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Nuclear Instruments and Methods in Physics Research B 223–224 (2004) 645–649
NIM B
Beam Interactions with Materials & Atoms
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The AMS dating of pollen from syngenetic ice–wedge ice

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Abstract

The features of pollen occurrence in ice–wedge ice such as: size of pollen of tundra plants, incoming pollen into frost cracks together with melt water, dust and partially from host sediment and also clay envelopes around pollen grains caused the pretreatment methods of ice–wedge ice samples. Good preservation of thin-wall pollen grains of *Salix* and Liliaceae after pretreatment evidenced appropriate pretreatment procedure. There is evident correlation between pollen dates and re-deposited pollen and spores content. However an interpretation of ^{14}C dating of pollen concentrate is required independent time scale such as annual laminas or the AMS dates of macro or microfossils.
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PACS: 06.60.Ei; 92.40.Sn; 93.30.Sq; 91.35.Nm
Keywords: Pollen; Ice wedges; AMS; ^{14}C; Siberia

1. Introduction

Pollen is well preserved in ice–wedge ice due to low temperature, stable condition of this close system and low microbial activity. ^{14}C dating of fossil pollen grains which are contained in ground ice could give new information about past environment. The pollen concentration in ice wedges is very close to those of Arctic ice caps approximately 10–100 grains/l [1]. Similar to Arctic ice caps the pollen assemblages from ice wedges in arctic and sub arctic tundra are composed of a high percentage of long distance transported pollen, primarily tree pollen. The regional tundra pollen input is essential also, and local pollen contribution is very small. The penecontemporaneous pollen grains and spores also found in ice wedges. It is usually possible to collect only 1–3 mg of pollen concentrate from several kilograms of the ice–wedge ice.

To follow the way of pollen assemblage formation we apply Vasil'chuk's multistage model of large syngenetic ice–wedge ice formation. The ice wedges form with big pulses of sub aqueous deposition alternating with sub aerial conditions of ice wedge growth [2]. Snow melt water at sub aerial stage and snow melt water with admixture river or lake water at sub aqueous stage are the main source for ice wedges. Water comes into frost cracks in spring, when snow melting and flooding take place. At sub aqueous stage there is a possibility for incoming of penecontemporaneous pollen and spores into ice wedges. Average percentages of penecontemporaneous pollen and spores in river

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doi:10.1016/j.nimb.2004.04.119

water in lacustrine and alluvial deposits in the North-West Siberia are about 10–15%.

Alkaline extract and micro organic inclusions (>100 μm) from syngenetic ice wedges in Seyaha cross-section were dated by AMS earlier [3,4]. The dates of micro-organic in all cases are younger than the dates of alkaline extracts, i.e. it is obvious a good safety of ancient organic matrix as presented in alkaline extract. However the number of dates from micro organic inclusions in ice–wedge ice shows that ^{14}C age decreased from bottom to the top. Evidently, micro organic inclusions are simultaneous to ice-wedge formation and could be reference for further dating of the cross-sections.

2. Regional setting

Seyaha cross-section located on the east coast of Yamal Peninsula (70°09′ N, 72°34′ E) in moss-dwarf shrub-sedge mezic tundra near the boundary between arctic and subarctic tundra (Fig. 1). Mean winter temperatures −16.4 °C, frost season 255 days. This sequence with a depth of 22–24 m is especially valuable for palaeo-geographical reconstruction thanks to the abundance of multistage ice wedges. The sediments accumulated continuously during almost 20,000 years: from 30,000 to 11,000 yr BP (according to ^{14}C dates of the host sediments) (Fig. 2) [3,4].

3. Methods

Every variety of sediments required some modification of methods of samples pretreatment

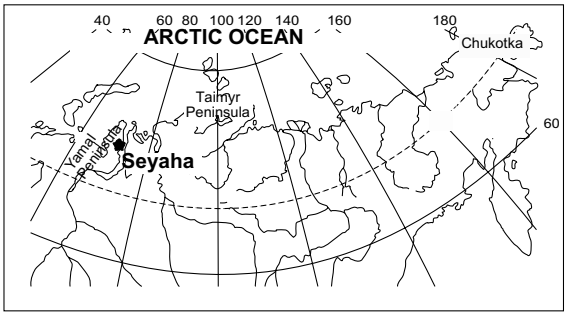

Fig. 1. Location of Seyaha cross section.

