AMS ^{14}C DATING OF VARVED SEDIMENTS FROM LAKE SUIGETSU, CENTRAL JAPAN AND ATMOSPHERIC ^{14}C CHANGE DURING THE LATE PLEISTOCENE

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ABSTRACT. We made accelerator mass spectrometry (AMS) ^{14}C measurements on terrestrial macrofossils from the Late Pleistocene/Holocene of the annually laminated sediments of Lake Suigetsu (central Japan). The AMS ^{14}C dates of terrestrial macrofossils showed agreement between varve counting years and calibrated ages (tree rings and U/Th on coral) in the interval of 10.5 and *ca.* 11.5 ka cal BP. Beyond 11.5 ka cal BP, the age difference between ^{14}C and varve counting years gradually diminish, contradicting published data on corals dated by U/Th and ^{14}C.

INTRODUCTION

Atmospheric radiocarbon change is not only crucial for absolute dating, but it also offers fundamental insights into the various changes of the Earth's system, such as solar activity and the global carbon cycle (*e.g.*, Stuiver *et al.* 1991). Thus, the extension of the ^{14}C calibration curve into the Last Glacial is important. A chronology extending back from the established tree-ring calibration curve has been constructed with U/Th and ^{14}C calibration of coral (Bard *et al.* 1993). To build an absolute/calendar time scale exactly, additional ^{14}C calibration, reaching back to the detection limit of the ^{14}C method (~40 ka BP), should be tested. One approach is to use terrestrial macrofossils from annually laminated lake sediments (Lotter 1991; Lotter *et al.* 1992; Goslar *et al.* 1992; Hajdas 1993; Hajdas *et al.* 1993, 1995) and glacial clay varved sediments (Wohlfarth *et al.* 1993; Wohlfarth, Björck and Possnert 1995; Björck, Wohlfarth and Possnert 1995), which can provide continuous chronologies of more than several thousand years.

The laminated sediments from the center of Lake Suigetsu (central Japan) provide a long chronology since at least the most recent interglacial time (Takemura *et al.* 1994). We have constructed a varve chronology for the upper part (*ca.* 16-m depth) of sediment cores from Lake Suigetsu. We report here the primary results of AMS ^{14}C measurements on terrestrial macrofossils of Late Pleistocene/Holocene annually laminated sediments from Lake Suigetsu.

METHODS

Lithology and Varve Chronology of Lake Suigetsu Sediments

Lake Suigetsu (35°35′N, 135°53′E), located near the coast of the Sea of Japan, is *ca.* 10 km around the perimeter and covers 4.3 ha^2 (Fig. 1). It is a kettle-type lake, nearly flat at the center and *ca.* 34 m deep. Four cores were collected from the center of the lake using piston-core samplers: in 1991 Cores SG1 (4 m long) and SG2 (11 m long); in 1993 Cores SG3 and SG4 (both 16 m long). A 75-m-long core, SG, was also collected in 1993 using a drilling machine (Takemura *et al.* 1994).

Distinct lamination is the most important sediment feature. The lithology of nearly all the cores is dominated by gray and dark-gray clay with white varves, consisting of diatom assemblages. The

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sets of white and gray layers were counted under a microscope using UV light. White layers of diatom assemblages could be distinguished clearly. Typical thickness of a pair of white and dark layers was somewhat <1 mm.

