Archaeological and Geological Applications of ^{14}C Dating with a Tandetron AMS at Nagoya University

名古屋大学タンデトロンAMSによる^{14}C年代測定とその考古学及び地質学への応用

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[**Abstract**] A Tandetron accelerator mass spectrometer (Tandetron AMS), an apparatus dedicated to high sensitivity radiocarbon (^{14}C) measurements, manufactured by General Ionex Corporation, USA, has been used since 1983 to measure the ^{14}C concentrations of environmental samples as well as ^{14}C dates of geological and archaeological materials, at the Dating and Materials Research Center (DMRC), Nagoya University. The author presents here a brief review of the present performance and some archaeological and geological applications of the Tandetron AMS, as well as a brief introduction to a so-called second generation AMS machine, an AMS ^{14}C dating apparatus, currently of the highest performance, manufactured by High Voltage Engineering Europe, BV, the Netherlands, which has been recently installed at the DMRC.

Key words: radiocarbon dating, accelerator mass spectrometry (AMS), tandetron, recombinator system, high-resolution chronology

1. Introduction

Radiocarbon (^{14}C) dating has played a very important role in archaeology since its appearance in the 1950's. Many ^{14}C dates for archaeological samples of known historical age from ancient Egypt have been accepted as proof of the method (Libby, 1955). In Japan, ^{14}C dating results have extended the Neolithic period (Jomon period) to almost double its previously accepted length, which had been established based on pottery chronology (Hamada, 1981). By using ^{14}C dates prehistoric events can be correlated among various archaeological sites in Japan, as well as in different countries around the world (Aitkin, 1990). Recently, ^{14}C dating, in particular, by the accelerator mass spectrometry (AMS) method, has been widely applied to historical events and samples (Bowman, 1990; Oda, *et al.*, 1998; Yoshizawa, *et al.*, 1996). Among various dating methods that are applicable to archaeological samples, the ^{14}C dating method is considered to be the most useful and reliable, because it is applicable to a large variety of archaeological and geological sample materials(any organic residue or inorganic material which contains carbon of atmospheric origen). It is now possible to obtain high-precision and high-accuracy measurements with less than 1 mg of carbon, and to attain wider age ranges than before (from recent to 50-60 ka B.P.).

Techniques of AMS developed since 1977, based mainly on a tandem accelerator and associated apparatus used to analyze charge state, energy, mass number, and atomic number of accelerated ions, have enabled us to measure extremely-low abundance nuclides (isotope ratio of 10^{-12} to 10^{-16} relative to its stable isotope), such as ^{10}Be (half life: 1.5×10^{6} yr), ^{14}C (5,730 yr), ^{26}Al (7.1×10^{5} yr), ^{36}Cl (3.0×10^{5} yr), ^{41}Ca (1.0×10^{5} yr), ^{129}I (1.57×10^{7} yr), etc., in natural samples. Among such nuclides, ^{14}C is the most useful for age determination in archaeology, mainly because carbon is contained in many archaeological remains. The least amount of carbon necessary for the AMS ^{14}C measurement has been reduced to about 0.1 mg and the oldest date measurable has been extended to about 50,000-60,000 yr. B.P., compared to a few grams and about 35,000 yr. B.P., respectively, for beta-counting measurements of ^{14}C.

The Tandetron AMS has been in use since 1983 to measure ^{14}C concentrations of environmental samples as well as ^{14}C dates of geological and archaeological materials at the Dating and Materials Research Center (DMRC), NagoyaUniversity (Nakamura, *et al.*, 1985; Nakamura, 1995). The author presents here a brief review of the present performance, as well as some archaeological applications of the Tandetron AMS. In addition, characteristics and performance of a new Tandetron AMS system, which has been installed at the DMRC (Nakamura, 1998), are described briefly.

2. Performance and applications of the Tandetron AMS

Present performance of the Tandetron AMS is summarized as follows. By using a graphitized target (Kitagawa *et al.*, 1993), the least amount of carbon necessary for a ^{14}C measurement is about 0.1 mg. However, 1 to 1.5 mg of carbon is routinely used, for ease of sample handling and for reducing the eggect of carbon contamination from external materials. The oldest measurable age was more than 50,000 yr. B.P. until 1990 (Nakamura *et al.*, 1992a, 1992b; Sawada *et al.*, 1992; Nakamura *et al.*, 1997a; Kawakami *et al.*, 1992; Sago *et al.*, 1992). However, the measurable age is presently limited to less than around 40,000 to 50,000 yrs. B.P., as the result of a gradual increase in ^{14}C background following the installation of a 28-sample loading system in the ion source in 1990. Fig. 1 shows the measurement error distribution for routine runs. The figure indicates that the measurement errors are ±60 to ±80 years for samples younger than 10,000 yr. B.P, after 2 to 3 hours measurement. The error gets larger for samples older than 20,000 yr. B.P.

