsu’s sediments so valuable to the field of Quaternary science. The stratigraphy provides the world’s longest continuous varve record yet identified, extending ~38,000 years between circa 10,200 and 48,000 SG06₂₀₁₈vyr BP (‘Suigetsu varve years Before Present’, where ‘Present’ is defined as 1950 CE; Schlolut et al.,

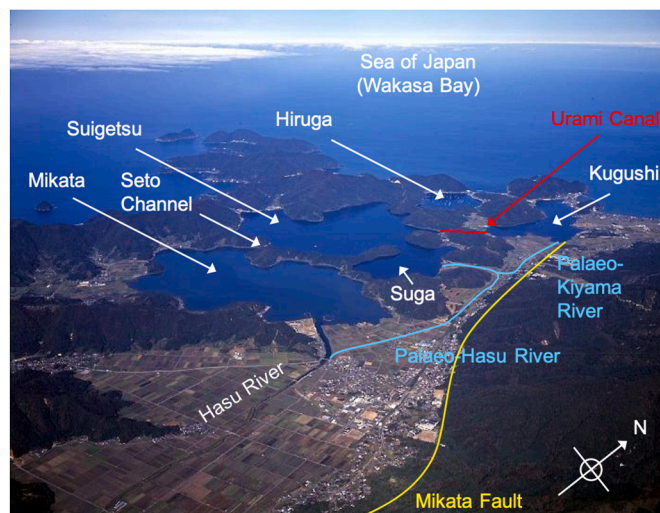


Fig. 1. Aerial photograph of the ‘Mikata-goko’ (the ‘Mikata Five Lakes’), comprising Lakes Mikata, Suigetsu, Suga, Hiruga and Kugushi, Fukui Prefecture, central Japan. In the present day, the major inflow to Suigetsu is from the south, via the Seto Channel from Lake Mikata and, ultimately, from the Hasu River. Prior to 39,242 ± 60 IntCal20 yr BP, however, we interpret that inflow to Suigetsu was from the east, via Lake Suga from the extended palaeo-Hasu River (light blue; Schlolut et al., 2014). The approximate positions of the active Mikata fault (yellow) and artificial Urami Canal (completed in 1664 CE; red) are also shown.

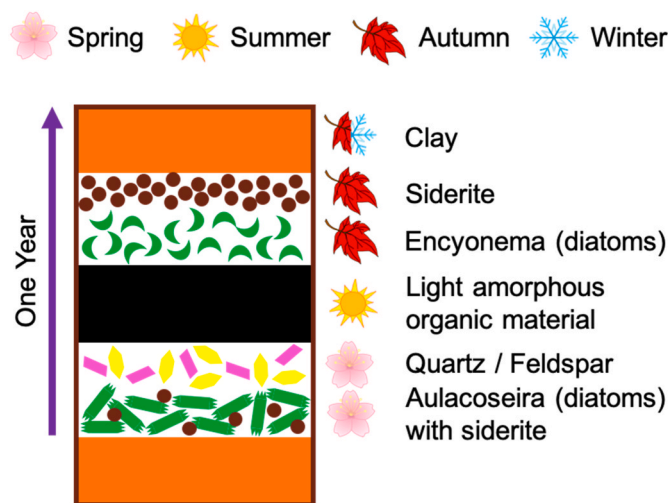


Fig. 2. Schematic representation of the seasonally-deposited layers that combine to form the varves (annual laminations) present down the Lake Suigetsu sediment profile (after Schlolut et al., 2012).

2018). In combination with this varve chronology, an extensive ($n > 800$) radiocarbon (¹⁴C) dataset contributes the only non-reservoir-corrected data to the pre-tree-ring (i.e., pre-13,900 IntCal20 yr BP) portion of the international consensus radiocarbon calibration curve, ‘IntCal’ (Bronk Ramsey et al., 2020; Reimer et al., 2020). Moreover, the global significance of the lake’s palaeoenvironmental record is demonstrated by its acceptance as an ‘auxiliary Global Stratotype Section and Point’ (GSSP) for the onset of the Holocene (Walker et al., 2009).

To investigate these sediments, Suigetsu has been subject to four deep drilling campaigns carried out in 1993, 2006, 2012 and 2014, retrieving composite sediment cores ‘SG93’, ‘SG06’, ‘SG12’ and ‘SG14’, respectively. Regular visibly distinctive macroscopic event layers enable

the ‘perfect’ alignment of the stratigraphies of these cores (Nakagawa et al., 2012, Fig. 3), at least for the upper ~45 m (representing the last circa 70,000 years), and thus the chronology developed (primarily from SG06, with additional ^{14}C analyses from SG93) is readily transferred to the parallel cores.