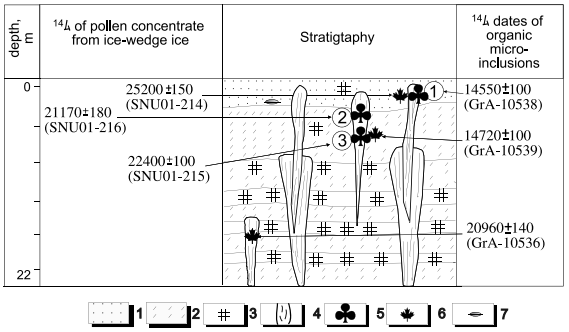

Fig. 2. ^{14}C dates of pollen concentrate, and micro organic (<100 μm) from Late Pleistocene syngenetic ice wedges in Seyaha cross-section, Yamal Peninsula: 1 – sand, 2 – sandy loam, 3 – allochthonous peat, 4 – ice wedge, 5 – sampling points for AMS ^{14}C dates of pollen concentrate, 6 – sampling points for AMS ^{14}C dates of organic micro inclusions (<100 μm), 7 – point of foram finding.

to find the optimal way of pollen and spore extraction [5–10]. The same samples from a Seyaha cross-section were used, as in [3,4] to work out a procedure of pollen concentrate extraction from ice–wedge ice for radiocarbon AMS-dating.

Our technique of pollen extraction from samples of ice–wedge ice is based on previous methods of samples pretreatment for AMS dating. The first results of pollen dating from sediments were obtained in 1989 by Brown [5]. In 1992 Long et al. [6] developed the technique of pollen concentrate purification from lacustrine sediments in hard water lakes. Also Regnell [7] worked out a special technique for pollen concentrate from a hard-water lake in Southern Sweden for AMS dating. The date of the bulk sample of the lacustrine sediments also was older in comparison with date of pollen.

There are some features of pollen occurrence in ice–wedge ice: such as size of tundra plants pollen, incoming pollen into frost cracks together with melt water, dust and partially from host sediments, and also clay envelopes around pollen grains of local species of plants. The pretreatment procedure of pretreatment ice–wedge ice samples includes both 40% HF pretreatment and centrifugation with heavy liquid. The main task of our methods was to remove organic admixtures and quartz debris and to prepare relatively pure pollen concentrates without using carbon-based chemicals.

At the first stage 10% KOH solution was used at 85 °C for 1 h for deflocculating of organic matrix and removing some of the diatoms and humic acids. Then the samples were washed on the precision-woven polyester meshes of the size 100 and 60 μm for further removing fibrous fraction. A final wash and retention of the sample was on 10 μm mesh because pollen of tundra plants close to this size. Only fraction 60–10 μm was used for further preparing. Silicates particles less than 60 μm were removed by dissolving in 40% HF. HCl (1 N) was used for SiF_4 removing. Then agitation in ultrasonic tank for 10 min improved the pollen concentration due to destroying of clay envelopes around pollen grains. In permafrost sediments and ice–wedge ice the clay particles very often surround the contemporaneous pollen grains and spores. The next step was centrifugation in $ZnCl_2$ solution with density 1.97 g/cm^3 twice for 10 min. This density is well suited for the separation of heavy inorganic fraction with density 2.6 g/cm^3 mostly contained silicates from the light organic fraction with density 1.3–1.5 g/cm^3 mostly consisting of pollen. After washing sample on 10 μm polyester mesh, a treatment with 2–3% $NaClO_2$ solution for 3 min was performed for finally deflocculation of amorphous organic material. This treatment allowed to exclude most part of non-pollen organic matrix, then nearly only the pollen grains persisted in final product. Finding of thin-wall pollen of *Salix* and Liliaceae in pollen spectra evidenced that the most part of pollen preserved after pretreatment. At the last stage we used the methods of micro-tubes collection of pollen [10], the most part of non-pollen organic material was excluded. Finally, the resulting 60–10 μm fraction was dried at 80 °C. After each step of chemical treatment, sample residues were recovered by centrifugation at 3000 rpm and washed with distilled water. We have not obtained enough pollen concentrate to separate it into fractions. All chemicals used were of pro-analysis quality, and only millipore water was used for rinsing purposes. The most part of re-deposited pollen grains identified due to their incompatible stratigraphic range and habitus, color intensity, etc. Finding such pollen in high percentage is a good indicator that the ^{14}C date will be older. Pre-Quaternary pollen and spores were identified according to morphology of fossil pollen and spores from North-West Siberia. In many cases reworked pollen is not obviously older than enclosing deposit, admixture of Quaternary pollen is very difficult to detect. Evidently some part of counted Quaternary pollen grains and spores could not be contemporaneous to ice formation.

