

Chapter 11

Comparison of Luminescence Dating Methods on Lake Sediments from a Small Catchment: Example from Lake Yogo, Japan

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Abstract When applying luminescence dating to sediment deposited in aquatic environments, a key issue for accurate age determination is resetting of acquired luminescence in sediment by surface exposure (bleaching) before burial. The time

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needed for bleaching is known to vary among the signals used in three methods: optically stimulated luminescence (OSL), infrared stimulated luminescence (IRSL), and post-infrared IRSL (pIRIR). A comparison of luminescence ages from these different signals is therefore useful to assess whether a sample was fully bleached before burial. In a comparison of OSL, IRSL_{50/225} and pIRIR₂₂₅ ages of eight samples of fine-grained sediment from a 294-cm-long sediment core from Lake Yogo, a small-catchment lake in central Japan, the IRSL_{50/225} and pIRIR₂₂₅ ages were much older than the OSL ages. The IRSL_{50/225} residual signals were close to zero, and the difference between pIRIR₂₂₅ and OSL signals was much larger than the pIRIR₂₂₅ residual signals. Thus, IRSL_{50/225} and pIRIR₂₂₅ signals were not completely bleached, which we attribute to the short sediment transport distance in this small catchment. Five corrected bulk radiocarbon (¹⁴C) ages agreed with the OSL ages, except for two intervals in which OSL ages were about 500 and 1,900 years older than the corrected ¹⁴C ages. These discrepancies are attributable to incomplete bleaching related to sediment transport, whereas the rest of the OSL ages show no evidence of incomplete bleaching. This study shows that even in samples in which the pIRIR₂₂₅ and IRSL_{50/225} signals are not well-bleached, OSL dating yields accurate age estimates because of the faster bleaching rate.

Keywords OSL dating • Post-IR IRSL dating • Bulk ¹⁴C dating • Lake sediments • Incomplete bleaching

11.1 Introduction

Lake sediments contribute to our understanding of past climate change in terrestrial areas (e.g., Colman et al. 1995; Williams et al. 1997). Depth profiles of lake sediment can provide proxy records that represent paleoenvironments (e.g., BDP-98 Members 2001; BDP-99 Members 2005). The determination of sedimentation age is critical for converting depth profiles of proxies to age profiles for reconstruction of paleoclimatic fluctuations. Radiocarbon (¹⁴C) dating is typically used for age determination of lake sediments for ages up to about 40 ka (e.g., Nara et al. 2005; Watanabe et al. 2007, 2009). Optically stimulated luminescence (OSL) and infrared stimulated luminescence (IRSL) dating methods also have the potential to date the depositional age of sediments when the acquired luminescence of sediment entering the lake is reset by sunlight (bleached) during its transport from the catchment area to the basin. The age range of OSL and IRSL dating is from the present to about 200 ka, and these methods have been successfully applied to lake sediments (e.g., Juschus et al. 2007; Zheng et al. 2010). Buylaert et al. (2013) successfully applied another luminescence method, post-IR infrared stimulated luminescence (pIRIR) dating, to K-feldspar in lake sediments older than about 70 ka. However, Zheng et al. (2010) showed that OSL ages of lake sediment younger than about 10 ka had been overestimated.

Murray et al. (2012) proposed an approach to assess the degree of bleaching based on the different bleaching rates for OSL and pIRIR signals: if feldspar pIRIR

ages are close to quartz OSL ages, then the quartz must be completely bleached because the bleaching rate of the pIRIR signal in feldspar is much slower than that of the OSL signal in quartz. Their approach identifies well-bleached quartz independently of other age controls.

Before AD 1604, Lake Yogo was a small enclosed lake with a catchment area of 7.97 km². At that time, it was artificially connected to the Yogo River and its catchment area expanded to 35.37 km². The original Lake Yogo catchment is underlain by alluvial sediment and rocks of a Jurassic accretionary wedge, and it contains no volcanic rocks (Wakita et al. 1992). In applying OSL dating to this sediment, incomplete bleaching during sediment transport presents a significant potential problem because the initial catchment area was very small and the sediment may have traveled short distances.

This study tests the proposed approach of Murray et al. (2012) on lake sediment from a small catchment by using samples from Lake Yogo. As was done by Murray et al. (2012), we determined IRSL and pIRIR ages in addition to OSL ages. We also determined ¹⁴C ages from plant residue and the bulk sediment to provide independent age controls. We then compared luminescence and radiocarbon ages to assess the effectiveness of OSL dating of the Lake Yogo sediment core, and the validity of the approach of Murray et al. (2012) for dating lacustrine sediments from a small catchment.