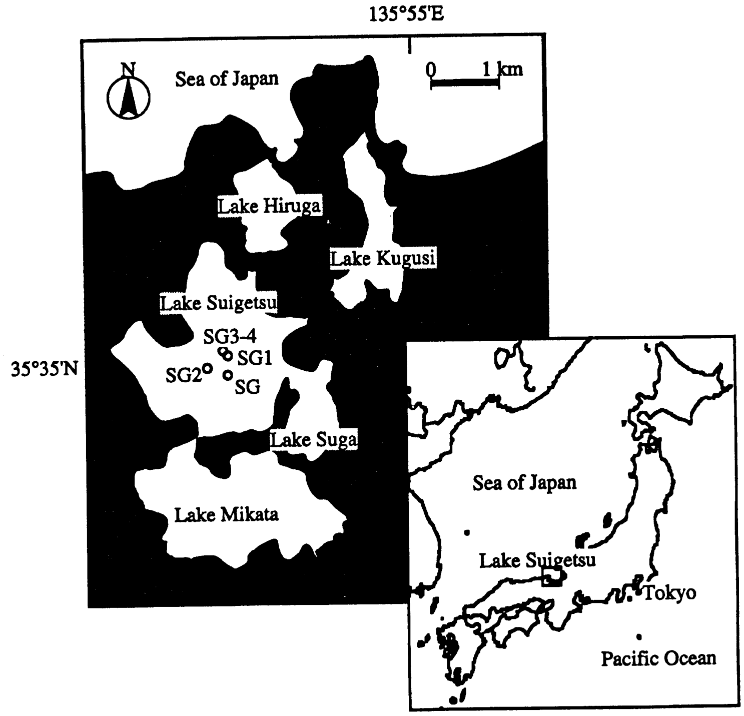

Fig. 1. Map of Lake Suigetsu and its surroundings

We confirmed the annual character of the laminations with: 1) detailed study of seasonal changes in diatom and mineral compositions; 2) microscopic inspection of thin sections; and 3) observations using a scanning electron microscope for the selected layers. The lamina consists of two layers; the clay mineral-poor and diatom-rich light-colored layer (corresponding to spring diatom blooming), and the clay mineral- and organic-rich dark-colored layer (corresponding to fall/winter deposition). Siderite ($FeCO_3$) or pyrite (FeS_2) is also concentrated in the transition between two layers (Fukuzawa, in press).

We analyzed the lamina for three cores: SG2, SG4 and the upper part of SG (Fig. 2). Lamina counting was possible only below *ca.* 29 cm depth because of the flocculent and disturbed uppermost sediments. It is difficult to determine the "zero-year" of the lake's varve chronology. We determined that many spike-like muddy turbidite layers are related to historical earthquake and flood events (some of known age). The turbidite layer below *ca.* 29 cm was caused by the Kanbun earthquake of AD 1662, based on comparison of each muddy turbidite layer and documented natural and human events (Fukuzawa *et al.* 1994). We adopted this age as the reference for the local varve chronology. Correlation among these cores was possible using the depth of characteristic marker beds, which are layers of mud-flow, including organic debris, silty clay and upwelling fining silt, as well as ash layers from Kikai-Akahoya Tephra (K-Ah) and Uzuryo-Oki Tephra (U-Oki).

