

ACCELERATOR MASS-SPECTROMETER SB RAS

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The accelerator mass spectrometer (AMS) created at BINP SB RAS is installed at CCU "Geochronology of the Cenozoic era" for sample dating by the ¹⁴C isotope and for biomedical applications. The most distinguishing features of BINP AMS is the use of the middle energy separator of ion beams, the magnesium vapors target as a stripper and time-of-flight telescope with thin films for accurate ion selection. Present status of AMS complex and the results of experiments for radiocarbon concentration measurements in test samples are presented.

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INTRODUCTION

Accelerator mass spectrometry – ultrasensitive method of radiocarbon analysis of matter used in various fields of science. Created in BINP AMS [1 - 5] is based on an electrostatic tandem accelerator with ion separation at medium energies, on magnesium vapor target as a stripper and time time-of-flight telescope with thin films for accurate identification of the ions.

The ion movement in a AMS is represented as follows. Negative ions are accelerated in the horizontal ion source to the injection energy, then turned by 90° in the magnetic field and accelerated straight up in the first accelerator tube to the positive potential of the high-voltage terminal. The ions are stripped to a positive charge in the on magnesium vapor target. Then ions are turned by 180° in the electric field, and accelerated down in the second accelerator tube to ground potential. The accelerated ions are turned by 90° in the magnetic field and horizontally entered into the particle detector.

In this configuration of the AMS, the ions background is significantly reduced by the energy filter in the high voltage terminal [6, 7]. For example, the negative nitrogen ions are unstable, but the nitrogen atoms can accelerate as part of the molecules, which can be in the negative charge state. Molecular ions formed from the residual gas in the first accelerating tube can be drawn into the acceleration. Such ions after passage of the first accelerating tube will have a lower energy than the analyzed ions. That is because the kinetic energy of the fragments of the molecule is always smaller than the energy of the molecule as a whole and the charge state of -1 is largest for acceleration in the first tube. The filtration of the ions by energy is very effective in the high voltage terminal, because the energy of ions background in the high voltage terminal is always less than the energy of the analyzed ions. Without the energy filter in the high voltage terminal, nitrogen ions of molecular fragments freely falling in the second accelerating tube, can compensate for the lack of energy by flying part of the tube in a higher charge state than the analyzed ions. Thus the energy of the analyzed ions and of background ions can be equal at the exit of AMS, and the subsequent separation is possible only by nuclear detector through the difference in energy loss during the passage of the substance. Not only the background ions of nitrogen from the fragments of molecules, but other fragments of molecules reliably filtered out in AMS SB

RAS. Another feature of our project is to use the magnesium vapor target [8] as a stripper. This target does not affect to the vacuum outside the heated volume. Degradation of the vacuum conditions, particularly in accelerator tubes, causes an increase ions background. Using TOF telescope at the exit of AMS allows not only to identify the ions by time of flight, but also clean up "technical" ions background from high-voltage breakdown by registering the arrival time of the particles in the detector.

Since 2009, the AMS SB RAS is used for radiocarbon dating of archaeological and geological samples. Over the past year have been analyzed more than 600 samples. The improvement of methods of AMS sample analysis, upgrade of elements of accelerator, replacement of the electronic units on a more reliable and stable are permanently carried out accordingly the financial capacity of AMS complex. Given the interest in using AMS in the biomedical field, recently carried out the adaptation of the AMS to biomedical applications. The first biomedical researches are started jointly with biologists.

AMS-ANALYSIS OF ARCHAEOLOGICAL AND GEOLOGICAL SAMPLES

The preparation procedure of the ion source (IS) and of the samples in the IS for AMS-analysis is necessary for the stable operation of the complex. The sputter ion source with a wheel rotation for 23 positions is used at AMS SB RAS. The similar IS, but without wheel rotation was used before and has been described [9]. The vacuum in system mainly provided by ion pumps is about 10⁻⁶ Torr. However, the turbomolecular pump is used at the exit of IS, because the ion pumps are not suitable for pumping out containing carbon gases, by reason of contamination by carbon, leakage currents, breakdowns, etc. Until recently, backing pump with oil is used with a turbomolecular pump. Although the carbon from the oil is weakly influenced on the sample measurement results, however, for safety, such a pump has been replaced by an oil-free.

