


A HOMEMADE SEMIAUTOMATIC GRAPHITIZATION DEVICE FOR AMS ¹⁴C DATING AT NTUAMS LAB

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ABSTRACT. A low-cost and computer-controlled graphitization system connected to an elemental analyzer (EA) has been designed and built at the NTUAMS Lab. This semiautomatic system equips 6-unit reactors for the graphitization of CO₂ with H₂ on the iron catalyst. The entire procedure takes about 7 hours for iron conditioning, sample combustion and loading, and graphitization. The system can produce good-quality graphite for samples containing 0.5–1.6 mg carbon mass, with the pressure yield of graphitization ranging from 57.7% to 87.1%. The average values of OXI and OXII agree well with the consensus value, but the result of ANU sucrose was observed to be slightly higher than the reported one. The background samples of anthracite over ten months yielded an average of 0.38±0.10 pMC (n=21) corresponding to a ¹⁴C age of 45 kyr BP. Intercomparison samples L and M of FIRI exhibit that the measured ¹⁴C ages are almost identical to the consensus values and have a small spread in these values. The system has been carrying out graphitization for total organic carbon (TOC) of peat samples, and providing a more efficient and convenient way for AMS ¹⁴C dating.

KEYWORDS: AMS ¹⁴C dating, graphitization system, hydrogen reduction, organic carbon.

INTRODUCTION

The HVEE 1.0 MV Tandetron Model 4110 BO-accelerate mass spectrometry (AMS) for radiocarbon (¹⁴C) dating has been performed at the NTUAMS Lab of National Taiwan University (NTU) since 2013. To eliminate Li₂⁺⁺ interference, the AMS measures ¹⁴C³⁺ ions with a transmission rate of ~18%. Every sample measurement is set up for four cycles and analyzed for 1500 seconds in every cycle, so that measurement time of a sample is normally 6000 seconds in total. During the analysis for every sample, the beam current of ¹²C³⁺ should remain steady and higher than 1.0 μA. Finally, the weighted mean and uncertainty values of ¹⁴C/¹²C and ¹³C/¹²C ratios are taken to calculate the sample age and the associated error (Li et al. 2022).

In order to convert sample CO₂ into graphite, three vacuum lines for CO₂ extraction and purification were installed with the TiH₂/Zn reduction method. At first, the quartz tube is purified in the vacuumed system under a pressure of 10⁻⁵–10⁻⁶ mbar. The organic sample reacts with CuO and Ag in the evacuated quartz tube at 850°C for 6 hours to acquire CO₂ gas (Barile et al. 2019; Li et al. 2022). The produced CO₂ in the quartz tube is purified cryogenically on the vacuum line. The next step is to transfer and seal the pure CO₂ into a combination glass tube (Xu et al. 2007). The combination glass tube contains a 9-mm diameter 6-inch long tube in which the reagents of TiH₂ and Zn exist at the bottom, with a small 6-mm diameter 2-inch long tube inside which has iron powder/catalyst. The CO₂ reduction to graphite is carried out for 6 hours in a muffle furnace at 550°C. Finally, the graphite is pressed into a target holder and measured with the AMS for ¹⁴C dating. It is a highly time-consuming experiment process and a sample takes four separate days.