4. Results

AMS measurements were performed at Tandetron 4130 AMS Seoul National University (Table 1). The radiocarbon dates are reported in BP. The ^{13}C values are expressed with respect to the PDB standard. Use of the calibration procedure is impossible because the radiocarbon ages were all

Table 1
The first AMS-^{14}C dates of pollen concentrate extracted from ice–wedge ice of Seyaha cross-section

Field number	Depth (m)	Laboratory code	Pollen concentrate age (uncal. yr. B.P.) (re-deposited pollen, %)	$\delta^{13}C$ (‰)	Dates of other organic fractions from the same samples
363-YuV/27	1.8	SNU01-214	25200 ± 150 (19.3)	−25.9	Organic micro inclusions (>100 μm) 14550 ± 100 (GrA-10538) Alkaline extract 19920 ± 130 (GrA-9847)
363-YuV/108	4.8	SNU01-216	21170 ± 180 (6.6)	−32.7	Not measured
363-YuV/87	12.0	SNU01-215	22400 ± 100 (15.4)	−25.1	Organic micro inclusions 14720 ± 100 (GrA-10539) Alkaline extract 23620 ± 160 (GrA-9848)

beyond the reliable part of the commonly available calibration model (INTCAL98).

The results show the inversion. At the depth 1.8 m the ^{14}C date is the oldest (25200 ± 150 B.P.). However, the sample at the depth 4.8 m is dated at 21170 ± 180 BP, the sample located downward at the depth 12.0 m is dated at 22400 ± 100 B.P.

The first sample 363-YuV/27 (dated 25200 ± 150 B.P.). 496 pollen grains and spores have been counted. Percentages of penecontemporaneous Pre-Pleistocene pollen and spores is 19.3%, they are presented by *Liquidambar*, *Pterocarya*, Taxodiaceae, Nudopollis sp., Trudopollis sp., etc. Quaternary pollen assemblage consist of long distance transported pollen (17%) Picea sp. −2%, *Pinus silvestris* −3%, *P. sibirica* 6.5%, Betula sp. −4.5%, Alnus sp. −1%; regional pollen (34.5%) *Betula sect. Nanae* −15%, Alnaster sp. −3%, *Salix* −3% Poaceae −13%, Ericaceae −0.5%, local pollen (4%) Polemoniaceae −1.5%, *Rubus chamaemorus* −0.5%, Apiaceae −0.5%, Lamiaceae −0.5%, Draba sp. −1%, spores 44%. Content of coal particles is 12% from total pollen and spore sum. Main part of older organic contains in the fraction of 10–60 μm.