The working vacuum is reached in about 30 minutes after installation of the samples in the sample wheel and replacement of the cesium in the oven. However, the degassing components of IS is required by heating the ionizer and cesium oven (with not broken ampules with cesium) to a temperature of about 10% above the oper-

ating mode. This process takes about 1.5 hours. Typically samples are measured the next day after degassing. It should be noted that although the ampules with cesium are not destroyed by heating the oven, however, cesium beam appears briefly by heating the ionizer (if not performed a thorough cleaning of the IS). During IS degassing, the graphite MPG in sample wheel is rotated under the cesium beam to avoid destruction of the samples. The graphite MPG is also used for "activation" of IS (with crushed ampules with cesium, before measuring samples). Slow rotation of the samples wheel is used, when a carbon current appears from PGM graphite. A typical time dependence of the carbon current from the samples is shown in Fig. 1.

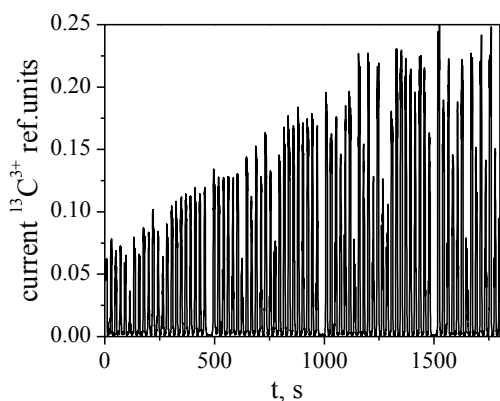


Fig. 1. The increase in the carbon current from the samples during first (after the change of samples) rotation of samples wheel under the beam of cesium

This process is accompanied by degassing, with a gradual increase in the current in all the samples, which we assume is due to the accumulation of cesium on the surface of samples required for the efficient production of negative ions of carbon. After the currents of the samples come into saturation, in the future, there are no rapid changes in carbon currents when installing a cesium beam. This is important for AMS-analysis based on switching measurement of carbon isotopes. In addition, the surface of the samples is cleaned by the cesium beam from possible contamination by external carbon before the measurement is started.

The 20 graphitized samples are setted in the IS sample wheel to measure the concentration of radiocarbon. Furthermore, the 3 control sample with a known concentration of radiocarbon is setted in IS sample wheel for control and normalization of the measurement samples. Typically, this sample of two carbon fiber with a carbon concentration on the natural content of modern plants and one sample of graphite MPG with radiocarbon concentration at $2 \cdot 10^{-3}$ compared to modern plants. It should be noted that the control samples did not require the procedure of graphitization and setted in the IS sample wheel in natural form. For this, using graphite from a single piece of machined graphite cylinders for installation in an aluminum piston of IS. The carbon filament is placed in an aluminum tube, then tube is radially compressed and then cut into cylinders for use in IS sample wheel.

The radiocarbon ions sputtered from samples, going through all stages of selection, is detected time in time-of-flight telescope [10] at the exit of AMS. Fig. 2 shows

a typical TOF spectra of modern (carbon fiber) and "dead" (graphite MPG) carbon samples. One recording channel of the detector is 70 ps. The location of the main peak (840 channels) corresponds to the time of flight of the radiocarbon ions. The location of the nearest background peak of the ion ^{13}C corresponds to a channel 810. It is seen the radiocarbon peak is dominant for modern sample, the influence of background ions is negligible. For the "dead" sample, the peaks of background ions are comparable to the peak of ^{14}C , but they are significantly separated in TOF space.