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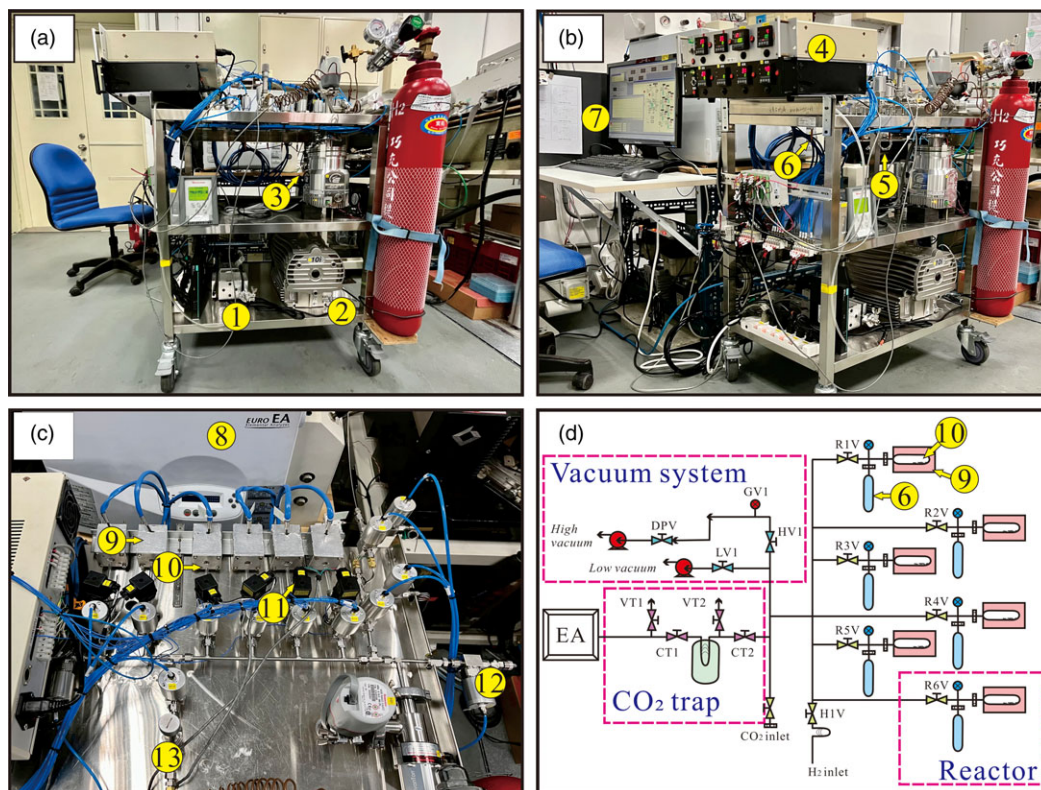


Figure 1 Photographs of the semiautomatic graphitization system and its devices. (a) The vacuum system included a diaphragm pump (1), dry scroll pump (2), and one high vacuum turbo molecular pump (3). (b) Picture shows the components of temperature electronics (4), CO₂ trap (5), reused tube (6), and computer for monitoring and controlling (7). (c) Picture shows EA (8) connected with the graphitization system which is comprised of six-unit reactors. Each reactor has an oven (9), a glass tube (10), and a pressure transducer (11). Gas inlet of CO₂ (12) and H₂ (13). (d) Diagram shows the schematic graphitization system.

It is also widely applicable that iron powder is used to catalyze graphite production from CO₂ with H₂ (Vogel et al. 1984). Moreover, the CO₂ gas from organic sample combustion using an EA, and then coupled to the graphite reduction system is developed universally (Aerts-Bijma et al. 2001; Wacker et al. 2010). Many AMS ¹⁴C labs are equipped with automatic graphitization system (e.g., AGE by *Ionplus*). However, the cost of such a system is a concern for our lab. Here we present the first establishment of a graphitization system with the H₂ reduction method and its operating results from the past a year or so.

Configuration

Our homemade graphitization system assembled by NTUAMS Lab has a compact design at a low cost (US\$27,000 not including the EA). All devices, including six reactors with oven, vacuum lines, turbo pumps, electronics components, etc., are built up on a 3-tier wheeled rack (500×900×1100 mm) (Figure 1). The system is connected with a EURO EA made in Germany, and also CO₂ can be brought into the system from an independent gas inlet. Six reactors are aligned parallel and each one is comprised of a “glass tube” with iron powder placed at the bottom (Figure 1c-10), a “reused tube” for the water trap (Figure 1b-6), and a DCbox