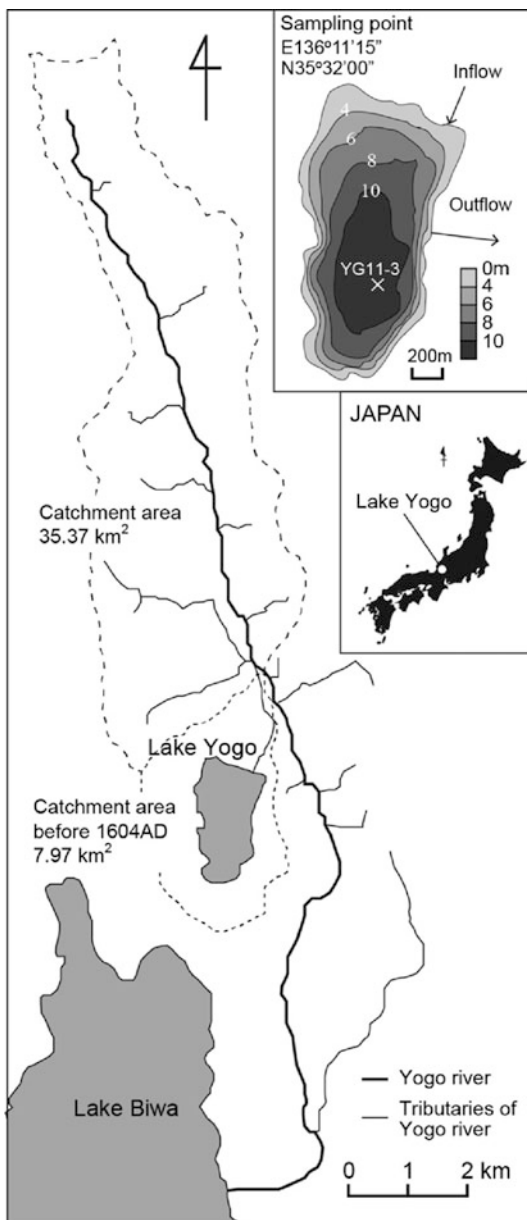
11.2 Sample and Methods

Samples for luminescence and ¹⁴C dating were taken from a 294-cm-long sediment core (YG11-3) from Lake Yogo. The core, which was obtained from the deepest part of the lake (12.5 m) by piston coring (Fig. 11.1), is composed of homogeneous brown silty clay without any distinctive features. Under subdued red light, samples were collected at 2-cm core intervals, and each sample was split in two. The first split was dried at 110 °C for 2 days and then used for measurement of water content and radioisotope concentration. The other split was not dried and was used for luminescence measurement. Plant residues were sampled from core depths of 182 and 278 cm.

11.2.1 Luminescence Dating

For luminescence measurement, wet samples were chemically treated at room temperature using 10 % hydrogen chloride to remove carbonate and then 10 % hydrogen peroxide to remove organic matter. Then, the 4–11 μm sediment size fractions were extracted by a method based on Stokes' law by suspending the samples in a sodium oxalate solution. Portions of these polymineral fractions

Fig. 11.1 Map showing the location of Lake Yogo and the sampling point. Inflow and outflow are through an artificial headrace



were used for IRSL and pIRIR dating, and the remaining fractions were further treated with 10 % hexafluorosilicic acid to remove feldspar and isolate the quartz fraction. The degree of contamination of the quartz fraction by feldspar, checked by observing IRSL emissions, was less than 1 % in all samples. The quartz fraction was

Table 11.1 SAR protocols for D_e measurements

Step	OSL	IRSL _{50/225} and pIRIR ₂₂₅
1	Given dose	Given dose
2	Preheat for 10 s at 200 °C	Preheat for 60 s at 250 °C
3	OSL for 40 s at 125 °C (L_x)	IRSL for 200 s at 50 °C (L_x for IRSL _{50/225})
4	–	IRSL for 200 s at 225 °C (L_x for pIRIR ₂₂₅)
5	Given test dose	Given test dose
6	Preheat for 10 s at 160 °C	Preheat for 60 s at 250 °C
7	OSL for 40 s at 125 °C (T_x)	IRSL for 200 s at 50 °C (T_x for IRSL _{50/225})
8	–	IRSL for 200 s at 225 °C (T_x for pIRIR ₂₂₅)
9	Hot bleach for 40 s at 280 °C	Hot bleach for 200 s at 255 °C
10	Return to step 1	Return to step 1

then dated by OSL. The dried polymineral and quartz fractions (about 1.0 mg each) were suspended in acetone and placed on stainless steel disks 10 mm in diameter, after which the acetone was evaporated in an oven.