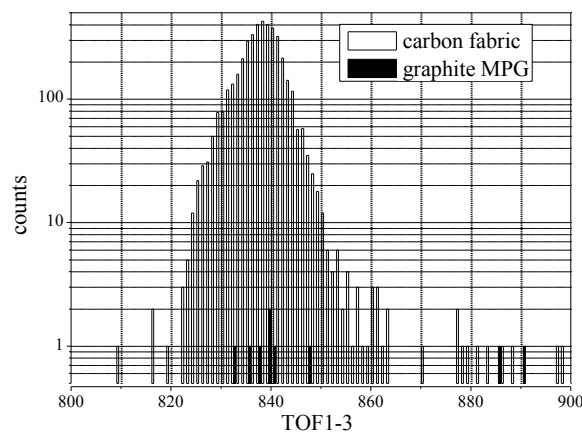


Fig. 2. TOF spectra for modern and "dead" carbon samples

When measuring the concentration of radiocarbon in the samples, the switching algorithm is used. The isotope ^{14}C are detected by TOF telescope and ^{13}C currents are measured at the exit of AMS. For switching algorithm the high voltage of IS is changed. The energy of the cesium ions remains constant. The electrostatic lens and correctors at the exit of the ion source are changed for each isotope. Thus, the passage of isotopes is carried out through a first dipole magnet, without changing the magnetic field. Note that, for reasonable changes of energy cesium, the carbon current is approximately the same, however, the change in energy cesium leads to the transient, lasting for about a minute, for which the carbon ion current can vary by several tens of percent. In recent years, the passage algorithm for different isotopes through the dipole magnet at the output of UMC is changed. Initially, the magnetic field of exit magnet was switched for different isotopes. The movable beam probe with Faraday cup and was used for measuring ^{13}C current. Although this algorithm works well, however, we were unable to achieve adequate reliability of the moving probe. It was possible to hit an intense beam of stable isotopes in the TOF detector, leading to the destruction of the films used in detector. Currently, radial aperture at the exit of AMS was increased that allowed to pass through the magnet the adjacent mass isotopes. The FC and TOF were permanently installed. It is noted that with new ^{13}C measuring algorithm, the TOF detector registers ^{13}C ions from energy tail with approximately 100 kHz, however, this load is not dangerous for the films and the detector as a whole. And given that measurement ^{13}C and ^{14}C are spaced in time, this background count are not affected to the measurement of radiocarbon concentration.

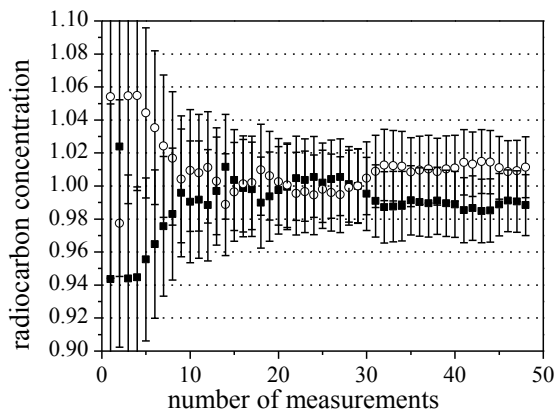


Fig. 3. An example of a set of statistics in measuring of the radiocarbon concentrations in modern samples

The cycle of AMS-analysis of samples is represented as follows. For each sample, the ^{14}C ions are twice counted (20 seconds each) and twice the ^{13}C currents are measured. After that, the samples wheel is turned to the next sample for process repetition. Measuring of whole sample wheel (23 samples) takes about 20 minutes. For a set of statistics the wheel are moving to the second turn, third, etc. Typically, the measurement will take approximately 8 hours, with a statistical error of measurement for modern samples of approximately 2%. An example of a set of statistics for the two samples of carbon fibers is shown in Fig. 3. Typically, the next day the cycle repeats itself measurements are compared to the results of measurements for different days, if they are within the statistical spread, the final result is given as a set of data for all measurements of these samples.

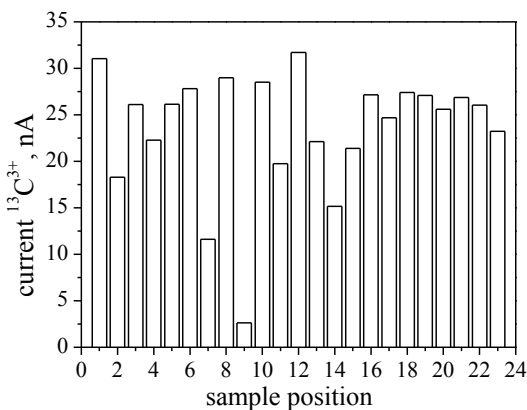


Fig. 4. Average ^{13}C current during the measurement for graphitized natural samples