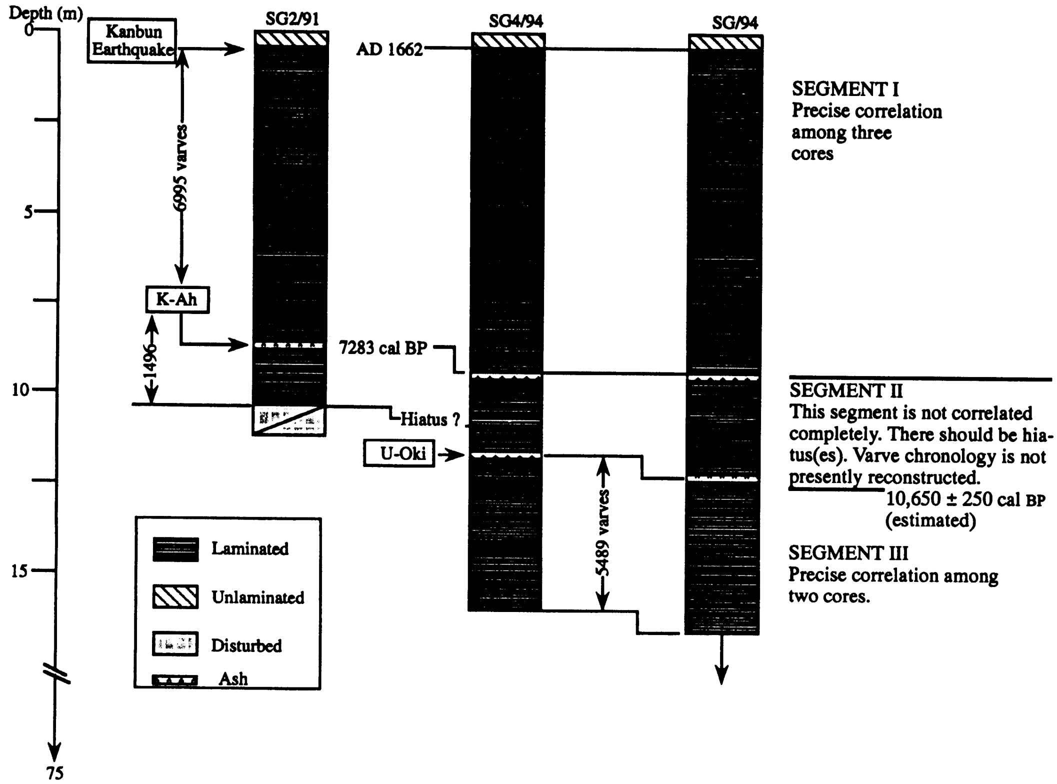

Fig. 2. Varve chronology of Lake Suigetsu (Cores SG2, SG4 and SG)

Precise correlation among the three cores is possible for the upper part of *ca.* 40 cm below the K-Ah layer (Segment I) and for the lower part of *ca.* 10 cm of U-Oki (Segment III). However, we have not been able to correlate completely each layer in Segment II. There should be sedimentary breaks (hiatus) between K-Ah and U-Oki because the characteristic muddy layers identified in cores SG2 and SG (which consist of a set of massive, relatively light gray and organic-rich dark gray layers) are absent in SG4. We constructed primary varve chronologies for Segments I and III, consisting of 6995 and 5489 pairs, respectively.

AMS ^{14}C Dating

The terrestrial macrofossil samples for AMS ^{14}C dating were collected from *ca.* 7-m sediments (969.5 cm to 1580 cm) of Core SG4. Single pieces of terrestrial macrofossils such as leaves, small branches and insect wings were selected and washed repeatedly with distilled water and then cleaned chemically by acid-alkali-acid (AAA) treatments to remove secondary contamination. For relatively large plant samples (>5 mg dry weight), holocellulose was also prepared from AAA-treated plant materials by benzene-ethanol extraction and delignification with sodium chloride (marked in Table 1). All processes were carried out in a dust-free room to reduce potential contamination during sample preparation.

The terrestrial macrofossils were sealed in an evacuated Vycor tube with CuO, and combusted at 850°C in an electronic furnace. The resulting CO_2 was purified cryogenically in a high-vacuum preparation system and then converted to graphite by reducing the CO_2 on Fe-powder with hydrogen gas in a sealed Vycor tube at 650°C (Kitagawa *et al.* 1993). ^{14}C ages of samples were measured along with the standard (NIST HOxII) using the Tandetron AMS at the Dating and Materials Research Center, Nagoya University (Nakai *et al.* 1984; Nakamura, Nakai and Ohishi 1987). We corrected carbon isotopic fractionation by $\delta^{13}C$, measured on a Finigan MAT 252 mass spectrometer. Conventional ^{14}C ages were calculated according to the procedure outlined by Stuiver and Polach (1977). The errors are at the one sigma level calculated from both the statistical uncertainty of sample and standard. The typical uncertainty was near ± 1% for samples with >0.5 mg carbon, primarily due to statistics. The results are listed in Table 1.

Results and Discussion

In Figure 3, the ^{14}C ages of 46 terrestrial macrofossils are plotted against sediment depth in Core SG4. The AMS ^{14}C dates of terrestrial macrofossils range between 6565 ± 70 and 14,665 ± 120 BP, thus encompassing the Late Glacial/Holocene transition. Two tephra layers of K-Ah and U-Oki are observed in core SG4, at 958–960 cm and 1163–1165.5 cm, respectively. The varve of upper parts of the K-Ah consists of 6995 pairs (corresponding to 7283 cal BP). The K-Ah layer dates to *ca.* 6500 BP, corresponding to 7300 cal BP based on the dendrochronologically based ^{14}C calibration curve. This is based on *ca.* 100 ^{14}C measurements all over Japan (*e.g.*, Machida and Arai 1991). The varve age of the K-Ah agrees closely with the dendrochronologically calibrated calendar age, supporting the annual character of the laminated sediments of Lake Suigetsu.