The luminescence signal was measured using a luminescence reader (DA-20, Risø) equipped with a $^{90}\text{Sr}/^{90}\text{Y}$ beta source at Geological Survey of Japan, AIST. Luminescence emissions in the ultraviolet region were measured through a Hoya U-340 filter for quartz OSL, and emissions in the blue-violet region were measured through Schott BG3, BG39, and GG400 filters, in ascending order, for polymineral IRSL and pIRIR. The single aliquot regenerative (SAR) protocol was applied to the determination of the equivalent dose D_e (Table 11.1; Murray and Wintle 2000). The OSL signal was measured for 40 s at 125 °C. The IRSL signal was measured for 200 s at 50 °C (IRSL_{50/225}), followed by pIRIR measurement for 200 s at 225 °C (pIRIR₂₂₅). The luminescence signal was sampled every 0.1 s. The luminescence intensities were derived from the integral of the first 0.5 s (5 channels) and 2.0 s (20 channels) after subtracting the last 5 s for the OSL decay curve and the last 10 s for the IRSL and pIRIR decay curves. Radioisotope concentrations of each sample were measured by laser-ablation inductively coupled plasma mass spectrometry and x-ray fluorescence (Ito et al. 2009, 2011) and converted into annual dose rates by the method of Adamiec and Aitken (1998). Cosmic dose rate was estimated following Prescott and Hutton (1994).

11.2.2 ^{14}C Dating

^{14}C dating of bulk sediment samples followed the protocol of Watanabe et al. (2009). Dried sediment was powdered, and then treated with 1.2 M HCl to remove carbonate. The sample was then combusted at 850 °C for 4 h with CuO wire, and the

resulting CO₂ was purified with ethanol, *n*-pentane, and liquid nitrogen in a vacuum line. The CO₂ was reduced to graphite by heating with iron and hydrogen at 650 °C for 6 h.

The procedure for ¹⁴C dating of plant samples was after Nakamura et al. (2003). Small pieces of leaves were picked from the bulk sediment sample under the microscope and washed with distilled water to remove mineral grains. The leaves were treated with 1.2 M HCl and 1.2 M NaOH (acid-alkali-acid treatment), dried, and combusted. The resulting CO₂ was purified and reduced to graphite by the same procedure used for the bulk sediment sample.

The ¹⁴C/¹²C ratio of graphite was measured with a Tandemtron accelerator mass spectrometer (Model-4130, HVEE) at the Center for Chronological Research, Nagoya University. The measured ratio was corrected by δ¹³C, which was measured with an isotope ratio mass spectrometer (MAT-252, Thermo Finnigan). All ¹⁴C ages were converted to calendar years using the INTCAL09 data set (Reimer et al. 2009).

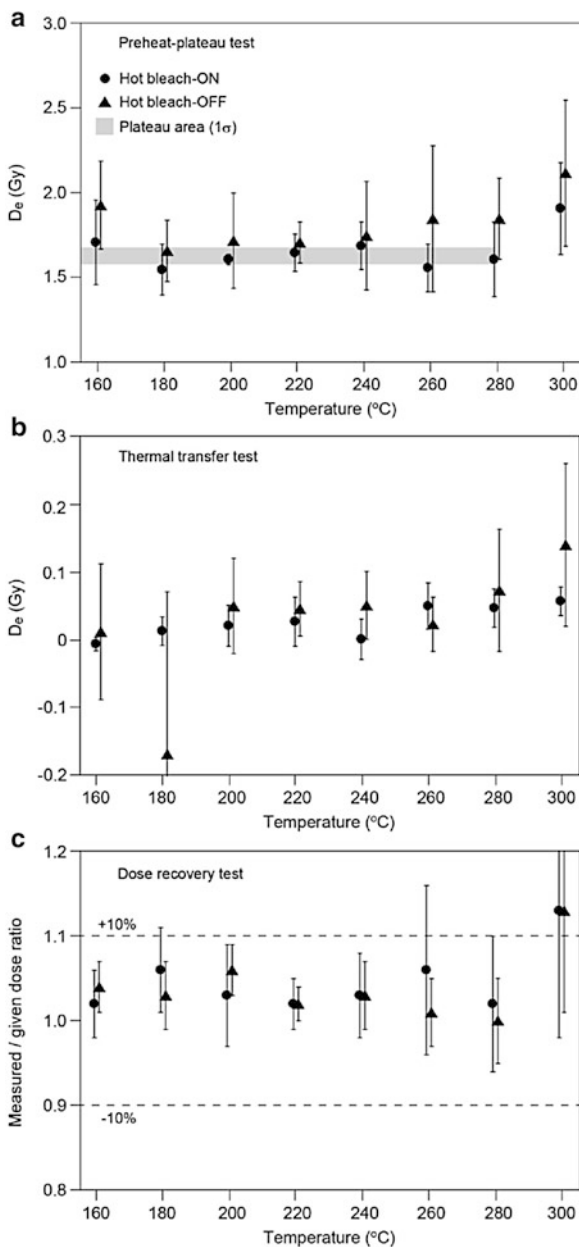
11.3 Results

11.3.1 OSL Dating

Quartz samples from Lake Yogo sediments exhibited OSL signals with a strong fast component (Fig. 11.2a), and the recycling ratio was close to unity. These samples thus differ from the volcanic quartz found elsewhere in Japan (e.g., Tsukamoto et al. 2003), which is characterized by anomalous fading and dominance of the slow component. Results of the preheat-plateau test, thermal transfer test, and dose recovery test for OSL analysis are presented in Fig. 11.3. The influence of hot bleaching was also investigated for these three tests, and the data with and without hot bleaching agreed within the standard error. The preheat-plateau test showed a plateau of D_e around 1.6 Gy over the preheat temperature range from 160 to 280 °C (Fig. 11.3a). The thermal transfer was almost negligible at all preheat temperatures except for 300 °C (Fig. 11.3b).