The typical average ^{13}C current at the exit of AMS for each samples are shown in Fig. 4. Here is the average current from the samples in 8 hours of measurement. Typically, the maximum current is obtained from carbon filaments without sample preparation (see positions 1 and 12 in Fig. 4). The currents from prepared (graphitized) archaeological and geological samples are in the range from carbon fibers currents level to much smaller level. At the time, the carbon currents from graphitized samples varies are not in sync, probably this depends on the special features of graphitization for each sample. It should be noted that a very small current from the samples is not often, approximately one sample for 50 graphitized samples. The contamination level

in the samples during the sample preparation procedure estimated by the radiocarbon content in graphite after combustion and graphitization (the radiocarbon concentration in graphite is insignificant before this procedure). Typically, the quantity of pollution (radiocarbon concentration) composes approximately 1% of the concentration level in the modern plants, which corresponds to 40 000 radiocarbon years of age. However, sometimes there are "epidemic", when the amount of contamination in the sample preparation procedure for graphite is 10 percent or more. In such cases, it is believed that the radiocarbon concentration in all samples into sample wheel does not reflect the concentration of radiocarbon in the original object. After cleaning the sample preparation equipment and graphitization of new samples, the process of analyzing of samples is repeated. Note that the radiocarbon concentration of graphite MPG without sample preparation procedure does not depend on these processes and is 0.2%. The experience of the analysis shows that the presence of small pollution into graphitized samples of graphite does not always guarantee the absence of contamination in the graphitized natural objects. Probably to improve the reliability of the analysis necessary graphitize known natural objects of the same type of the analyzed samples of material (bone, charcoal, etc.) and use them to monitor sample preparation instead of pure graphite. In addition, it is necessary to manufacture independently two samples for each archaeological samples, and only at a reasonable coincidence in analysis of pairs of samples assume that the radiocarbon concentration in samples after sample preparation procedure corresponds to the initial archaeological sample. However, for reasons beyond the control of the authors of such methods to improve the reliability and validity of the analysis has not yet been implemented.

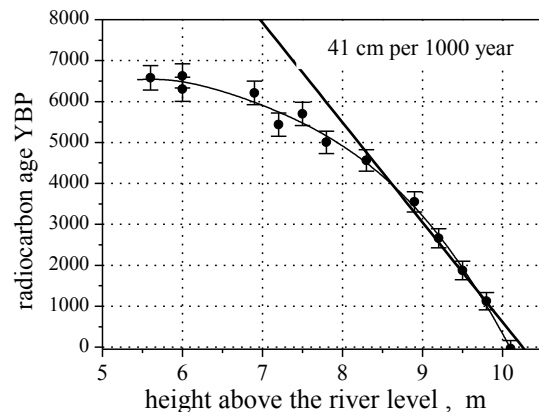


Fig. 5. The radiocarbon age of peat deposits, depending on the height above the river level

As an example of AMS-analysis, the data from geological samples – peat deposits of the river Dem'yanka, Tyumen region, depending on the height above the river level (samples of Leshchinsky VS, TSU), presented in Fig. 5. Such analyzes are necessary to obtain a timescale for peat deposits. Such results are quite revealing, since in the absence of mixings deposits should be observed dependence – the deeper the ancient.

SUMMARY

The AMS SB RAS created in BINP for reliable identification of radiocarbon ions from the ion background is used for AMS-analysis of archaeological and geological samples. The algorithm of the AMS-analysis of the samples and the present status of complex are described.

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УСКОРИТЕЛЬНЫЙ МАСС-СПЕКТРОМЕТР СО РАН

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Созданный в ИЯФ СО РАН ускорительный масс-спектрометр (УМС) установлен в ЦКП «Геохронология кайнозоя» для датирования образцов по изотопу ^{14}C и биомедицинских применений. Сепаратор ионов на средних энергиях, мишень на парах магния в качестве обдирочной мишени и телескоп тонкопленочных времяпролетных детекторов являются отличительными особенностями УМС ИЯФ для надежной селекции ионов. Представлены текущее состояние комплекса УМС и результаты экспериментов по измерению концентрации радиоуглерода в тестовых образцах.

ПРИСКОРЮВАЛЬНИЙ МАСС-СПЕКТРОМЕТР СО РАН

С.А. Растігеев, А.Р. Фролов, А.Д. Гончаров, В.Ф. Клюєв, Е.С. Константинов, Л.А. Кутнякова, В.В. Пархомчук, А.В. Петрожицький

Створений у ІЯФ СО РАН прискорювальний мас-спектрометр (ПМС) встановлено у ЦКП «Геохронологія кайнозою» для датування зразків по ізотопу ^{14}C і біомедициних застосувань. Сепаратор іонів на середніх енергіях, мішень на парах магнію в якості обдирної мішені і телескоп тонкоплівкових часопролітних детекторів є відмінними рисами УМЗ ІЯФ для надійної селекції іонів. Представлені поточний стан комплексу УМЗ і результати експериментів з вимірювання концентрації радіовуглецю в тестових зразках.