Taking advantage of this chronology, much palaeoenvironmental research has already been undertaken on these sediments, including: diatom species analysis (Kossler et al., 2011; Saito-Kato et al., 2013); palynology (e.g., Nakagawa et al., 2021); and stable isotope analysis of diatom silica ($\delta^{18}\text{O}$) and plant lipid biomarkers ($\delta^2\text{H}$; Rex, 2023). Detailed sedimentological analysis has been performed by both ultra high resolution ITRAXTM x-ray fluorescence (μXRF) core scanning and more classical optical microscopy (Marshall et al., 2012). Of the abundant macroscopic event layers present, ≥ 362 have been identified from the last ~40,000 years alone as being the result of flooding, with a further ≥ 7 event layers attributed to earthquakes (Schlola et al., 2014). Furthermore, the stratigraphy includes 32 visible volcanic ash (tephra) layers, with an additional 39 distinct cryptotephra layers also having been identified from the sediments (e.g., Smith et al., 2013; McLean et al., 2018; Albert et al., 2024), although on-going work will increase the number of the latter yet further.

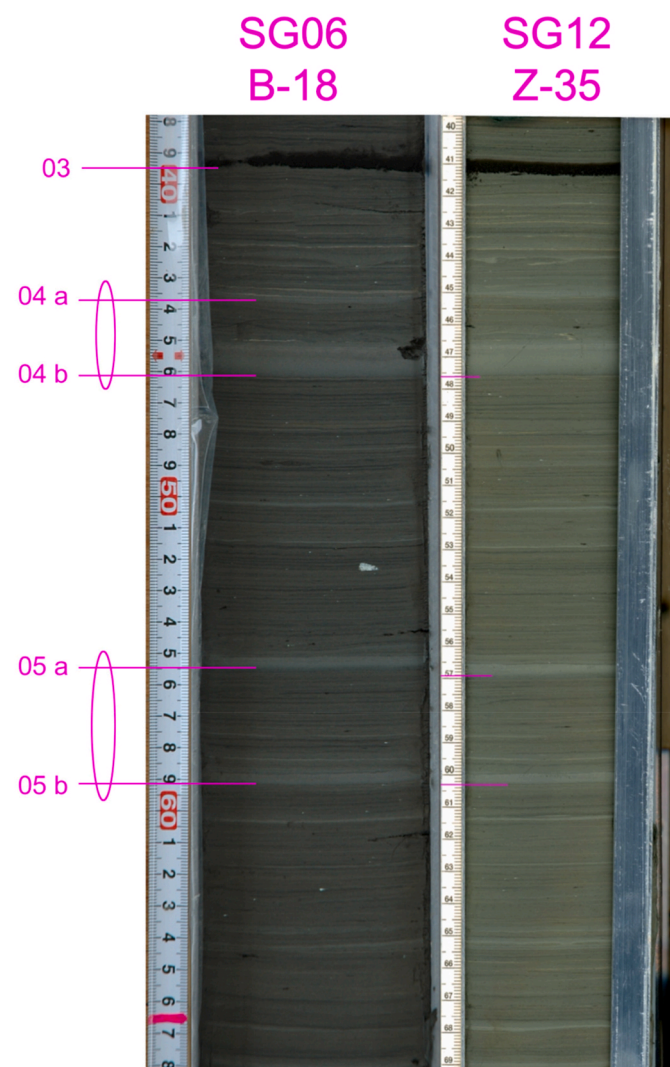


Fig. 3. Stratigraphic alignment of parallel sediment cores ‘SG06’ and ‘SG12’ enabled via the regular, visibly distinctive macroscopic event layers (Nakagawa et al., 2012). Example shown is between SG06 core section B-18 and SG12 core section Z-35, representing sediment accumulation from circa 43,950 to 43,650 IntCal20 yr BP.

Recently, Rex et al. (2022) expanded this corpus of palaeoenvironmental investigation by performing pilot luminescence analyses to assess the characteristics of the Lake Suigetsu sediments. To this end, four distinct time intervals were investigated, chosen such that they covered a selection of environmental features of interest. These were: (1) the last ~500 years (i.e., the uppermost metre of the stratigraphy, spanning the anthropogenic connection of the lake with the nearby Sea of Japan and, hence, transition from freshwater to brackish water in Suigetsu); (2) circa 45,000 to 35,000 IntCal20 yr BP (coinciding with the relatively abrupt climatic fluctuations of Greenland Interstadials 12 to 7; Rasmussen et al., 2014); (3) circa 73,000 to 69,500 years ago (spanning the onset of varve preservation within Suigetsu); and (4) circa 139,000 to 119,000 years ago (Glacial Termination II). Because of the range of time spans represented by these four intervals, different sampling approaches were undertaken, with periods (1) and (3) sampled contiguously (at ~25 year and ~310 year resolution, respectively), whilst periods (2) and (4) were point sampled (both with approximately millennial spacing). The study successfully demonstrated that the luminescence profiles registered variability in catchment behaviour. Of particular note were the prominent peaks in net luminescence responses (to both infra-red and blue light stimulation) that coincided with visibly distinctive flood layers from the last 500 years (time period (1)). An additional conclusion of the study was that the luminescence signals were more readily interpretable, and therefore most useful in terms of understanding the underlying sedimentological characteristics, when contiguous sampling was performed. Accordingly, the present study builds upon this previous work, applying a high chronological resolution contiguous sampling approach to investigate in greater detail the intriguing low resolution data structure evident from the ~45,000 to 35,000 IntCal20 yr BP time interval (period (2)) investigated by Rex et al. (2022).