For the dose recovery test, the natural sample was initially bleached using a blue LED to reduce the natural OSL to almost the background level at room temperature. After bleaching, the sample was given a beta dose of approximately 2.0 Gy, which is close to the natural D_e , and then OSL was measured using preheat temperatures from 160 to 300 °C (Fig. 11.3c). The dose recovery ratio was close to unity at preheat temperatures from 160 to 280 °C. A preheat temperature of 200 °C for 10 s and a cut heat of 160 °C were chosen for all of our OSL measurements. Dose recovery tests were carried out on every sample at the preheat temperature of 200 °C, and the measured dose/given dose ratio was close to unity (1.01–1.03). The mean D_e values of repeated OSL measurements were determined by the central age model of Galbraith et al. (1999) and are shown in Table 11.3. Aliquots were accepted for mean D_e determination only if they yielded a recycling ratio within 1.0 ± 0.1 .

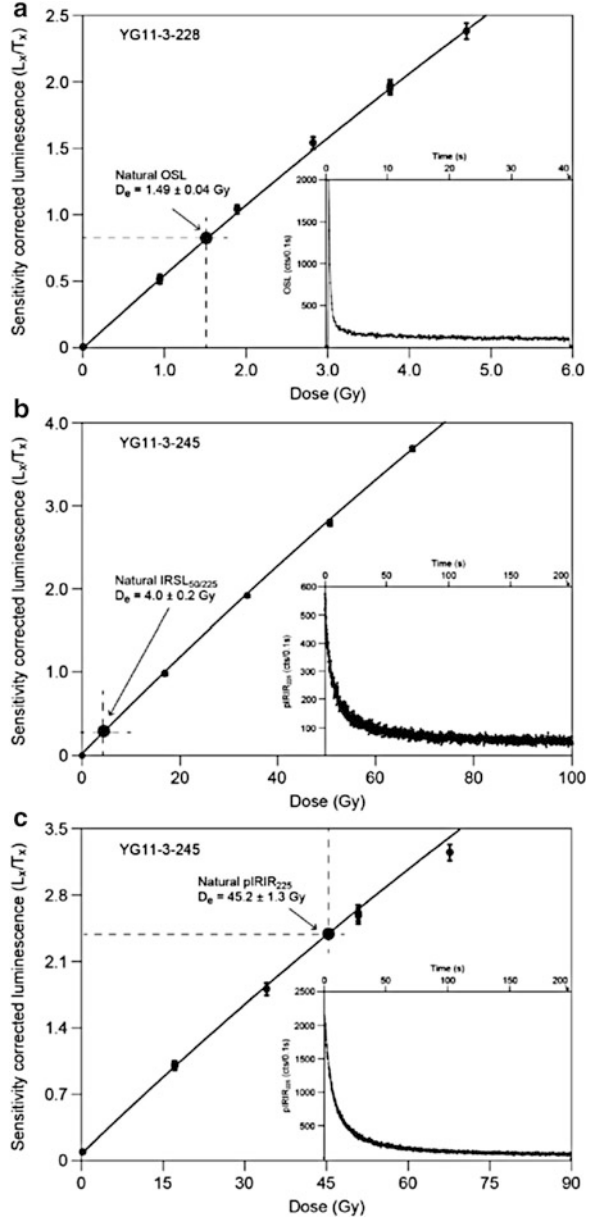
Fig. 11.2 Typical dose response curve for (a) OSL, (b) IRSL_{50/225}, and (c) pIRIR₂₂₅, together with decay curves (inset). A single exponential function was fitted to the dose–response curves



11.3.2 IRSL_{50/225} and pIRIR₂₂₅ Dating

Examples of polymineral IRSL_{50/225} and pIRIR₂₂₅ decay curves are shown in Fig. 11.2b, c. Unbleachable residual signals of IRSL_{50/225} and pIRIR₂₂₅ were estimated in parallel with the D_e measurement and dose recovery test at a preheat

Fig. 11.3 Results of the (a) preheat-plateau test, (b) thermal transfer test, and (c) dose recovery test for OSL analysis on sample YG11-3-316. Each test was conducted with and without hot bleaches



temperature of 250 °C, following the protocol of Lowick et al. (2012). After the samples were exposed to natural sunlight for 2 days, the apparent residual doses were measured and plotted against the measured natural dose (Fig. 11.4). Although the residual dose of IRSL_{50/225} was negligible, the pIRIR₂₂₅ signal contained a residual that was positively correlated with D_e . The residual dose was thus

