



ELSEVIER

Nuclear Instruments and Methods in Physics Research A 470 (2001) 376–379

**NUCLEAR  
INSTRUMENTS  
& METHODS  
IN PHYSICS  
RESEARCH**  
Section A

www.elsevier.com/locate/nima

# Scanning SR-XRF beamline for analysis of bottom sediments

K.V. Zolotarev<sup>a,\*</sup>, E.L. Goldberg<sup>b</sup>, V.I. Kondratyev<sup>a</sup>, G.N. Kulipanov<sup>a</sup>,  
E.G. Miginsky<sup>a</sup>, V.M. Tsukanov<sup>a</sup>, M.A. Phedorin<sup>b</sup>, Yu.P. Kolmogorov<sup>c</sup>

<sup>a</sup> Budker Institute of Nuclear Physics, Lavrentiev av 11, SB RAS, 630090 Novosibirsk, Russia

<sup>b</sup> Limnological Institute of SB RAS, 664033 Irkutsk, Russia

<sup>c</sup> United Institute of Geology, Geophysics and Mineralogy, SB RAS, 630090 Novosibirsk, Russia

## Abstract

The XRF beamline at the VEPP-3 (Budker INP, Novosibirsk, Russia) storage ring was modified for the performance of scanning analysis. A description and parameters of this facility are presented in the current paper. The initial results of testing of scanning analysis of bottom sediments from Lake Baikal are also presented. A spatial resolution of the scanning analysis is 0.1–1 mm which corresponds to 2–50 years of resolution. Such a scanning analysis of lake sediments provides high quality data as a requirement for paleoclimate reconstructions. © 2001 Elsevier Science B.V. All rights reserved.

PACS: 07.85.Qe; 92.70.G; 92.40.Ni; 91.35.Nm

Keywords: SR-XRF; Trace elements; Microscanning; Lake Baikal; Paleoclimate

## 1. Introduction

Lately, the SR-XRF analysis has been used intensively for studies of bottom sediments of continental lakes with the aim of reconstructing the global climate changes during the last million years [1–5]. Such changes can be revealed via decoding the time series of contents of some trace elements in sediment cores. It has been recently discovered that the climate of Holocene and the last interglacial period was not changing smoothly. High-resolution records of isotopes  $\delta^{18}\text{O}$  [6,7] and

$\delta^{13}\text{C}$  [8] with sampling of 5–150 years in cores of Greenland ice and oceanic sediments manifested many rapid climatic shifts with the duration of ca. 1–5 ky. A spatial resolution up to 0.1 mm is needed to observe such a phenomenon in the sediments of Lake Baikal. So, it is necessary to replace the traditional methods of discrete analysis of sediments with the continuous scanning analysis of sediment cores. The XRF beamline of Siberian Synchrotron Radiation Center (SSRC) has been equipped with a special scanner to perform such a scanning procedure.

The goals of this paper are to describe the scanning XRF beamline parameters and a test experiment of the scanning analysis of sediment cores from Lake Baikal.

\*Corresponding author. Tel.: +7-383-239-4154; fax: +7-383-234-2163.

E-mail address: Zolotarev@inp.nsk.su (K.V. Zolotarev).

## 2. Methods and samples

The scanning analysis was performed at the XRF beamline of the VEPP-3 storage ring (Budker Institute of Nuclear Physics, Novosibirsk, Russia). The energy of an electron beam at VEPP-3 is 2 GeV; the magnetic field at the point of irradiation is 2 T.

The samples were prepared from sediments taken from a gravity core. They were  $200 \times 30 \times 5$  mm slabs of sediments cut out along the axis of the core to save the natural structure of layers; the slabs were dried in vacuum, embedded into epoxy resin, and polished on both sides. The reference samples were prepared in a similar way. In principle, such a difficult procedure for a sample preparation can be simplified significantly with the new method being developed now.

The beam line was equipped with a special scanner. The scanner permits the pulling of a sample with a length of up to 400 mm across a monochromatic SR beam and a reception of XRF spectra. The minimal spatial step of scanning is equal to 0.1 mm.

Special software completely controls the spectra acquisition during the scanning of a sample. Besides that, the software tests the presence of the SR beam from the storage ring and performs

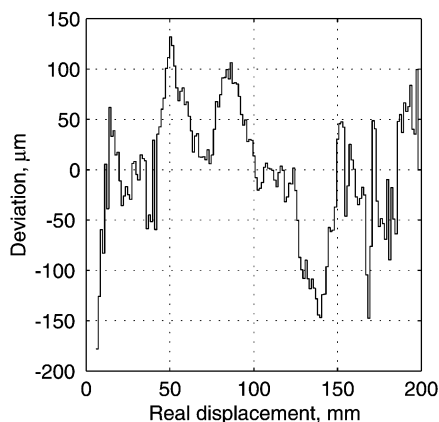


Fig. 1. The results of testing the scanner mechanical accuracy. The absolute displacement was measured by the displacement sensor (Burleigh Instruments, Inc) with a 0.1  $\mu\text{m}$  resolution.

repeats of the spectra acquisition if the electron beam changes in the storage ring.