3. Material and methods

Whereas the previous study by Rex et al. (2022) involved millennially-spaced point sampling between 45,000 and 35,000 IntCal20 yr BP from Lake Suigetsu sediment core SG06, herein we collected 303 contiguous, 3 cm-thick sediment samples from the parallel core SG12, i.e., between 2650.3 and 3593.8 cm composite depth (CD; version Dec 07, 2021), spanning a time range of circa 45,000 to 30,200 IntCal20 yr BP. This represents an extension of the interval previously investigated by Rex et al. (2022) (their period (2)) by a further 4800 years, with the upper boundary of our extended interval marked by the ~35.1 cm thick visible Aira-Tanzawa (AT) tephra (Smith et al., 2013). The mean sampling resolution (i.e., integrated time span represented by a single sample) of our present study was 49 years across the entire study period (inter-quartile range of 35–60 years) although, due to variability in sedimentation rate, this approximately halved from a mean sampling resolution of 35 years in the oldest 5000 years (inter-quartile range of 29–40 years; i.e., a sedimentation rate of ~0.86 mm per year) to 70 years in the youngest 5000 years (inter-quartile range of 60–83 years; i.e., a sedimentation rate of ~0.43 mm per year).

Sampling was performed under dim red light conditions from previously intact archive half core sections, removing the top ~2–3 mm of sediment from the surface that would have been light-exposed during the original longitudinal splitting of the core sections (Armitage and Pinder, 2017). With an awareness from the study by Rex et al. (2022) that flood-related material carries an enhanced luminescence response, we aimed to cut out such layers from our samples, since our aim in the present study was to seek to understand the additional factors impacting upon luminescence characteristics of the sediment. However, removal of event layers is practically challenging and thinner flood layers in particular may nevertheless have escaped such exclusion.

Each sample was physically homogenised and then spread evenly across the surface area of a 5 cm diameter Petri dish, prior to measurement with a SUERC portable luminescence (POSL) reader

(Sanderson and Murphy, 2010). The default ‘continuous wave proxy’ (‘CWproxy’) measurement scheme was followed, consisting of two consecutive exposures of 30 s each to both infra-red (IRSL; 880 nm) and blue light (OSL; 470 nm) stimulation, bracketed by 15 s dark counts (Supplementary Fig. 1). From this scheme, a suite of parameters is defined, principally: net IRSL and net OSL counts (i.e., total counts for the full 60 s of IR and blue light stimulation, respectively); IRSL and OSL depletion indices (i.e., the ratio of counts from the first 30 s to that of counts from the second 30 s of IR and blue light stimulation, respectively); IRSL:OSL ratio (total IRSL counts divided by total OSL counts); and post-stimulation phosphorescence (PSP; i.e., any increase in the 15 s dark count after IR or blue light stimulation compared to the 15 s dark

count preceding stimulation). For greater detail of the calculation of these parameters, see Supplementary Text. Empty chamber measurements were interspersed after every fourth sample to check for contamination in the instrument, and standard samples (Morar Sand and Granulite) were run at the start and/or end of each sample batch to verify instrument performance.

Quasi repeat analyses were undertaken by thoroughly re-mixing the sediment within the Petri dish and re-measuring with the POSL reader. It should therefore be noted that these repeat analyses are not true duplicates since a slight attenuation of the photon counts might be expected, with a minor proportion of the material having potentially already been exposed to light in the reader during the initial