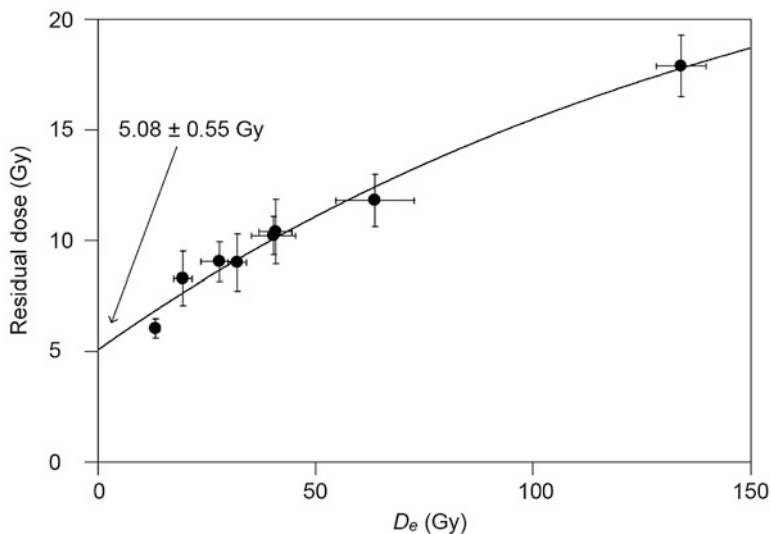


Fig. 11.4 The pIRIR₂₂₅ residual dose in the samples. The errors represent one standard error

calculated with an intercept value at $D_e = 0$ after the method proposed by Sohbati et al. (2011). The residual dose of 5.08 ± 0.55 Gy was subtracted from all D_e values of pIRIR₂₂₅. Dose recovery tests were carried out on three samples (YG11-3-124, -245, and -349) at the preheat temperature of 250 °C, and the dose recovery ratio was close to unity (1.03–1.10 for IRSL_{50/225}, 0.95–1.08 for pIRIR₂₂₅). D_e values of five aliquots were determined for both IRSL_{50/225} and pIRIR₂₂₅, and their mean was used for final age determinations (Table 11.2).

The stability of the pIRIR₂₂₅ signal was checked by estimating the fading rate (*g*-value) on every aliquot, which was measured and calculated following the conventional procedure (Huntley and Lamothe 2001; Auclair et al. 2003) and expressed as the percentage of luminescence decay in a decade. Most *g*-values for IRSL_{50/225} and pIRIR₂₂₅ were 2.5–3.4 %/decade and smaller than 1.5 %/decade, respectively (Table 11.2). These *g*-values are consistent with the understanding that pIRIR₂₂₅ signals are more stable than conventional IRSL signals (Thomsen et al. 2008; Buylaert et al. 2009; Thiel et al. 2010). A fading correction was not done for pIRIR signals, as Buylaert et al. (2012) suggested that samples with relatively low pIRIR₂₉₀ $g_{2\text{days}}$ (1.44 %/decade) do not require the correction. To confirm that fading was negligible, it would be preferable to measure saturated samples (Buylaert et al. 2011). However, samples of that age were not available in the Lake Yogo sediment core. Therefore, we show pIRIR ages both uncorrected and corrected for fading. For IRSL_{50/225} dating, we used a fading correction because of the higher *g*-values.

Radioisotope concentrations are shown in Table 11.3. The alpha dose rates for fine-grained quartz and fine-grained polymineral samples were calculated using *a*-values of 0.04 and 0.08, respectively (Rees-Jones 1995). The water content of the

Table 11.2 Values of D_e and fading rate (g-value) for fine-grained quartz OSL and for fine-grained polymineral IRSL_{50/225} and pIRIR₂₂₅