Our varve chronology of Lake Suigetsu is presently floating in Segment III. To match the ^{14}C ages of the terrestrial macrofossils on the calibration curve, we estimated the calendar age of U-Oki using the weight-mean values of reported ^{14}C ages of U-Oki (Machida and Arai 1991), the dendrochronological calibration (Kromer and Becker 1993) and the calibration program CAL15 (van der Plicht 1993). The reported ^{14}C age of U-Oki is 9551 ± 73 BP (weighted mean on 4 dates) and its corresponding calendar age is 10,650 ± 250 cal BP (10,400 to 10,900 cal BP). We used this age as the reference for the floating varve chronology in the discussion below.

Figure 4 compares our AMS ^{14}C dates of terrestrial macrofossils on varve chronology of Lake Suigetsu to the ^{14}C calibration curve (Stuiver and Reimer 1993). The existing dendrochronological curve and the results of U/Th and ^{14}C dating on coral (Bard *et al.* 1993; Edwards *et al.* 1993) are also included for reference. The comparison shows good

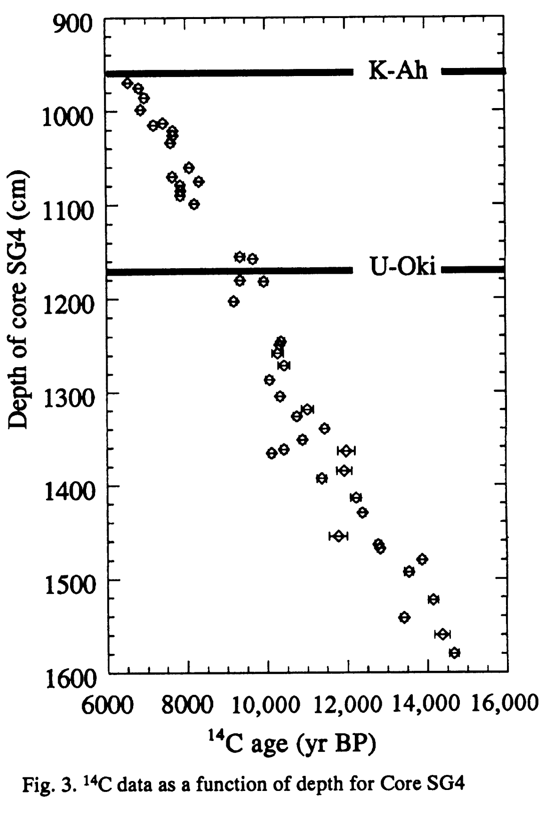

Fig. 3. ^{14}C data as a function of depth for Core SG4

TABLE 1. Results of AMS ^{14}C Dating on Terrestrial Macrofossils Selected from the Core Sediment (Core SG4) of Lake Suigetsu (Central Japan)