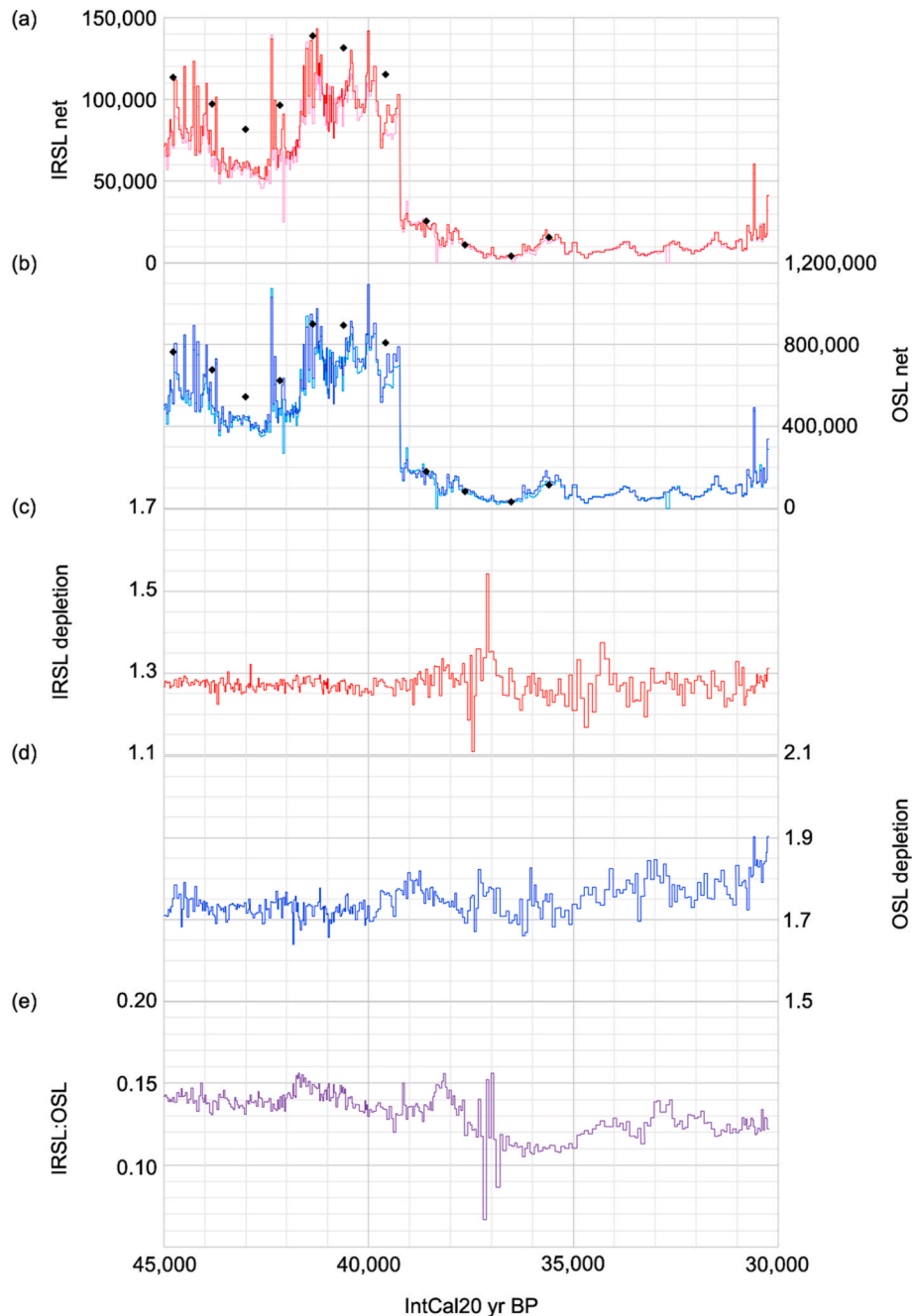


Fig. 4. ‘Natural’ (i.e., non-irradiated) portable luminescence (POSL) signals from Lake Suigetsu (SG12 sediment core) between 45,000 and 30,200 IntCal20 yr BP: (a) Net IRSL counts; (b) net OSL counts; (c) IRSL depletion index; (d) OSL depletion index; (e) IRSL:OSL ratio. Panels (a) and (b) include the repeat analyses (pink and light blue, respectively) in addition to the initial measurements (red and dark blue, respectively); the lower resolution, point sampling data produced by [Rex et al. \(2022\)](#) from parallel sediment core SG06 are also shown (black diamonds). To simplify the plots, only the initial measurements are shown on panels (c) to (e).

measurement. That being said, any such attenuation should be minimal and, indeed, this is borne out by the data obtained (see [Supplementary Table 1](#) and [Fig. 4a](#) and [b](#), below).

Having obtained these initial measurements, it was decided to additionally perform sensitivity analysis to further interrogate the 'raw' signals. Accordingly, material from the initial aliquots was redistributed on to 3 cm diameter aluminium planchettes and re-measured. It should be noted that a further slight attenuation of the photon counts would be expected. However, since the sediment was physically mixed, it is likely that the overwhelming majority of sediment transferred to the planchette would not have been previously light exposed (i.e., not at the surface for the original light exposure on the Petri dish), and so any such attenuation should again be minimal. The responses of the sediment on the planchettes would, however, be significantly reduced, compared to the Petri dish values, as a result of the reduced geometry. Following this re-measurement, the sediment on the planchettes was heated in a Buehler muffle furnace (stabilised at 350 °C for ≤ 60 min) to remove the natural signals ([Meldrum, 1996](#); [Francoz, 2023](#)). POSL measurement on a subset of samples confirmed that the signals had indeed been successfully zeroed (i.e., data statistically indistinguishable from the instrumental background 'empty chamber' measurements). A 10 Gy dose was subsequently applied to each sample using a ^{90}Sr beta radiation source, and the luminescence measurements again performed using the same CWproxy scheme on the POSL reader.

4. Results

The net IRSL and net OSL signals on the 'raw' Suigetsu sediment exhibit a range of noteworthy features ([Fig. 4a](#) and [b](#)). It should be emphasised that the repeat measurements are consistent with the initial measurements, supporting the assertion that the signals seen are genuine sedimentary features, rather than any artefact of the measurement process. This assertion is further supported by the fact that the structure seen by both net IRSL and net OSL signals is also very consistent.