The number of samples measured per year and the total number of samples analyzed up to present are shown in Fig. 2. Over the last five years, about 700-800 samples have been consistently dated annually, and a total of 7,540 samples from various research fields were measured from the installation of the machine to the end of March 1998. Although the Tandetron AMS has been intensively utilized, the number of samples analyzed annually is rather limited when compared to the number of samples submitted to the AMS facility of the DMRC by many users from various research fields. This is mainly due to low negative current intensity from the ion source (a HICONEX-844 ion source, modified for loading up to 18 targets at a time), as well as rather low throughput of the total system. Thus a new AMS system was installed at the DMRC to overcome

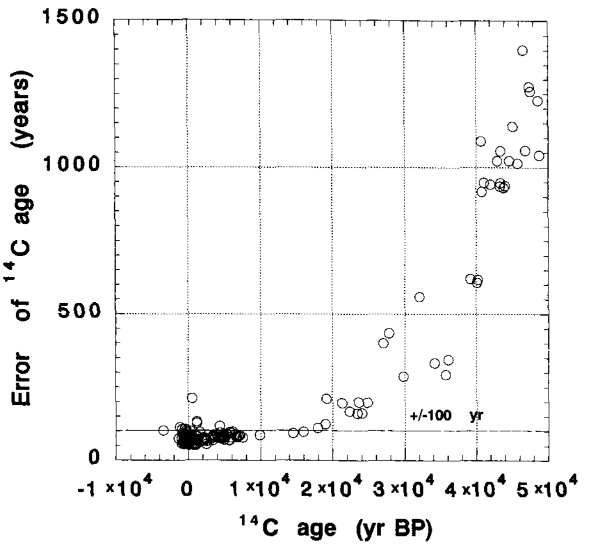

Fig.1 Uncertainties (±1 sigma) of ^{14}C dates measured by the old Tandetron AMS at Nagoya University

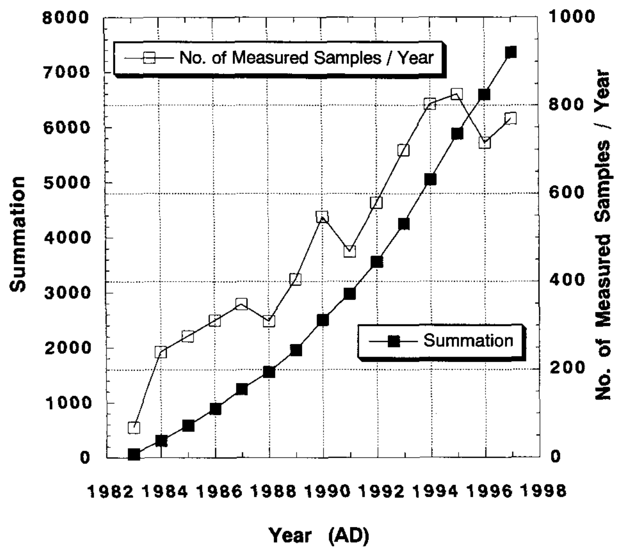

Fig.2 Number of samples measured annually (open square) and the total number of samples (closed square), measured by the old Tandetron AMS