BVS-N11 pressure transducer (0–2000 mbar). Two tubes are made of SIMAX 70 mm long tube with 6 mm outer diameter (OD), and 4 mm inner diameter (ID). The volume of each reactor including two tubes is estimated at about 5.2 mL based on the ideal gas law. The “glass tube” is horizontally placed in an oven (Figure 1c-9), and the “reused tube” is vertically connected with the “glass tube” for purification purpose. A temperature sensor is in the oven to record the actual temperature during the reaction process. A vacuum line is built up from 1/4” OD 316L stainless steel and connected to the reactors with FITOK® pneumatic diaphragm valves. The vacuum system contains two low vacuum stations with a Pfeiffer Vacuum MVP 015-4 diaphragm pump and an EDWARDS nXDS10i dry scroll pump, and a high vacuum pumping system using an EDWARDS nExt240 turbo molecular pump. The pressure measurement is read by an EDWARDS Wide Range Gauge that offers the capability range from the atmosphere to 10^{-8} mbar. The background pressure of the graphitization system is achieved around low 10^{-7} mbar. The H₂-injected flow is controlled by Swagelok NUPRO SS-4BMG-VCR Metering Bellows Sealed valve. All valves as well as temperature and pressure electronic sensors are automatically controlled and monitored by HAPS (HQT Auto Process System) program that is commercially available from HQT Inc.

GRAPHITIZATION PROCEDURES

Iron Conditioning

The iron catalyst (-325 mesh, Alfa Aesar®) is weighed in a range of 3–4 mg and then placed into the bottom of a “glass tube”. At first, the iron powder is heated with air for 150 s at 500°C; afterwards wasted gases are pumped away until the system reached below 35°C. Then, to inject pure H₂ with 800 mbar into the reactor, baking at 500°C for 30 min. The process would be effective in activating the catalyst to remove any present carbon-bearing contaminations and iron oxide by reduction mechanism (Němec et al. 2010; Wacker et al. 2010).

Sample Preparation and Combustion

In order to test the new H₂ reduction graphitization system, the organic carbon background made from anthracite (BKG), oxalic acid standards (OXII, NIST SRM 4990C), and intercomparison samples L and M of FIRI (distributed by the University of Glasgow during the fourth international radiocarbon intercomparison) are adopted. All samples containing about 0.3–1.5 mg of carbon mass were wrapped in tin capsules (6×4 mm, OEA labs, United Kingdom). The wrapped samples are combusted using the EURO EA in CNH mode. Pure helium is used as the carrier gas in the graphitization system to avoid air in the system. The temperature of 1030°C in the front furnace and oxygen injection for 8.7 s are set up to ensure complete decomposition of the samples. Excess oxygen is consumed in the reduction column with copper at 650°C.

Loading CO₂ Sample into the Reactor

The combustion gases, such as N₂, CO₂ and H₂O, are produced by EA and flush along with helium carrier flow into the CO₂ trap of the graphitization system. The abundant nitrogen is pumped away and the resultant CO₂ and H₂O are trapped with liquid nitrogen (LN₂) cooled trap. Next step, the dry ice/ethanol slurry with a temperature of about -60°C replaces LN₂ to keep the trapped H₂O frozen. Finally, the pure CO₂ is transferred into the “reused tube” of a selected reactor cooled by LN₂. The pressure of CO₂ sample is recorded when the CO₂ is filled