Moreover, the agreement of our new, contiguous, high chronological resolution data with the \sim millennial spot sampling approach of [Rex et al. \(2022\)](#) provides further independent support for the integrity of the signals, with the respective data being obtained from separate sediment cores (SG12 versus SG06). This agreement between sediment cores is also testament to the integrity (lateral continuity) of the Suigetsu stratigraphy across the lake bed.

As previously noted by [Rex et al. \(2022\)](#), the contiguous sampling approach is advantageous in terms of more clearly demonstrating the authentic structure of the data. This structure includes features spanning different temporal durations. Firstly, there are high frequency maxima evident, consisting of single samples. We reiterate that, following our assertions above, we believe these signals to be genuine. It is likely that such samples include stochastic sediment inwash that differs from the longer-term, 'background' sedimentation, perhaps, and most likely, associated with the type of flood events identified by [Rex et al. \(2022\)](#) from the uppermost metre (the last ~ 500 years) of the Suigetsu stratigraphy. As noted in the Methods, we sought to avoid sampling such material; but, to exclude all such material, including smaller scale events, would be practically impossible to achieve. Although further investigation of such fine chronological resolution signal might prove fruitful, we do not discuss it any further in the present manuscript, preferring to focus on the longer-term (centennial to millennial) structure evident in the dataset.

The second feature of the data structure that we identify is the apparent millennial-scale periodicity evident in both net IRSL and net OSL signals, most clearly seen between circa 38,000 and 31,000 IntCal20 yr BP, with apparent maxima circa 35,650, 33,700, 32,550 and 31,500 IntCal20 yr BP, interspersed by apparent minima circa 36,850, 34,680, 33,230, 32,080 and 30,860 IntCal20 yr BP. This periodicity will be discussed further, below.

Thirdly, both net IRSL and OSL measurements exhibit a dramatic step-change at $39,242 \pm 60$ IntCal20 yr BP, with net counts of both indices decreasing by $\sim 75\%$ at this time ([Fig. 4a](#) and [b](#)). Such a stark transition can only be generated by an instantaneous shift in some fundamental characteristic of the sediment, and this feature of the data will also be discussed further below. We again draw attention to the fact that it is only with the contiguous sampling approach that such a step-change is able to be identified within the data, as opposed to the likely presumption of a relatively rapid (i.e., within the sampling resolution), yet steady decline in the luminescence characteristics if an alternative point sampling approach had been followed (c.f. [Rex et al., 2022](#)).

Both IRSL and OSL depletion indices remain relatively constant across the $\sim 14,800$ year study interval, although OSL depletion does trend slightly higher towards the younger end of the period ([Fig. 4c](#) and [d](#)). Unlike the net measurements, there is no step-change in these parameters at $39,242 \pm 60$ IntCal20 yr BP. Likewise, the IRSL:OSL ratio also does not demonstrate a step-change at this point in time, although there is more prominent structure in this parameter compared to the depletion indices ([Fig. 4e](#)); there is a general decreasing trend in IRSL:OSL across the full study period, particularly so between circa 41,700 and 35,300 IntCal20 yrs BP (i.e., continuing across the step-change in net counts). Post-stimulation phosphorescence (PSP; to both IR and blue light) is negligible across the entirety of the study interval ([Supplementary Fig. 2](#)).

5. Discussion

5.1. Analysis of the Lake Suigetsu sediment

As introduced above, a step-change in a data series requires a fundamental shift in the material under investigation. One such explanation for the sudden $\sim 75\%$ decrease in luminescence response in the present study at $39,242 \pm 60$ IntCal20 yr BP would be if there were a (lengthy) hiatus in the stratigraphy; however, a hiatus (longer than a handful of years) is not supported by the accompanying high resolution radiocarbon dataset from the site ([Bronk Ramsey et al., 2012, 2020](#); [Reimer et al., 2020](#)). Instead, we causally relate the transition observed here to the coincident macroscopic event layer ('EL-3107') that has previously been interpreted by [Schlolut et al. \(2014\)](#) as having been generated by an earthquake. This event layer marks a boundary between two sedimentary facies, with a higher proportion of detrital mineral grains prior to the transition. The interpretation of [Schlolut et al. \(2014\)](#) is that, prior to the earthquake, inflow into Suigetsu was not via Lake Mikata, as in the present day, but rather via an extended 'palaeo-Hasu' River into Lake Suga, which is effectively an extension of Suigetsu ([Fig. 1](#)). As a result of the earthquake blocking/uplifting the former river channel, inflow to Suigetsu switched to the current configuration of passing over the shallow sill (the Seto Channel) from Lake Mikata, with the latter acting as a filter for the coarser-grained detrital material. An analogous event is that of the historically recorded 1662 CE Kanbun earthquake, which resulted in uplift of the outflow channel from Suigetsu (the palaeo-Kiyama River) and necessitated construction of a new, artificial outflow from Suigetsu, the Urami Canal, to alleviate the resultant flooding around Suigetsu and Mikata. We emphasise that this latter event would not have affected sediment delivery to Suigetsu and, accordingly, [Rex et al. \(2022\)](#) demonstrated no observable effect on the luminescence characteristics of the sediment above and below the 1662 CE earthquake event (within period (1) of that study). As illustrated in [Fig. 4](#), the other luminescence characteristics (depletion indices, IRSL:OSL ratio and PSP) do not show a demonstrable coincident shift in values across EL-3107. This implies that, whilst the revised hydrological situation has resulted in the filtering out of coarser-grained material from reaching Suigetsu, the source of the sediment reaching Suigetsu remains fundamentally unchanged (i.e., the vast majority of material being transported from the

broader catchment upstream from Mikata), with sediment chemistry and luminescence bleaching histories of the transported minerals similarly not being affected by the earthquake.