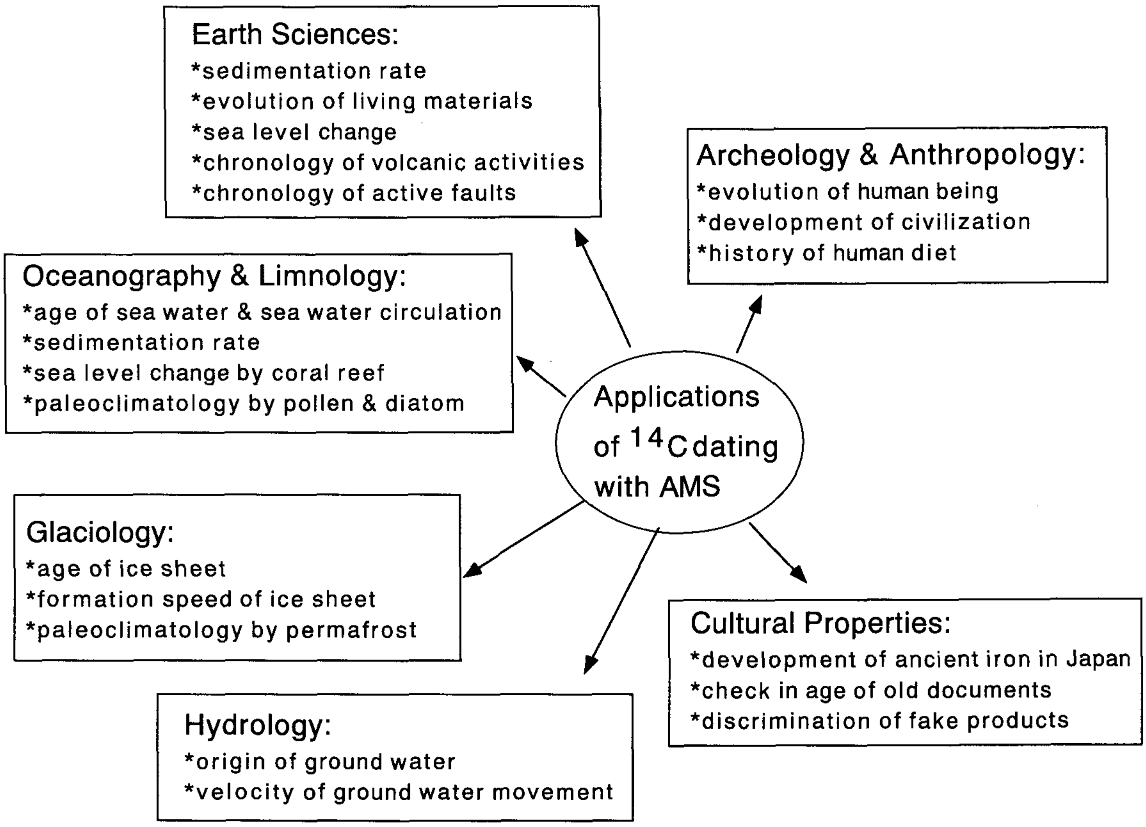

Fig.3 Six main research fields studied by using ^{14}C dates measured with theTandetron AMS at Nagoya University

this limitation, which is now being tuned up for ^{14}C measurements.

3. Some applications of ^{14}C dating with the old AMS machine

AMS ^{14}C dating at the DMRC has been applied to many archaeological and geological samples. Fig. 3 shows six research fields in which AMS ^{14}C dating is intensively used. Some results of interesting applications are described below.

(1) Dating of foraminifera fossil samples from ocean sediments

To demonstrate the reliability of the ^{14}C dates measured using the Tandetron AMS, ^{14}C dates of planktonic foraminifera samples collected from ocean sediments are shown against the sediment depth in Fig. 4. A piston-core sample of ocean sediments, KT89-18, p4, was collected from the 2,800 m deep ocean floor of the Nankai truogh, off Shikoku Island, Japan (Fig. 5). Several hundred pieces of forminifera shell fossil of ca. 400 μm in diameter, composed mainly of $CaCO_3$, were collected by hand-picking under a microscope, and treated with phosphoric acid to obtain CO_2, which was finally changed to graphite to be used in the ion source of the Tandetron (Murayama *et al.*, 1993). Fig. 4 shows good consistency between the ^{14}C ages and sample horizons, i.e., foraminifera collected from deeper sediment layers give systematically older ages. From the surface to the 8-meter layer, the ^{14}C age increases monotonically from 0 yr. B.P. to ca. 35,000 yr. B.P. Layers of tephra erupted from Kyushu Island volcanoes accumulated and were well preserved on the ocean floor. The ^{14}C ages of these tephras, already determined by using terrestrial samples as 6,300 yr. B. P. for the Kikai-Akahoya (K-Ah) tephra and 25,000 yr. B.P. for the Aira-Tanzawa (AT) tephra, are consistent with the corresponding ages of the layers estimated from ^{14}C ages measured for foraminifera samples (Murayama *et al.*, 1993). This shows that the treatment of small size foraminifera was not affected