Sample name	Depth (cm)	Lab. code	OSL				IRSL _{50/225}				pIRIR ₂₂₅	
			n ^{1a}	n ^{2a}	D_e (Gy)	OD ^b (%)	n	D_e (Gy)	g _{24days} ^c (%/decade)	n	D_e^d (Gy)	g _{24days} ^c (%/decade)
YG11-3-001	0	gsj13-98	23	21	0.30 ± 0.01	14	5	3.60 ± 0.21	2.62 ± 0.20	5	22.7 ± 4.1	0.88 ± 0.38
YG11-3-124	56	gsj13-99	25	21	0.42 ± 0.01	12	5	2.10 ± 0.16	2.53 ± 0.63	5	8.0 ± 0.8	0.77 ± 0.20
YG11-3-207	106	gsj13-100	21	20	0.55 ± 0.01	8	5	2.74 ± 0.16	2.87 ± 0.20	5	14.3 ± 2.6	0.67 ± 0.18
YG11-3-228	148	gsj13-101	22	21	1.51 ± 0.01	2	5	6.39 ± 0.37	3.31 ± 0.53	5	58.5 ± 10.2	1.03 ± 0.25
YG11-3-245	182	gsj13-102	21	19	1.18 ± 0.01	4	5	4.21 ± 0.13	3.17 ± 0.43	5	35.2 ± 5.3	1.25 ± 0.25
YG11-3-316	224	gsj13-103	38	28	1.61 ± 0.04	8	5	3.77 ± 0.12	3.11 ± 0.76	5	26.8 ± 4.3	0.96 ± 0.34
YG11-3-343	278	gsj13-104	25	21	3.50 ± 0.08	10	5	25.66 ± 3.46	3.14 ± 0.42	5	129.1 ± 11.4	1.37 ± 0.37
YG11-3-349	290	gsj13-105	22	20	2.78 ± 0.02	2	5	5.76 ± 0.27	3.35 ± 0.47	5	35.7 ± 6.0	1.16 ± 0.39

Note: Errors represent one standard error

^a“n1” and “n2” represent number of measured and accepted aliquots, respectively

^bOD over dispersion

^cFor each aliquot used for D_e measurement, fading test was also performed

^dThe D_e values of pIRIR₂₂₅ have a residual dose of 5.08 ± 0.55 Gy subtracted from the natural D_e values

Table 11.3 Concentration of radioisotopes from LA-ICP-MS analysis, water contents, cosmic ray and annual dose

Sample name	Depth (cm)	Lab. code	²³⁸ U (μg/g)	²³⁴ U (μg/g)	²³² Th (μg/g)	⁸⁷ Rb (μg/g)	Water content ^a (wt.%)	Cosmic ray (mGy/year)	Annual dose for quartz (mGy/year)	Annual dose for polycrystalline (mGy/year)
YG11-3-001	0	gsj13-98	2.05 ± 0.12	(1.12 ± 0.21) × 10 ⁻⁴	7.98 ± 0.88	20.26 ± 0.23	423	0.1	0.59 ± 0.06	0.65 ± 0.07
YG11-3-124	56	gsj13-99	1.89 ± 0.14	(1.56 ± 0.15) × 10 ⁻⁴	9.27 ± 1.94	21.74 ± 1.08	285	0.1	0.81 ± 0.10	0.92 ± 0.11
YG11-3-207	106	gsj13-100	1.59 ± 0.12	(1.12 ± 0.06) × 10 ⁻⁴	6.42 ± 0.66	19.31 ± 0.64	342	0.1	0.57 ± 0.05	0.64 ± 0.05
YG11-3-228	148	gsj13-101	2.16 ± 0.18	(1.41 ± 0.39) × 10 ⁻⁴	9.77 ± 1.50	28.01 ± 1.50	239	0.1	1.03 ± 0.13	1.15 ± 0.16
YG11-3-245	182	gsj13-102	1.46 ± 0.08	(0.69 ± 0.24) × 10 ⁻⁴	5.65 ± 0.25	22.19 ± 1.00	369	0.1	0.49 ± 0.05	0.53 ± 0.06
YG11-3-316	224	gsj13-103	1.78 ± 0.15	(1.35 ± 0.29) × 10 ⁻⁴	7.28 ± 0.29	22.37 ± 0.07	318	0.1	0.68 ± 0.06	0.76 ± 0.08
YG11-3-343	278	gsj13-104	1.91 ± 0.15	(1.20 ± 0.13) × 10 ⁻⁴	8.03 ± 0.55	23.57 ± 0.20	299	0.1	0.75 ± 0.06	0.84 ± 0.07
YG11-3-349	290	gsj13-105	2.01 ± 0.26	(1.34 ± 0.10) × 10 ⁻⁴	7.99 ± 1.23	28.36 ± 0.98	280	0.1	0.91 ± 0.10	1.00 ± 0.11