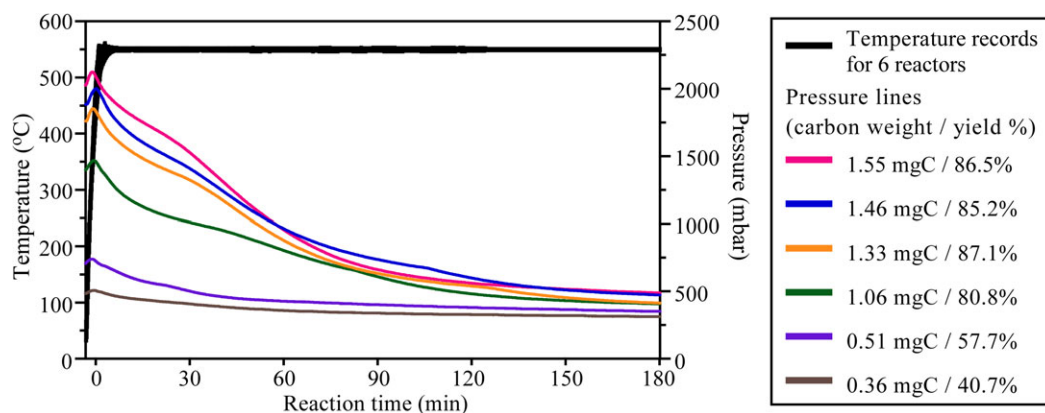


Figure 2 Reduction performance of the graphitization system. Samples with carbon weight of 0.36–1.55 mg show that the graphitization reaction can be finished within 3 hours, but samples with carbon weight less than 0.36 mg are unable to provide reliable graphite due to low yield.

into the entire reactor at room temperature. After the reactor is cooled again with LN₂ to freeze CO₂ sample, inject H₂ of about 2.4 times the recorded CO₂ pressure.

The CO₂ trap is utilized for transferring CO₂ into six reactors. To prevent cross contamination, the CO₂ trap is heated to 120°C before processing the next sample. The system is then evacuated by the high vacuum pump for 10 min to ensure that the graphitization system is clean. To examine the memory effect, two sample batches, including 6 BKG samples and 6 OXII standards, were tested in July 2022. The arrangement was that every OXII standard was followed by a BKG sample. If the cleaning procedure is proper, the background results should not be affected by OXII (modern carbon). Such a test shows that the graphite samples produced from BKG samples displayed a low background with an average of 0.49 ± 0.22 pMC, indicating that the former sample was unable to pollute the next one. After all of six reactors are loaded with CO₂ and H₂, the system will process graphitization.

Graphitization and Its Pressure Yield (%)

After loading the CO₂ sample into the reactor, the graphite reduction is carried out at the temperature of 550°C for 3 hours. Meanwhile, the “reused tube” is immersed in the dry ice/ethanol slurry at about –60°C to keep the reduction product water frozen and ensure the good efficiency of the reduction process. Once the reduction time is finished, the ovens are turned off and the system is evacuated automatically. The temperature and pressure of the six reactors are respectively monitored and logged data once in 2 seconds. For each reactor, CO₂ pressure of about 390 mbar corresponds to 1 mg carbon mass. Figure 2 displays a case of the temperature-pressure-time record for 0.36–1.55 mg carbon samples during the graphitization. All pressure lines decreased gradually and then stabilized within about 2.5 hours, indicating that three hours of is sufficient for completed graphitization.

The pressure yield (%) is an indicator of the efficiency of graphitization suggested by Rinyu et al. (2007). The case results (Figure 2) showed that the pressure yield is estimated to be 40.7% when the sample contains only 0.36 mg carbon. The produced graphite has an unacceptable ¹²C³⁺ current and a relatively large error in ¹⁴C counts. In contrast, the 0.51–1.55 mg carbon samples which have better pressure yield in a range of 57.7–87.1%. The graphite can present an