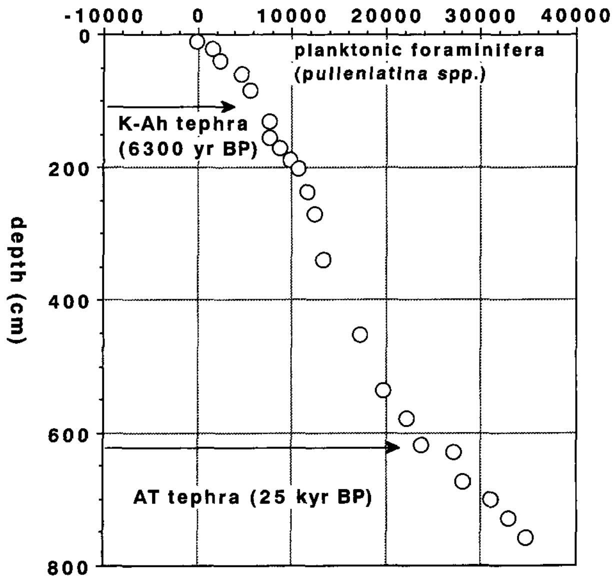

Fig.4 ^{14}C dates of planktonic foraminifera samples collected from ocean sediments plotted against the sediment depth
One signa errors are within the circle. The ocean sediment core, KT89-18, p4,was collected from the 2,800 m deep ocean floor of the Nankai trough, off Shikoku Island, Japan. The arrows indicate the horizons at which the K-Ah and AT tephras with eruption age of 6,300 yr. B.P. and 25,000 yr. B.P. were detected

Fig.5 Locations of archaeological and geological sites providing samples for ^{14}C dating studies

much by modern carbon contamination, and that an acceptable accuracy of ^{14}C measurements was attained with the Tandetron.

(2) Eruption history of the Aira Caldera

AMS ^{14}C dating was used to establish a detailed eruption history of the Aira Caldera, located in the northernmost part of Kagoshima Bay, southern Kyushu, Japan (Fig. 5). In total, 60 samples, collected from paleosol sediments immediately below and above the tephra layers, and charcoal remains from within the tephra layers, were ^{14}C dated. The results are given in Fig. 6. The sample ^{14}C dates are consistent with stratigraphic relations among them. Based on the ^{14}C dates, a well-constrained eruptive history of the Sakurajima volcano was established, from its formation by the huge Aira eruption of ca. 25,000 yr. B.P. until the historical An-ei eruption of AD 1779 (Okuno *et al.*, 1997). In this study, it became clear that ^{14}C dates of a paleosol sample collected just below a tephra layer can provide an age that is very close to the eruption age of the tephra (Okuno, 1997).

(3) ^{14}C ages of peat layers intercalated by tephra deposits at Minami Karuizawa

Several tens of tephras erupted from the Asama-yama volcano, located in Nagano Prefecture, central Japan, have accumulated in the Minami-Karuizawa lacustrine sediments, located in the Saku Basin, Nagano Prefecture (Fig. 5). The lacustrine sediments are made not only of tephra but also peat layers containing a lot of buried trees and plant residues. Thus, charcoal, tree trunk and peat samples that were clearly related to the tephras could be ^{14}C dated using the Tandetron AMS. Fig. 7 shows the ^{14}C ages of peat layers and the tephra horizons from ca. 20,000 yr. B.P. to 11,000 yr. B.P. The ^{14}C dates of plant samples increase almost monotonically as their horizons become deeper. However, some older dates are somewhat discrepant with one another (Nakamura *et al.*, 1997b). Thus, buried trees and plant remains from the same peat layers should be carefully examined to see whether they give ^{14}C ages which are consistent with each other. In addition, the present discrepancy of the ^{14}C dates suggests that stratigraphy of these sediments should be re-examined carefully.

(4) ^{14}C dates of wood, mammalian bone and shell fossils from a shell mound at the Awazu submarine site

The Awazu submarine archaeological site, 2-3 meters below the water surface, is located in the southern basin of Lake Biwa, near where the Seta River flows out from the lake, in Shiga Prefecture, Central Japan. A shell mound was excavated during the 1990-1991 survey of the site. Seven sets of wood, animal bone, and shell fossil samples, collected from each of the seven layers of the shell mound, were dated using the AMS ^{14}C method (Nakamura *et al.*, 1997c). The results are shown in Fig. 8.