Note: The errors represent one standard error

^aWater content is calculated by the model assuming the compaction

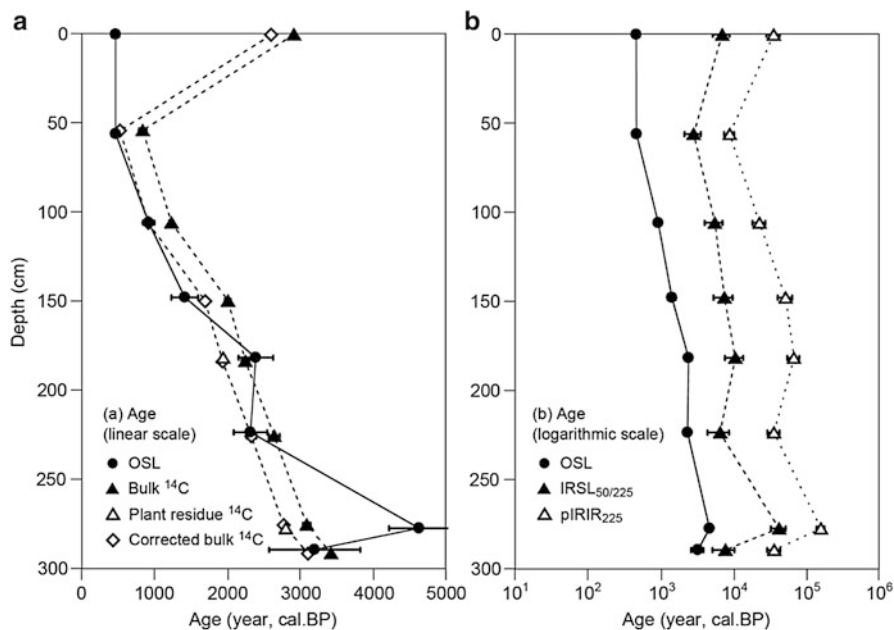


Fig. 11.5 Depth profiles of the Lake Yogo core showing (a) OSL, bulk ^{14}C , plant ^{14}C , and corrected bulk ^{14}C ages and (b) OSL, fading-corrected IRSL_{50/225}, and fading-uncorrected pIRIR₂₂₅ ages. The errors represent one standard error

samples decreased with increasing depth in the sediment core. We thus assumed an average water content for each sample as the mean between the water content of lake-floor sample YG11-3-001 and that of the sample. This modified water content (Table 11.3) was used for age determination. The calculated ages are shown in Table 11.4 and Fig. 11.5.

11.3.3 ^{14}C Dating

The bulk ^{14}C ages of samples YG11-3-246 and YG11-3-342 were generally about 300 years older than the plant residue ^{14}C ages of the adjoining samples YG11-3-245 and YG11-3-343 (Table 11.4). This overestimation is ascribed to the well-known old-carbon effect (e.g., Watanabe et al. 2009). We assumed that 300 years is the average overestimation of the bulk ^{14}C ages and subtracted it to yield the corrected bulk ^{14}C ages, which were compared with the OSL ages in this study.

Table 11.4 The $\delta^{13}\text{C}$ values and the ages for bulk ^{14}C , plant residue ^{14}C , quartz OSL, fading corrected $\text{IRSL}_{50/225}$, and uncorrected and corrected pIRIR_{225} ages

Sample name	Depth (cm)	Lab. code	Material for ^{14}C dating	$\delta^{13}\text{C}$ (‰)	^{14}C age (BP)	Calibrated ^{14}C age (cal.BP)	Lab. code	OSL		pIRIR ₂₂₅	
								Age (y)	Corrected age (y)	Uncorrected age (y)	Corrected age (y)
YG11-3-001	0	NUTA2-19999	Sediment	-26.9	2,800 ± 20	2,900 ± 30	gsj13-98	520 ± 50	6,860 ± 1,760	378,000 ± 7,240	37,580 ± 7,930
YG11-3-123	54	NUTA2-20000	Sediment	-26.1	895 ± 20	830 ± 50					
YG11-3-124	56						gsj13-99	520 ± 60	2,790 ± 690	8,680 ± 1,400	9,250 ± 1,500
YG11-3-207	106	NUTA2-20001	Sediment	-28.2	1,270 ± 20	1,220 ± 30	gsj13-100	970 ± 80	5,440 ± 1,480	22,380 ± 4,520	23,700 ± 4,800
YG11-3-228	148						gsj13-101	1,470 ± 190	7,340 ± 2,130	50,850 ± 11,280	55,740 ± 12,440
YG11-3-229	150	NUTA2-21289	Sediment	-28.1	2,040 ± 20	2,000 ± 40					
YG11-3-245	182	NUTA2-20010	Plant residue	-32.8 ^a	1,985 ± 25	1,930 ± 30	gsj13-102	2,440 ± 240	10,360 ± 2,900	65,960 ± 12,170	73,960 ± 13,780
YG11-3-246	184	NUTA2-20003	Sediment	-29.2	2,235 ± 20	2,235 ± 50					

(continued)

Table 11.4 (continued)

Sample name	Depth (cm)	Lab. code	Material for ^{14}C dating	$\delta^{13}\text{C}$ (‰)	^{14}C age (BP)	Calibrated ^{14}C age (cal.BP)	Lab. code	OSL		IRSL _{50/225}		pIRIR ₂₂₅	
								Age (y)	Corrected age (y)	Corrected age (y)	Uncorrected age (y)	Uncorrected age (y)	Corrected age (y)
YG11-3-316	224						gsj13-103	2,370 ± 230	6,430 ± 2,110	6,430 ± 2,110	35,240 ± 6,630	38,330 ± 7,310	
YG11-3-317	226	NUTA2-20007	Sediment	-29.4	2,530 ± 20	2,630 ± 75							
YG11-3-342	276	NUTA2-20008	Sediment	-29.1	2,925 ± 20	3,075 ± 50							
YG11-3-343	278	NUTA2-20011	Plant residue	-30.5 ^a	2,690 ± 20	2,790 ± 25	gsj13-104	4,680 ± 420	41,170 ± 9,330	41,170 ± 9,330	154,500 ± 19,060	175,940 ± 22,740	
YG11-3-349	290						gsj13-105	3,060 ± 330	7,610 ± 2,540	7,610 ± 2,540	35,500 ± 7,080	39,340 ± 7,970	
YG11-3-350	292	NUTA2-20009	Sediment	-28.7	3,185 ± 20	3,410 ± 25							