Having accounted for this unique step-change in net luminescence signals at $39,242 \pm 60$ IntCal20 yr BP, as well as having posited an explanation for the highest frequency signals throughout the record (i.e., stochastic flooding events), we turn our attention to the longer term structure of the dataset. As noted above, this longer-term structure demonstrates millennial-scale periodicity in both net IRSL and net OSL signals, certainly following the earthquake (with maxima circa 35,650, 33,700, 32,550 and 31,500 IntCal20 yr BP) and, potentially, also evident in the preceding time period (a maximum circa 44,500 IntCal20 yr BP and either an extended maximum between circa 41,500 and 39,700 IntCal20 yr BP, or separate, closely spaced maxima circa 41,500, 40,400 and 40,000 IntCal20 yr BP; Fig. 4a and b).

To better understand the likely driver(s) of this periodicity, our follow-up ‘dosing’ experiment sought to explore whether the net signals were the result of varying sensitivity of the sediments or of varying dose received. From the repeat ‘natural’ measurements on the 3 cm diameter planchettes (Supplementary Figs. 3b and 4b) and, after zeroing, the responses of the same materials to a 10 Gy dose (Fig. 5b), we can calculate an estimated equivalent dose (ED_e) for each sample (based on linear extrapolation of the 10 Gy response; Supplementary Figs. 3d and 4d). Moreover, utilising the radiocarbon and varve-based chronology (Bronk Ramsey et al., 2020), we can calculate an estimated dose rate received (ED_R) for each sample (Fig. 5c). It can be seen that, broadly speaking, the millennial-scale periodicity is maintained through this ‘calibration’ process, but that the earthquake-induced step-change has been removed. The fact that the periodicity remains evident in the 10 Gy-dosed dataset (Fig. 5b) argues against variable bleaching history as being responsible for this pattern.

As described in the Introduction, there has been much palaeoenvironmental research already performed on the Lake Suigetsu sediments, and we can therefore compare our suite of luminescence data with that of various palaeoenvironmental proxies. Of the many elemental profiles produced by ultra high resolution ITRAX™ μ XRF analysis (Marshall et al., 2012), the best correlation with our luminescence (ED_R) data is provided by potassium (K) (Fig. 5d). Such a finding makes intuitive sense, since K indicates the presence of clays and/or K-feldspar which, elsewhere, may provide candidate minerals for luminescence dating (e.g., Smedley et al., 2012).

Further to this mineralogical comparison, we can also compare our luminescence data to palaeoclimatic reconstructions provided by palynological analysis of the Suigetsu sediments (Nakagawa et al., 2021; Nakagawa et al., unpublished data). There is a clear correlation with the pollen-inferred mean annual temperature (Fig. 5e; note the inverted y-axis), whereas there is no obvious correlation with pollen-inferred annual precipitation receipt (Fig. 5f). Finally, we also plot the Greenland ice-core (NGRIP) $\delta^{18}O$ data of Rasmussen et al. (2014) to place our present dataset within the context of North Atlantic palaeoclimate with which the readership may be more familiar (Fig. 5g). As with our (palynological) climate reconstructions from Suigetsu, the correlation between our luminescence data series and NGRIP $\delta^{18}O$ is readily apparent.

To identify such a clear relationship between mean annual temperature and the luminescence properties of a sedimentary stratigraphy may not have been anticipated *a priori*. The relationship is not direct but, rather, mediated via subtle differences in the composition of the sediment delivered to the lake bed under warmer or cooler climatic conditions, as supported by the correlation with K contribution. One might have imagined this correlation with K to have been driven by increased minerogenic input at times of greater precipitation (and therefore terrigenous inwash); however, this explanation is not supported by the pollen-inferred precipitation data. Rather, it is the pollen-inferred temperature data that better correlate and we therefore surmise that when the temperature was warmer, there was greater autochthonous organic