The second sample 363-YuV/87 (dated 22400 ± 100 B.P.). 260 pollen grains and spores penecontemporaneous Pre-Pleistocene pollen and spores are 15.4% (Pinaceae, Taxodiaceae, Schizaceae). Quaternary part of pollen spectra consist of long distance transported pollen (9%) *Betula sect Albae* −6.3%, *P. sibirica* −2.7%, regional pollen (18.9%) *Betula sect. Nanae* −9%, Alnaster sp. −1.8%, Salix sp. −1.8%, Cyperaceae −1.8%, Poaceae −4.5%, local pollen (46.1%) Liliaceae −8.1%, Sparganium sp. −34.2%, Rosaceae −0.9%, *Polygonum bistorta* −0.9%, spores (24.2%) *Bryales* −21.6%, Polypodiaceae −1.8%, *Equisetum* −0.8%. Remarkable feature of the pollen assemblage is high percentages of local pollen Sparganium sp., which is evidently autochthonous. This indicated inundation. Content of coal particles is 25%. The age of alkaline extract is the oldest. We supposed that the ancient organic material contains in 10–60 μm fraction as re-deposited Quaternary pollen and spores and coal particles.

The third sample 363-YuV/108 (dated 21170 ± 180 BP) from 240 pollen grains and spores re-deposited Pre-Pleistocene pollen and spores are 6.6% (Pinus sp., Carya sp., Taxodiaceae). Quaternary part of pollen spectra consists of long distance transported pollen 12.1% (*Betula sect Albae* −6.6%, *P. silvestris* −5.5%), regional pollen 72.5% (*Betula sect. Nanae* −30.8%, Alnaster sp. −6.6%, Cyperaceae −22%, Poaceae −11.1%, Caryophyllaceae −1.1%, Ericaceae −1.1%), local pollen 3.1% (Liliaceae −0.9%, Sparganium sp. −1.1%, Papaveraceae −1.1%), 13.2% (*Bryales* −6.6%, Sphagnum sp. −1.1%, *Equisetum* −1.1%, *Lycopodium clavatum* −2.2%, *Selaginella sibirica* −1.1%, indeterm. spores −1.1%). Content of coal particles is 5% from total pollen and spore sum. The ^{14}C date of this sample is more reliable due to insignificant content of coal particles and the lowest content of pre-Quaternary palynomophs as indicator of the re-deposition.

5. Discussion

Main problem in pollen ^{14}C dating is penecontemporaneous pollen and spores or another organic matrix in pollen assemblages in permafrost.

Since re-deposited pollen and spores content is 15.4%, 6.6%, 19.3% there is evident correlation between pollen dates and re-deposited pollen and spores content. The youngest date corresponds to the sedimentary environment where the influx of re-deposited pollen and spores was minimized. If we compare the dates of various fraction and select the youngest dates of organic micro inclusions remains (>100 μm) as basis we conclude that dead carbon effected on pollen concentrate age (Table 1). Admixture of "dead" carbon is confirmed by finding of Pre-Pleistocene pollen and spores. Having extrapolated Olsson's curves [11] to establish age of sample with admixture of "dead" carbon we conclude that if admixture of "dead" carbon should be much more than 19.3%. If we supposed the real age of the sample 363-YuV/27 with 19.3% of Pre-Pleistocene pollen and spore is 14550 yr, in order to obtain the date 25 ka B.P. It is evidently that the most part of Quaternary pollen is re-deposited from older sediments. We suppose that this is typical for ice–wedge ice in tundra. Re-deposition processes are common in permafrost conditions. Pollen and other organic

material accumulated in frozen lake bank may be washed out just after accumulation.

However, the ^{14}C dates of pollen concentrate from ice wedges which in forest are the youngest in comparison with dates of micro inclusions (>200 μm) and alkaline extract [4,12] because the concentration of contemporaneous pollen is more than in tundra by tens times.

6. Conclusions

(1) There is evident correlation between ^{14}C dates of pollen concentrate and re-deposited pollen and spores content. The youngest date corresponds to minimum of re-deposited pollen and spores.
(2) Interpretation of ^{14}C dating of pollen concentrate is required independent time scale such as annual laminas or the AMS dates of macro or microfossils.

Acknowledgements

We are grateful to J. van der Plicht and H. Jungner for ^{14}C dating of organic micro inclusions in ice–wedge ice and organic material in host sediments, J.H. Park and J. Kang for AMS-samples preparation.

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