The ^{14}C dates for each of the three kinds of samples did not show big differences between the seven layers, as shown in Fig. 8. This tendency is consistent with the archaeological estimation that the shell mound was formed within about 100 years,

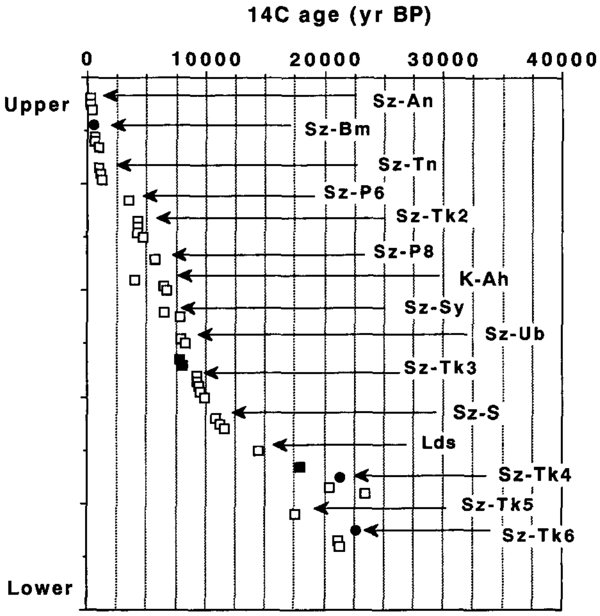

Fig.6 ^{14}C dates for samples collected from paleosol sediments immediately below and above the tephra layers, and for charcoal remains in the tephra layers erupted from the Aira Caldera, Kagoshima Bay, southern Kyushu.
Closed circles, open and closed squares indicate charcoal samples found in the tephra layers, and samples collected from paleosol sediments immediately below and above the tephra layers, respectively. Arrows indicate the horizons of the tephra layers.

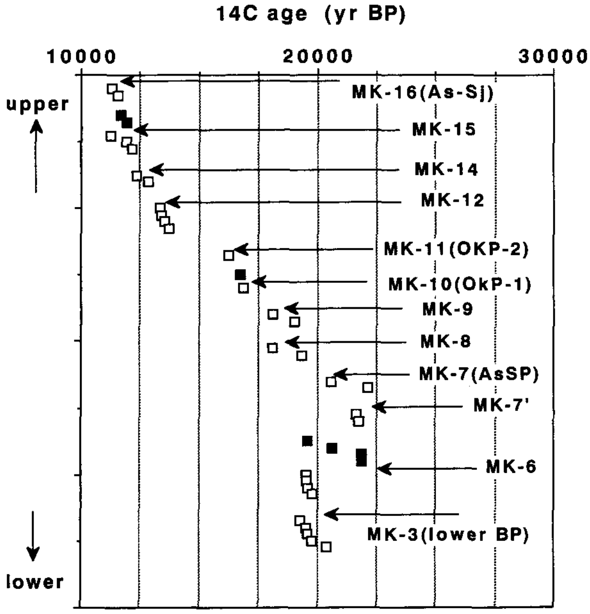

Fig.7 ^{14}C dates for charcoal, tree trunk and peat samples collected from the Minami-Karuizawa lacustrine sediments in the Saku Basin, Nagano Prefecture.
Open and closed squares indicate wood or plant residue samples collected from peat sediments immediately below and above the tephra layers, respectively. Arrows indicate the horizons of the tephra layers.

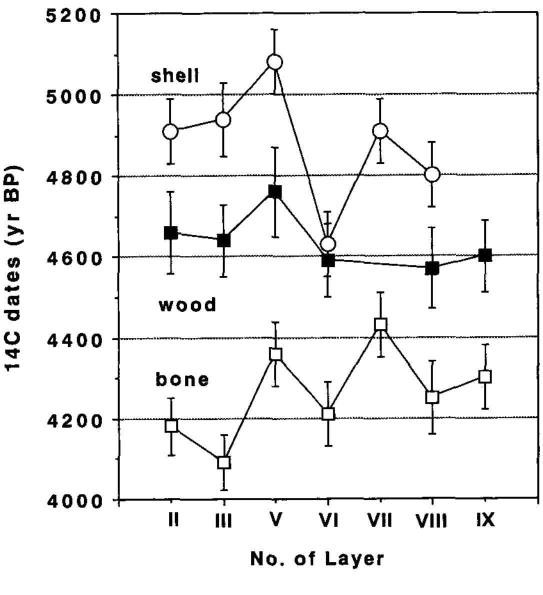

Fig.8 ^{14}C dates for seven sets of wood, animal bone, and shell fossil samples collected from each of the seven layers (from II down to IX layers) of the shell mound at the Awazu submarine archaeological site in Lake Biwa, Shiga Prefecture.
Open circles, closed and open squares indicate shell carbonate, wood and bone fossil samples, respectively.

because fragments of the Funamoto-Ⅰ type pottery, which correspond to an early stage of the middle Jomon, were predominant in every layer of this shell mound. However, ^{14}C dates were systematically different between the three types of samples: shell fossil samples showed the oldest dates from 4,800-5,080 yr. B.P., except for a very young date (4,630±80 yr. B.P.) for the layer Ⅵ; wood samples provided the middle dates (4,570-4,760 yr. B.P.); while bone fragment samples provided the youngest ones (4,090-4,430 yr. B.P.).