Sample	Depth (cm)	Material type*	Weight before combustion (mg)	Carbon content (mg/%)	Varve time (yr BP)	AMS ^{14}C age (± 1 σ BP)
Segment II						
SG4-II-1	969.5	L	5.8	3.1 / 53		6565 ± 70
SG4-II-2	975.4	L	4.2	2.5 / 59		6835 ± 85
SG4-II-3	985.8	L	4.2	2.5 / 59		6970 ± 85
SG4-II-4	998.5	L†	4.9	2.7 / 55		6875 ± 75
SG4-II-5	1013.0	L†	5.5	3.2 / 57		7195 ± 85
SG4-II-6	1015.0	L†	5.5	3.2 / 57		7675 ± 80
SG4-II-7	1021.0	L†	4.7	2.6 / 55		7675 ± 80
SG4-II-8	1026.0	B‡	6.0	2.4 / 40		7680 ± 80
SG4-II-9	1034.0	L†	4.8	2.8 / 60		7625 ± 80
SG4-II-10	1060.4	L	2.1	1.1 / 52		8080 ± 85
SG4-II-11	1070.0	L	1.4	0.7 / 50		7660 ± 90
SG4-II-12	1075.0	I	0.8	0.4 / 52		8330 ± 90
SG4-II-13	1079.0	L	2.9	1.5 / 52		7860 ± 90
SG4-II-14	1085.0	B	1.6	0.7 / 43		7870 ± 90
SG4-II-15	1090.0	L	0.8	0.5 / 55		7860 ± 90
SG4-II-16	1099.0	L	1.0	0.3 / 31		8210 ± 85
SG4-II-17	1155.5	B	0.8	0.5 / 57		9360 ± 115
SG4-II-18	1158.0	L	5.5	2.8 / 51		9680 ± 85
Segment III						
SG4-III-1a	1181.0	B	2.9	1.3 / 45	10,862 (±250)	9360 ± 90
SG4-III-1b	1182.0	B	2.9	1.4 / 47	10,865	9950 ± 100
SG4-III-2	1203.0	L†	4.2	2.1 / 49	10,978	9200 ± 90
SG4-III-3	1245.5	L	2.8	1.1 / 39	11,240	10,380 ± 85
SG4-III-4	1249.0	L	2.4	0.7 / 30	11,256	10,340 ± 90
SG4-III-5	1258.2	L	0.9	0.3 /30	11,260	10,295 ± 145
SG4-III-6	1271.3	L	1.6	0.4 / 26	11,503	10,450 ± 150
SG4-III-7	1287.0	B	0.7	0.3 / 46	11,679	10,080 ± 85
SG4-III-8	1305.0	L	1.2	0.3 / 25	11,846	10,350 ± 85
SG4-III-9	1319.3	L	0.9	0.3 / 32	12,017	11,030 ± 150
SG4-III-10	1326.5	L	2.9	0.8 / 26	12,078	10,760 ± 85
SG4-III-11	1340.0	L	0.4	0.2 / 39	12,169	11,460 ± 85
SG4-III-12	1352.0	L	2.4	0.4 / 18	12,228	10,905 ± 85
SG4-III-13	1362.5	L†	0.7	0.3 / 37	12,335	10,440 ± 85
SG4-III-14	1364.0	L	1.1	0.5 / 43	12,344	12,000 ± 215
SG4-III-15	1366.0	L	1.1	0.6 / 54	12,358	10,130 ± 85
SG4-III-16	1385.0	L	1.1	0.3 / 24	12,488	11,950 ± 190
SG4-III-17	1393.5	L	0.6	0.3 / 46	12,530	11,390 ± 115
SG4-III-18	1414.0	L	0.9	0.5 / 51	12,793	12,235 ± 130
SG4-III-19	1430.0	L	1.7	0.9 / 54	12,987	12,395 ± 85
SG4-III-20	1455.4	L	0.5	0.2 / 51	13,234	11,790 ± 225
SG4-III-21	1464.0	L	2.1	1.1 / 52	13,331	12,780 ± 85
SG4-III-22	1468.0	L	4.8	2.0 / 42	13,355	12,825 ± 85
SG4-III-23	1480.3	L	2.7	1.1 / 39	13,450	13,875 ± 85
SG4-III-24	1493.0	L	2.5	1.2 / 47	13,599	13,540 ± 105
SG4-III-25	1523.3	L	1.6	0.8 / 49	13,844	14,150 ± 130
SG4-III-26	1542.0	L	2.0	1.0 /49	14,081	13,410 ± 90
SG4-III-27	1560.3	L	0.7	0.3 /44	14,339	14,375 ± 185
SG4-III-28	1580.0	B‡	2.7	1.0 / 37	14,440	14,665 ± 120

*L=leaf, B=branches, I=insect wing; †Cellulose from AAA-treated leaf; ‡Cellulose from AAA-treated branches agreement between the Lake Suigetsu varve chronology and the tree-ring ^{14}C calibration curve between 10.5 and 11.5 ka cal BP. Beyond 11.5 cal BP, the age difference with ^{14}C gradually diminishes and the varve age agrees closely with ^{14}C age *ca.* 13 ka cal BP. This contradicts with the U/Th and ^{14}C calibration data from corals (Bard *et al.* 1993; Edwards *et al.* 1993), which suggest higher $\Delta^{14}C$ values for the same period. Our ^{14}C trend seems to be approximately consistent with that of Swedish varve chronology (Wohlfarth *et al.* 1993; Björck, Wohlfarth and Possnert 1995; Wohlfarth, Björck, and Possnert 1995) and chronologies for Lake Soppensee in Switzerland and Lake Holzmaar in Germany (Hajdas 1993; Hajdas *et al.* 1993, 1995).