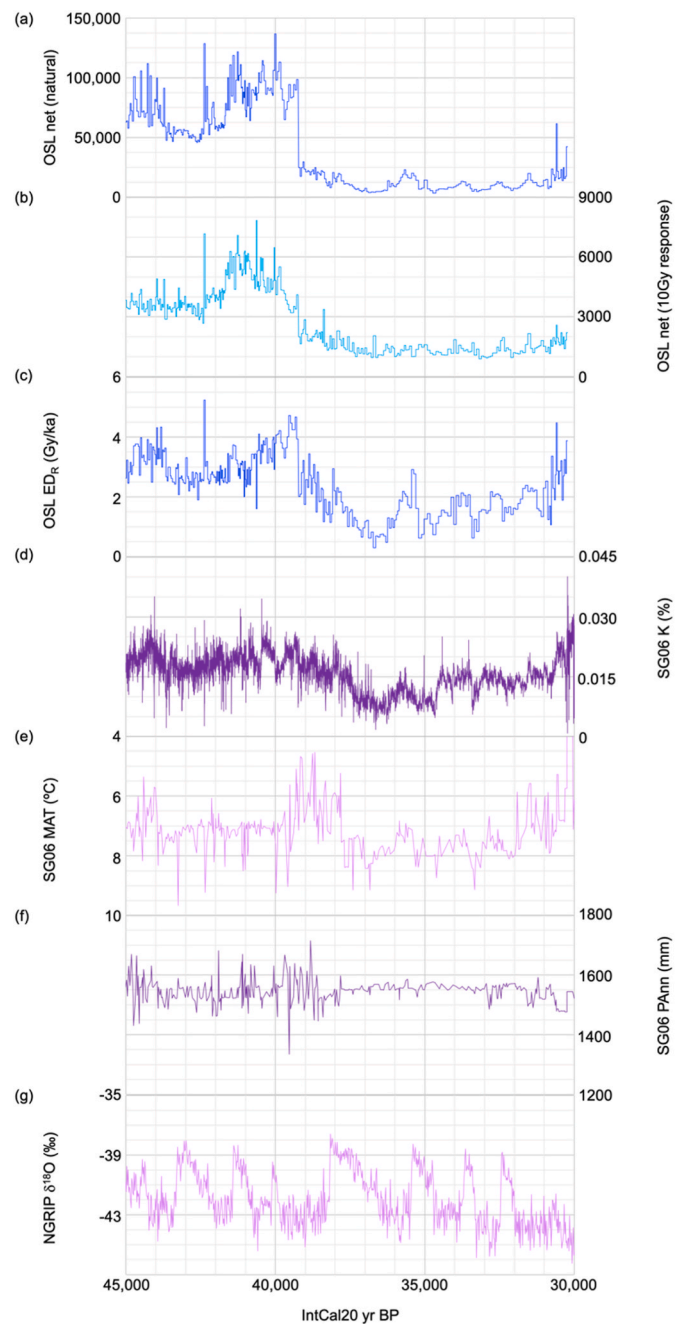


Fig. 5. (a) Net OSL counts from the ‘natural’ (i.e., non-irradiated) sediment from Lake Suigetsu (SG12 sediment core) between 45,000 and 30,200 IntCal20 yr BP (i.e., the same data as panel (b) of Fig. 4); (b) net OSL counts of samples irradiated with a 10 Gy dose (after zeroing); (c) estimated dose rate (ED_R); (d) SG06 K content derived by ITRAX™ μ XRF (Marshall et al., 2012); (e) SG06 palynologically-derived mean annual temperature (MAT; Nakagawa et al., unpublished data); (f) SG06 palynologically-derived mean annual precipitation (PAnn; Nakagawa et al., unpublished data); (g) NGRIP oxygen isotope data ($\delta^{18}O$; Rasmussen et al., 2014).

productivity and/or allochthonous inwash of organic matter which instead served to ‘dilute’ the luminescence signals borne by the sediments during such intervals.

5.2. Methodological considerations with respect to future application

Portable luminescence (POSL) readers of the type utilised for the present study have been operational for almost two decades (Sanderson

and Murphy, 2010). Their initial development was intended to facilitate rapid stratigraphic profiling during fieldwork in order to assist with better directed sampling strategies prior to sample collection and return for conventional laboratory OSL analysis (e.g., Kinnaird et al., 2012; Ghilardi et al., 2015). However, as demonstrated by our present study, they also allow rapid laboratory profiling, elucidating a detailed appreciation of luminescence ‘cryptostratigraphy’ (‘hidden stratigraphy’). Since measurement time for each sample is under 3 min (using the default CWproxy measurement scheme), extensive datasets can be rapidly produced, limited only by speed of sampling into Petri dishes (or on to planchettes). Even shorter measurement schemes could be implemented, but with a resultant reduction in measurement precision or range of parameters measured (e.g., if only blue light response were measured).

The rapidity of measurement also allows for the ready performance of replicate analyses. However, within the remit of the present study, measurement of true sample duplicates was not undertaken due to limitations on the amount of material available for analysis. Although a strength of the POSL technique is that it is non-destructive in the sense that material can be further analysed for other purposes, it is destructive in the sense that material is necessarily removed from its original core position within the sediment stratigraphy. In the present study, each sample was homogenised to try to ensure that a representative (true average) signal was obtained for each 3 cm-integrated sample, further ‘destroying’ the sediment. Nevertheless, such material would still be useable for other palaeoenvironmental analysis, so long as the same sampling interval were required, and we suggest that such considerations would be useful where POSL analysis might be used alongside similar multi-proxy palaeoenvironmental studies elsewhere in the future.