The reasons for the differences in ^{14}C dates among the three kinds of samples collected from the same horizons are not clarified yet. Shell carbonate originates from dissolved inorganic carbon in the lake water, which carbon was derived partly from the dissociation of old organic materials in the lake sediment, possibly including dead carbon from limestone rock surrounding Lake Biwa (Nakamura *et al.*, 1998a). Thus the shell carbonate samples can be older than the formation age of the shell mound. In addition, younger ^{14}C dates for collagen separated from bone samples indicate that younger carbon may have contaminated the bone samples when they were in the sediment, and may not have been removed completely during chemical preparation of collagen. Thus, amino acids, that were more essential to bones, have been extracted to provide ^{14}C dates for these bone samples (Minami and Nakamura, 1998). The amino acid fractions of the bone samples collected from the Awazu shell mound tend to show older ^{14}C ages than the corresponding collagen, which are almost consistent with the ^{14}C ages of wood materials from the same layers.

(5) ^{14}C dates of charcoal samples from the Sannai Maruyama site

The Sannai-Maruyama site, located in Aomori Prefecture, the northernmost on Honshu Island, is a huge ancient residential site used by humans from the middle to the end of the middle Jomon period. From around the No.6 Iron Tower, located in the north-west part of the site, a lot of animal and plant remains were collected, along with many fragments of Jomon pottery. These potsherds were estimated typologically to belong to an Ento-Kaso type that is peculiar to the early Jomon period. The sediments at the No. 6 Iron Tower were divided into 6 layers, as shown in Fig. 9, and the lowest horizon, No. Ⅵ, was subdivided further into two layers, Ⅵ-a and Ⅵ-b, according to difference in the facies and their remains (Fig. 9). Five charcoal samples collected from each of Ⅵ-a and Ⅵ-b layers were ^{14}C dated with the Tandetron AMS (Nakamura *et al.*, 1998b). The results are shown in Fig. 10. ^{14}C dates are consistent within errors for both layers, though the dates for Ⅵ-a and Ⅵ-b give about 200 to 300 years difference. One ^{14}C age for the Ⅵ-a layer is clearly older by about 500 years than the other 4 dates. This could be due to contamination from the lower horizon, Ⅵ-b. It would be very interesting to investigate whether any typological difference can be found in the pottery from layers Ⅵ-a and Ⅵ-b, which gave a ^{14}C age difference of about 250 years.

4. Expected performance of the 2nd generation Tandetron AMS

Recently, we have added a second-generation Tandetron AMS (a Model 4130-AMS, radiocarbon dating system) manufactured by High Voltage Engineering Europe

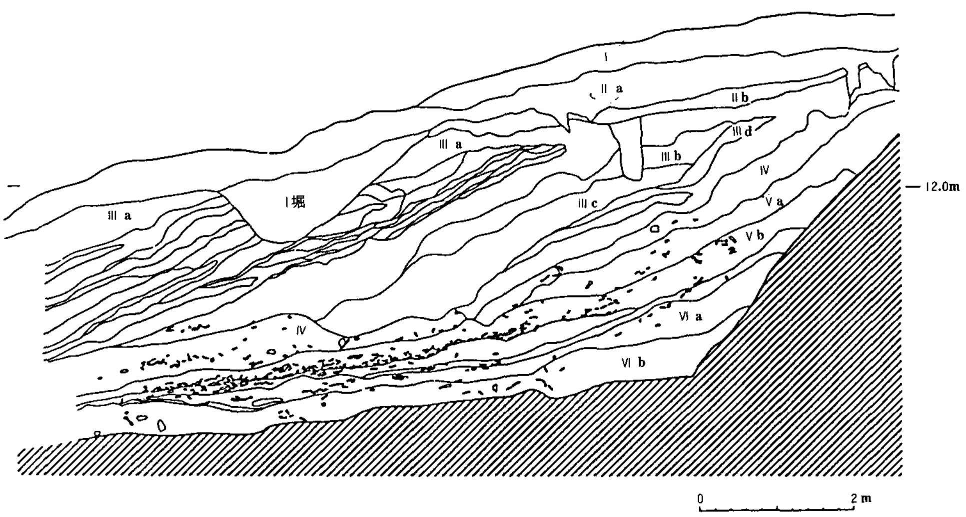

Fig.9 Representative columnar section of nine layers from I to VI-b excavated near No. 6 Iron Tower of the Sannai-Maruyama site, Aomori Prefecture.