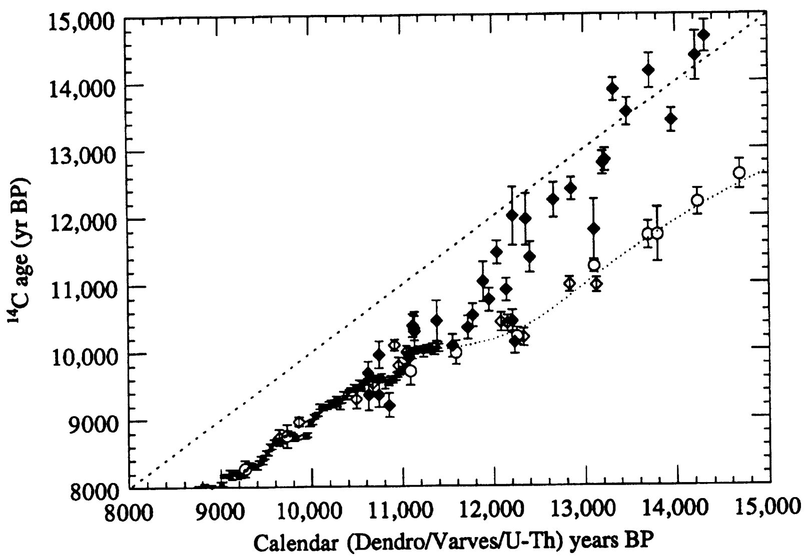

Fig. 4. Comparison between AMS ^{14}C dates of terrestrial macrofossils on varve chronology of the Lake Suigetsu, Central Japan (◆) and ^{14}C calibration curve (····· = Stuiver and Reimer 1993). The existing tree-ring curve (Kromer and Becker 1993) and U/Th and ^{14}C dates on coral (O: Bard *et al.* 1993;◇: Edwards *et al.* 1993) are also included for reference. All statistical uncertainties are quoted at 2 σ.

For construction of the absolute time scale using the laminated sediment, we must consider the possibility of hiatus and miscounting of varve numbers. However, based on the lithological observation and the precise correlation among cores (Cores SG4 and SG), there is no evidence of miscounts of considerably large numbers of varves or long-lived hiatus for the lower parts of the U-Oki (Segment III). To come close to the age obtained by U/Th calibration of the ^{14}C time scale, one must assume a miscount of >2000 varves or a hiatus of *ca.* 2 m in 4.25 m sediment, which we consider highly unlikely.

Conclusion

From AMS ^{14}C measurements on terrestrial macrofossils from Late Pleistocene/Holocene annually laminated sediments from Lake Suigetsu, we conclude the following:

1. The AMS ^{14}C age of laminated sediments from Lake Suigetsu is *ca.* 15 ka BP at a depth of 16 m, encompassing the Late Glacial as well as the Holocene. The ^{14}C profiles exhibited an approximately continuous feature with an absence of long-lived sedimentary breaks. The Lake

Suigetsu varve sediments are an excellent tool with which one may obtain an absolute time scale as a basis for high-precision studies of environmental change and the extension of ^{14}C calibration back from the established tree-ring calibration curve, especially to the detection limit of the ^{14}C method (~40 ka yr BP).

2. The ^{14}C measurements of terrestrial fossil plants agree well with varve and tree-ring calibrated ages between 10.5 and 11.5 ka cal BP. The age difference between varve and ^{14}C years is *ca.* 1 ka in this interval. However, this age difference gradually diminishes before 11.5 ka BP, whereas the differences between dates of U/Th and ^{14}C increase steadily. The discrepancies between these results cannot yet be explained. More detailed chronological studies of varve sediments and high-precision ^{14}C measurements for older parts of Lake Suigetsu sediments, which are in progress, are needed to resolve the calibration problem during the Late Pleistocene.

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