The amount of material used per sample in the present study; i.e., $\sim 2 \text{ cm}^3$ ($= 3 \text{ cm} \times 1 \text{ cm} \times 0.7 \text{ cm}$, as obtained from 1 cm width LL-channel sampling (Nakagawa et al., 2012), and removing $\sim 2\text{--}3 \text{ mm}$ from the surface of each sample to account for prior light exposure (Armitage and Pinder, 2017)) equates to a covering of $\sim 1 \text{ mm}$ depth across the surface of a 5 cm diameter Petri dish. This sampling approach (into Petri dishes) was chosen to maximise the surface area for measurement in the POSL reader to, as best as possible, mitigate against imperfect homogenisation of the sediment across the 3 cm sampling depth range. However, an alternative approach could have been to instead homogenise each sample as best as possible, and then to distribute material across two separate (e.g.) 3 cm diameter Petri dishes or planchettes and thus generate ‘true’ replicate measurements. Such a choice may be somewhat equivocal (in the sense that, with a finite amount of material to work with, measurement of a single aliquot from a larger surface area or two aliquots both with a reduced surface area will both mitigate against inhomogeneity within a sample), but is certainly worth considering by others if performing similar work with limited sediment availability in future.

In a similar vein, as a result of the evolution of the present study, we ultimately transferred sediment from the Petri dishes on to the smaller, 3 cm diameter planchettes anyway, both because the samples were then heated to 350°C , which the plastic Petri dishes cannot tolerate, and also because the beta source irradiator at SUERC would only accommodate the smaller planchettes. Had we anticipated performing this follow-up sensitivity analysis at the outset, we would have instead performed our initial analyses directly on the planchettes, saving time and effort, as well as presumably producing even more accurate equivalent dose and dose rate estimates.

6. Conclusion

The profile generated from the Lake Suigetsu sediments in the present study represents the largest contiguous (i.e., longest and most numerous) dataset of stimulated luminescence measurements produced to date; it comprises measurement of >300 samples spanning $\sim 14,800$

years (between 45,000 and 30,200 IntCal20 yr BP) in triplicate (quasi duplicate measurements upon 5 cm diameter Petri dishes, plus an additional ‘natural’ signal on a 3 cm diameter planchette), followed up with a further measurement after application of a 10 Gy dose. Such a sizeable dataset was enabled via use of the SUERC portable luminescence (POSL) reader, which enables rapid field- or, in this case, laboratory-based profiling. Our study was iterative in nature, with initial data generating ideas for follow up investigation and, hence, there were methodological adjustments that could have been made to improve efficiency of the work scheme yet further.

A cryptostratigraphy (‘hidden stratigraphy’) of the Lake Suigetsu sediments has been revealed, comprising luminescence-stimulated signals related to short-term, transient events (stochastic flooding in the catchment), longer-term (\sim millennial scale) periodicity correlated to (pollen-derived) mean annual temperature, and an earthquake-induced permanent change to catchment hydrology.

Our study illustrates the power of POSL, demonstrating the additional information that can be gleaned as increasingly rigorous analyses are performed, for example, moving from lower chronological resolution point sampling to higher resolution, contiguous sampling, and supplementing the raw, ‘natural’ signals with responses to a prescribed artificial dose. We emphasise that in the present study, for which we already have a genuinely world class radiocarbon and varve-based chronology, the luminescence-stimulated signals produced were interrogated solely as a proxy of palaeoenvironmental change, but there is no reason why such a methodology could not be combined with even more rigorous protocols to supplement OSL dating efforts elsewhere and, indeed, to help to guide those efforts (i.e., to optimise dating sampling strategy) in the first place.

CRediT authorship contribution statement

Richard A. Staff: Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Visualization, Writing – original draft, Writing – review & editing. **David C.W. Sanderson:** Conceptualization, Funding acquisition, Investigation, Methodology, Resources, Supervision, Writing – review & editing. **Charlie L. Rex:** Conceptualization, Formal analysis, Funding acquisition, Investigation, Methodology, Visualization, Writing – review & editing. **Alan Cresswell:** Investigation, Methodology, Resources, Supervision, Writing – review & editing. **Masayuki Hyodo:** Conceptualization, Funding acquisition, Investigation, Writing – review & editing. **Ikuko Kitaba:** Investigation, Writing – review & editing. **Michael H. Marshall:** Formal analysis, Investigation, Writing – review & editing. **Gordon Scholout:** Formal analysis, Investigation, Writing – review & editing. **Keitaro Yamada:** Investigation, Writing – review & editing. **Yoshiaki Suzuki:** Investigation, Writing – review & editing. **Vanessa Nowinski:** Formal analysis, Investigation, Writing – review & editing. **Ryuji Tada:** Investigation, Project administration, Resources, Writing – review & editing. **Takeshi Nakagawa:** Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Software, Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Richard Staff, Charlie Rex, David Sanderson, Masayuki Hyodo, Takeshi Nakagawa, reports financial support was provided by Daiwa Anglo-Japanese Foundation. Charlie Rex reports financial support was provided by Natural Environment Research Council. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.quageo.2024.101588>.

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