(HVEE), BV, the Netherlands. Two similar HVEE AMS systems have been installed successfully at the University of Groningen, the Netherlands (Mous *et al.*, 1994), and at the University of Christian-Albrechts, Kiel, Germany (Nadeau *et al.*, 1997). They have already proved excellent performers in carbon-isotope-ratio measurements for graphite targets prepared from carbonaceous materials, giving a reproducibility in $^{13}C/^{12}C$ ratio of $\pm0.1\%$; statistical uncertainties and reproducibility in $^{14}C/^{12}C$ ratio of ±0.15-0.22% and $\pm0.3\%$, respectively. These results imply that the error in ^{14}C ages could be reduced to about ±25 years.

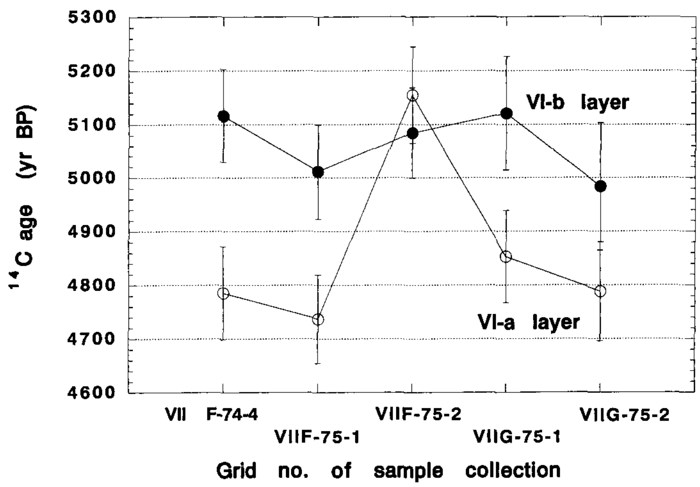

Fig.10 ^{14}C dates for five charcoal samples collected from each of VI-a and VI-b layers excavated near No. 6 Iron Tower of the Sannai-Maruyama site, Aomori Prefecture. Open and closed circles indicate charcoal samples collected from VI-a and VI-b layers, respectively.

The main improvements of the 2nd-generation Tandetron, compared with the old Tandetron, are summarized in Table 1. They are: (1) a high intensity cesium sputter ion source is provided with the new system, so that the ^{14}C counting rate is almost one order higher than that for the old system. In addition, since up to 59 targets can be loaded at a time and can be measured automatically, measurements are conducted more efficiently. (2) carbon isotopes $^{12}C^-$, $^{13}C^-$ and $^{14}C^-$ are injected into a tandem accelerator simultaneously, by using a recombinator system, which archives high accuracy measurements of the carbon isotope ratio. (3) the terminal voltage of the tandem accelerator is 2.5 MV, which gives the maximum yield when producing C^{3+} from C^- in the charge exchange process in the accelerator. In addition, a slit feedback system with a position sensitive Faraday cup to monitor the

Table 1 New Tandetron AMS from HVEE
The main improvements from the old Tandetron AMS and the expected performance of the new Tandetron AMS are summarized.

1. High intensity ion source:
*HICONEX 488 ion source ⇨ HVEE ion source
*C^- ion intensity is 10 times more ⇨ higher counting rate,
⇨ shorter counting time
*up to 59 targets can be loaded ⇨ more efficient measurement

2. Recombinator, a simultaneous three carbon isotopes injection system:
*simultaneous injection of $^{12}C^-$, $^{13}C^-$, $^{14}C^-$
⇨highly stable isotpe ratio measurement
*simultaneous measurment of ^{12}C, ^{13}C, ^{14}C
⇨ correction of isotope fractionation induced by machine,
⇨ high accuracy measurement of the isotope ratio

3. Therminal voltage of accelerator:
*2.5 MV ⇨ optimum for yielding highest intensity of C^{3+} from C^-,
⇨ higher detection efficiency & higher counting rate

4. Heavy ion detector:
* ΔE-$E_{residual}$ measurement ⇨ ^{14}C background reduction,
⇨ clear ^{14}C identification

5. Computer control
*optical link ⇨ protect damages of computer system from high voltage sparks
*automatic measurement ⇨ reduce the operators duty,
⇨ more efficient measurement