Note: The errors represent one standard error

^aThe $\delta^{13}\text{C}$ value of plant residue was measured by Tandemron Accelerator Mass Spectrometry

11.4 Discussion

The IRSL_{50/225} and pIRIR₂₂₅ ages were much older than the OSL ages (Table 11.4), and the residual could not account for the excess. The slower bleaching rates for IRSL and pIRIR signals thus appear to have contributed to the excess in equivalent dose. Lake Yogo has a small catchment characterized by a short sediment transport distance, which would provide less opportunity for bleaching by sunlight and result in incomplete bleaching of IRSL and pIRIR signals. Nevertheless, except for samples YG11-3-001, YG11-3-245, and YG11-3-343, five OSL ages were concordant with corrected bulk ¹⁴C ages, suggesting that there was generally enough sunlight exposure to reset the OSL signal. The IRSL signal also showed evidence of more complete bleaching than the pIRIR signal.

With regard to the three exceptional samples, there are two possible explanations for why the OSL ages were not concordant with the corrected bulk ¹⁴C ages. First, for YG11-3-245 and YG11-3-343, the OSL ages were probably older than the corrected bulk ¹⁴C ages because of incomplete bleaching. Mass movements such as river floods and landslides may lead to less exposure to sunlight and account for the poor bleaching of OSL. These two samples showed a larger difference between OSL and pIRIR ages than other samples, which is consistent with a short exposure to sunlight because the difference between the OSL and pIRIR residual signals is large for bleaching times of only a few seconds (Murray et al. 2012). In other samples, quartz was well bleached for OSL dating before deposition and gave reliable ages concordant with ¹⁴C ages. Second, for sample YG11-3-001, from near the top of the core (8 cm depth), both OSL and corrected bulk ¹⁴C ages may be overestimated. After 1960, the water balance of Lake Yogo came under artificial control for irrigation and flood-control purposes (Shimada et al. 2002); thus, changes in the organic carbon source and transport processes may have resulted in overestimated corrected bulk ¹⁴C and OSL ages. The presence of radioactivity from ¹³⁷Cs (42.5 ± 5.7 Bq/kg) is evidence for the deposition of YG11-3-001 after 1960.

For coarse-grained sediments, the effects of bleaching on age determinations are best evaluated by the single-grain method and corresponding age models (such as MAM, Galbraith et al. 1999). However, for fine-grained sediments, the methods used in this study are most suitable. On the other hand, comparisons of quartz OSL and pIRIR ages (Murray et al. 2012) may be ineffective in establishing the accuracy of OSL ages when the OSL signal is completely reset but IRSL and pIRIR signals are not fully reset, because of the difference in bleaching rates for the three signals. In that situation, one solution is to compare luminescence ages with an independent age control, and this study using sediment from Lake Yogo is an example of that approach's success. Laboratory studies suggest that sun exposure of no more than a few tens of seconds is enough to perturb OSL age estimates (Murray et al. 2012). If bleaching rates of OSL, IRSL, and pIRIR signals are accurately known, these may be used to constrain the approximate time of sediment's sunlight exposure before burial. Although OSL is the most reliable technique for age determination of

young sediment, the degree of sunlight exposure in a given sediment system may be important for estimating effects of incomplete bleaching when applying the pIRIR method to much older sediments.

11.5 Summary

1. The IRSL_{50/225} and pIRIR₂₂₅ ages were much older than the OSL ages in samples from Lake Yogo, whereas the OSL ages were generally concordant with the corrected bulk ¹⁴C ages. The difference in bleaching rates of these three luminescence signals appears to have contributed to these age differences. In the case of Lake Yogo, there was enough sunlight exposure to mainly reset the OSL signal but not enough to reset the IRSL and pIRIR signals. Therefore, the bleaching period and conditions are key issues for accurate age determination of samples from small catchment areas.
2. For incompletely bleached samples, comparisons between OSL and independent age controls may be more reliable than comparisons between different luminescence techniques. However, overestimated ages may be useful to constrain the approximate time of sunlight exposure before burial, based on the results of laboratory bleaching studies. For old samples, incomplete bleaching of the pIRIR signal is negligibly small compared to D_e and the pIRIR method may be used to determine the sedimentation age.

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