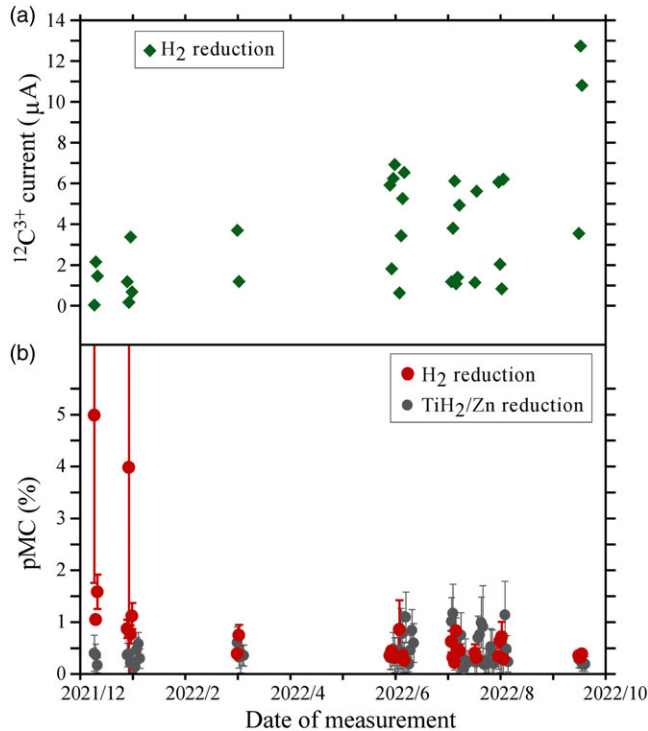


Figure 3 (a) AMS $^{12}\text{C}^{3+}$ currents of the graphite, produced by the H_2 reduction method, from the anthracite background (BKG) samples. (b) The pMC results using two reduction methods are shown in different colors. The higher pMC values are due to the weak $^{12}\text{C}^{3+}$ current. The blank of the AMS is an average of ~ 0.08 pMC.

optimal $^{12}\text{C}^{3+}$ current of 1.59–5.47 μA and provides reliable statistical results for AMS measurement. Therefore, our H_2 reduction graphitization system is appropriate for samples containing a range of 0.5–1.6 mg carbon and capable of obtaining good quality graphite.

RESULTS AND DISCUSSION

Uncertainty Test

To ensure the reliability of the graphitization system, standards of 6 OXII, 4 OXI, and 2 ANU sucrose were processed by our new device and carried out in the same wheel for AMS measurement. The average values of OXIs and ANUs are 104.47 pMC and 153.18 pMC, respectively, when using OXII results as a primary standard. The measured value of OXI is in good agreement with the accepted values, but the ANU result is slightly higher than the consensus value of 150.61 pMC (Rozanski et al. 1992). The activity ratio of OXII/OXI for our measurement is 1.2833 ± 0.0009 which is close to the reported ratio of 1.2933 ± 0.0004 (Stuiver, 1983).

Background

From December 2021 to October 2022, a total of 32 BKG samples were run by the new H_2 -reduced graphitization system. The results showed a declining trend in pMC values over time (Figure 3b). At the beginning of the test (before March 2022), the samples showed higher

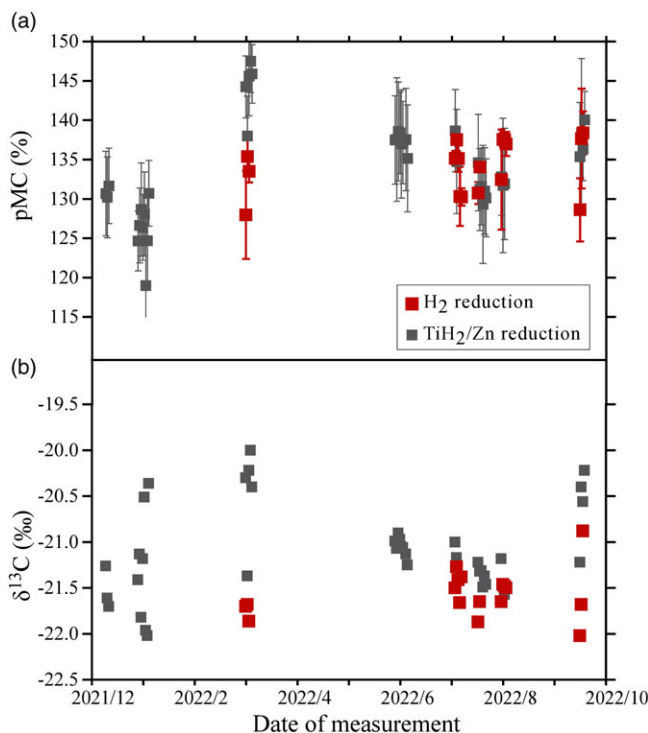


Figure 4 (a) The pMC results from the OXII standards using H₂ and TiH₂/Zn reduction methods respectively. The pMC results of OXII standards were subtracted by a value of AMS background (0.08 pMC). (b) The difference in δ¹³C suggests that the carbon isotope fractionation during the graphitization resulted from the different reduction methods. The isotope fractionation using AMS δ¹³C corrected to -17.8‰ .