6. Overall performances
*measurement error: ±60— ±80 yr BP ⇨ ±20— ±30 yr BP
*measurement capacity: 700—800 samples/yr ⇨ 3000 samples/yr

energy of acclelrated $^{13}C^{3+}$ ions stabilizes the terminal voltage at a level of $\Delta V/V \approx 6 \times 10^{-4}$ (Mous *et al.*, 1994), which furnishes highly stable isotope-ratio measurements. (4) to separtate $^{14}C^{+3}$ ions from various background ions, a heavy ion detector (an ionization detector) measures the total kinetic energy of incoming ions, E_{total}, as well as the residual kinetic energy after energy loss depending on their atomic numbers, $E_{residual}$, by passing them through a gas absorber (isobutane gas column of 10 mbar pressure and 10 cm long). Provided that the background ions are rejected efficiently, ^{14}C ages as old as 60,000 yr. B.P. will be measurable with this system. (5) a computer control system is provided with the AMS instrument which automates the carbon-isotope-ratio measurement for multiple samples. This provides us a high-throughput measurement.

As a result of these improvements, the improvements in ^{14}C measurements with the new Tandetron are: (1) the measurement error on the ^{14}C age may be as small as ± 25 years, with a measurement time of a few tens of minutes for a carbon sample of about 1 mg; (2) a fully-automatic measurement can be routinely performed; (3) more than 3,000 samples can be measured annually.

The new system will be used efficiently and speedily for ^{14}C dating of various kinds of carbonaceous samples, submitted by domestic researchers as well as by those outside of the university, if graphite targets are provided to us, which have been prepared by the researchers themselves.

Acknowledgments

The author would like to thank Prof. M. Adachi, the director of the DMRC, for his continuous support of the AMS group. He also thanks all the members of the AMS group of the DMRC, all the users, as well as technical staff of the Equipment Developing Facility of School of Science, Nagoya University, for their kind support during improvements to the AMS dating system. He thanks Dr. Brian Chisholm of the University of British Columbia, Canada, for his careful reading of the manuscript.

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国立歴史民俗博物館研究報告
第81集 1999年3月

名古屋大学タンデトロンAMSによる^{14}C年代測定とその考古学及び地質学への応用

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タンデム加速器と質量分析計を組み合わせた加速器質量分析（AMS）技術による天然の極微量元素測定の方法は，アメリカ合衆国とカナダを舞台にして1976年から1977年にかけて開発され，1980年代には早くも実用の段階に入った。その一つが放射性炭素^{14}C測定による年代測定であり，考古学・地質学の年代測定に関連して新たな応用研究の分野が開拓されている。

AMSの発展の初期の段階では，物理学の実験などに用いられていた既存の汎用タンデム加速器（加速電圧5～12MV）を改造してAMSに利用することが一般的で，全世界で30を越える施設でAMSが利用可能となっている。こうした既存のタンデム加速器の改造とは別に，小型タンデム加速器（加速電圧2～3MV）を用いたAMS専用のシステム（タンデトロン加速器質量分析計）が米国 General Ionex 社によっていち早く開発された。その1台が1981～1982年に名古屋大学に導入され，さまざまな研究に利用されてきた。成果の一部が，本稿に紹介されている。

さらに，1991年以降は，形状こそ従来のタンデトロン分析計と同程度であるが，最新のコンピュータ・機械制御の技術を取り入れた高性能の最新型タンデトロン分析計が開発されている。この第二世代の^{14}C測定専用の分析計は，イオン源の出力が従来のタンデトロン分析計のイオン源の出力に比較して約10倍も大きく，かつ，^{14}Cの検出効率が高いため，現代のショ糖試料から調製された1mgのグラファイトについて，わずか20分間の測定で20万個を越える^{14}Cが計数される。従って比較的若い試料については，20分間の測定で年代値の誤差で±20年の統計誤差は容易に達成できよう。また，測定操作はコンピュータによる自動制御となり，省力化，高生産性（年間3,000個の測定能力を持つとされる）が期待される。新型機は現在，米国のWoods Hole 海洋研究所，オランダのGroningen 大学，ドイツの Christian-Albrechts 大学に設置されており，また1996年3月には名古屋大学に設置され，現在調整が進められている。この第二世代分析計を用いて，土器編年検討用試料，文化財試料，樹木年輪試料，活断層関連試料，火山噴火関連試料などの高精度の年代測定が計画されている。

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