values with an average of 2.05 ± 1.71 pMC ($n=7$). It is mainly attributed to the weak $^{12}\text{C}^{3+}$ current (below $1 \mu\text{A}$) due to the poor quality of graphite (Figure 3a). Those graphite samples contained less than 0.4 mg of carbon mass. After the initial test, 25 BKG samples generated relatively lower values, ranging from 0.86 pMC to 0.22 pMC with an average of 0.44 ± 0.18 pMC. The $^{12}\text{C}^{3+}$ current of those samples is higher than $1.0 \mu\text{A}$ resulted in a relatively small statistical error, representing good quality graphite. The background samples with the carbon mass of 0.5 – 1.6 mg yielded an average of 0.38 ± 0.10 pMC, which implies that our new H₂ reduction system is able to date samples within ^{14}C age of 45,000 yr BP.

For comparison, 74 BKG samples were produced to graphite through the TiH₂/Zn reduction method during the same period in the NTUAMS Lab. The results showed a stable range with an average of 0.43 ± 0.25 pMC, which is equivalent to a ^{14}C age of 44,000 yr BP (Figure 3b). The comparable results from two different reduction methods suggest that the background test of our new H₂ reduction graphitization system is reliable.

OXII

Figure 4 shows the comparison of the graphite of OXII standards produced using H₂ (red color) and TiH₂/Zn (black color) reduction methods respectively. The produced graphite of 17 OXII standards by the H₂ reduction method, ranged from 127.98 pMC to 138.38 pMC with a

Table 1 ^{14}C dating results of samples from the fourth international radiocarbon intercomparison (FIRI) through the H_2 reduction method. The age is within 1σ uncertainties.

Standard	Date of measurement	n	Average $^{12}\text{C}^{3+}$ current (μA)	Average measured values (^{14}C yr BP)	Consensus value ^a (^{14}C yr BP)
FIRI-M	2022.03	3	0.7	11711±233	11139±49
	2022.08	2	3.1	11169±57	
FIRI-L	2022.08	4	5.1	2526±28	2505±39

^aThe consensus values of intercomparison samples are from Naysmith et al. (2019) and Scott et al. (2019)

relative standard deviation of 2.57% (Figure 4a). For the results of the TiH_2/Zn reduction method, 39 OXII standards were graphitized and showed between 124.6 pMC and 147.48 pMC with a scatter of 4.61%. Besides, the isotope correction using measured AMS $\delta^{13}\text{C}$, showing the $\delta^{13}\text{C}$ of $-21.57\pm 0.26\text{‰}$ for H_2 reduction method as well as $-21.09\pm 0.50\text{‰}$ for TiH_2/Zn reduction method (Figure 4b). The $\delta^{13}\text{C}$ results of H_2 -reduced graphite are slightly lighter and less scattered than those of TiH_2/Zn reduction graphite. It indicates that the carbon isotope would occur differently fractionated during the graphitization reaction due to the different reduction processes. The degree of fractionation is associated with the carbon content in a sample, the amounts of reagents for reduction, temperature, and time of the process (Xu et al. 2007; Marzaioli et al. 2008; Macario et al. 2017). The similar observation regarding the lighter $\delta^{13}\text{C}$ graphite generated by the zinc reduction was also documented by Xu et al. (2007). Although the corrected $\delta^{13}\text{C}$ from both methods are lighter than OXII standard (-17.8‰), the constant AMS $\delta^{13}\text{C}$ over a range suggested that the fractionation effect can be evaluated as long as the samples are processed in the steady condition.

Intercomparison Sample

In this study, the sample M and sample L of the fourth international radiocarbon intercomparison (FIRI) were adopted for graphitization system testing. FIRI-M is a whole peat sample from a coastal cliff deposit at St Bees Head in northwestern England which had been exposed by erosion. The consensus ^{14}C age is 11139±49 yr BP (Naysmith et al. 2019). FIRI-L is a wood sample from the burial mound of Dogee Barrow, grave 8 (the Tuva king barrows from Scythia), and its reported ^{14}C value is 2505±39 yr BP (Scott et al. 2019).

The initial test result on March 2022, three measured ^{14}C age of sample FIRI-M was in an average of 11711±233 yr BP which has a large variation to the reported age (Table 1). Those samples produced poor-quality graphite with a weak $^{12}\text{C}^{3+}$ current below 1.0 μA . Two measurements in August of 2022 were significantly improved, yielding an average ^{14}C age of 11169±57 yr BP which is identical to the consensus value. In addition, the average measured ^{14}C age of sample FIRI-L is 2526±28 yr BP based on four measurements. The measured ^{14}C ages of intercomparison samples achieved a comparable result with the consensus values, indicating that our graphitization system is capable of providing a valid dating quality on organic material.

Case Study of JCE Peat Core Samples

A case study of JCE core drilled in Jinchuan Mire of Jilin Province, northeastern China was carried out with the semiautomatic graphitization system. Four moss samples were picked up

Table 2 Results of ^{14}C ages from the JCE core, Jinchuan Mire of NE China. The error for pMC (percent Modern Carbon) and ^{14}C age is given 1σ uncertainties. The ages were calibrated by IntCal20.

Lab code	Sample ID	Depth (cm)	pMC (%)	D ^{14}C (‰)	^{14}C age (yr BP)	Calibrated ^{14}C age (cal BP)
EAAMS-44	JCE_68-70	69	94.81±0.59	-51.9±0.3	428±50	485±60
EAAMS-45	JCE_78-80	79	88.98±0.55	-110.2±0.7	938±49	835±95
EAAMS-46	JCE_88-90	89	84.13±0.58	-158.7±1.1	1388±55	1315±70
EAAMS-47	JCE_98-100	99	87.52±0.51	-124.8±0.7	1071±47	990±85

from the depths of 68–70, 78–80, 88–90, and 98–100 cm of the core. Those organic samples were graphitized by H_2 reduction method using the new graphitization system and subsequent AMS ^{14}C dating in the NTUAMS Lab. The calibrated ^{14}C ages are processed on Behron program (Haslett and Parnell 2008) which is using IntCal20 calibration curve for calendar age calibration. The AMS results show an increasing trend in ^{14}C ages with depth (Table 2). However, the older ^{14}C age of about 1315±70 cal BP at the depth of 88–89 cm is out of depositional sequence. The old carbon influence on the ^{14}C age of the peat samples has been studied by Li et al. (2019) and Misra et al. (2023): dissolved CO_2 in peat water produced by the decomposition of older peat plants can be uptake by some peat roots during photosynthesis.

A 92-cm-long JCA core was taken nearby the JCE core site in the same peatland. Li et al. (2019) proposed the AMS calibrated ^{14}C ages of the JCA core, ranging from the modern time at the topmost to 880 cal BP at the depth of 87.5 cm. The AMS ^{14}C dating results of the four moss samples from the JCE core could correspond to the JCA core below 60 cm depth. Therefore, the depositions and chronologies between the JCA and JCE profiles should be comparable.

CONCLUSION AND OUTLOOK

A semiautomatic graphitization system has been well-developed at the NTUAMS Lab which has been running steadily for over a year. The reliability of the instrument is well-proven by the accuracy and repeatability in AMS ^{14}C ages and isotope ratios, using the backgrounds, intercomparison samples and standards. The entire procedure of iron conditioning, sample combustion, gas loading, and graphitization takes about 7 hours for six samples. The operation of graphite reduction with H_2 allows for the efficient production of high-quality graphite from organic materials containing 0.5–1.6 mg carbon mass.

It is optimistic to make more improvements in the future. The production of high-quality graphite requires a sufficient carbon content (>0.5 mg), therefore it is inconvenient for materials with low-carbon content such as sediments, which demand a large amount of sample weight. To address this issue, a smaller reactor volume will be designed in the future to enhance the reaction pressure and increase graphitization efficiency. In addition, the graphitization system is anticipated to be fully automated, and will be tested on carbonate samples.

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