The samples were irradiated with a monochromatized and polarized SR beam. Two energies of incident photons were used. The 22–26 keV band was used for the determination of elements from K up to Mo. The 45 keV band was used to determine the light lanthanides, Sb, Sn, I, and Ba. The concentrations of the following elements: K, Ca, Ti, V, Cr, Mn, Fe, Cu, Zn, Mo, Pb, Rb, Ba, Sr, La, Ce, Y, Nd, Sn, Sb, Br, I, As, Se, Nb and a few more were measured.

We studied the sediments of the core of Station 2GC ( $53^\circ 33'04''\text{N}$ ,  $107^\circ 54'53''\text{E}$ ) taken from a gravity core in 1998 on top of the underwater Akademichesky Ridge of Lake Baikal. The station is described in Ref. [9].

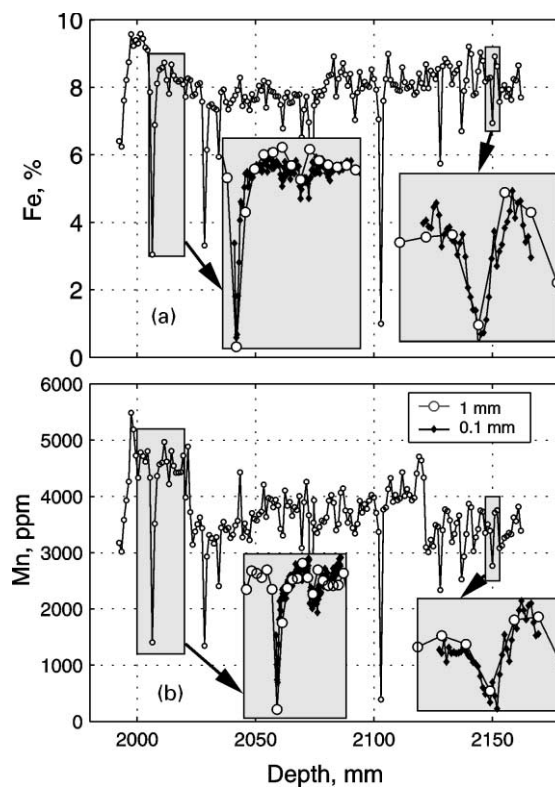


Fig. 2. The results of scanning reproducibility tests. The main profiles for Fe (a) and Mn (b) with a 1 mm step were checked by rescanning the outlet contain intervals (see insertions) with a 0.1 mm step.

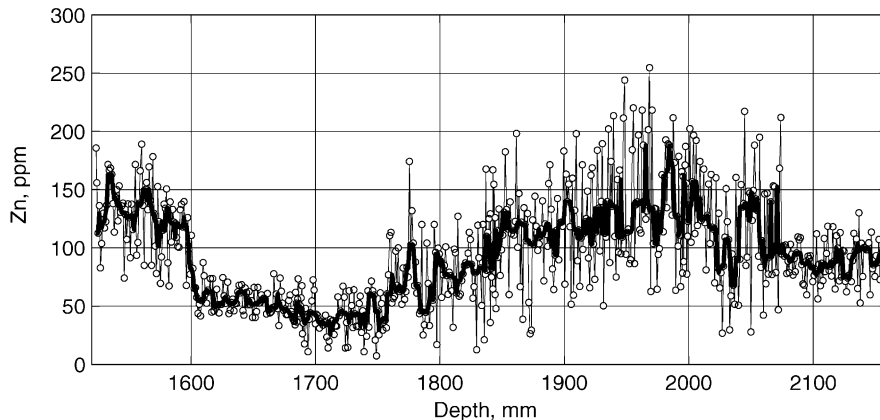


Fig. 3. An example of a raw experimental profile and median filtering of the experimental data (thick line).

### 3. Results and discussion

The mechanical accuracy of the scanner was checked with the special absolute displacement sensor (Burleigh Instruments, Inc.) with a  $0.1\ \mu\text{m}$  resolution. Fig. 1 shows how the displacement performed by the scanner deviates from the real one registered by the sensor. It is evident that the deviation does not exceed  $100\ \mu\text{m}$  over the  $200\ \text{mm}$  length of the scanning interval. Such a precision is quite enough for the paleoenvironmental investigations.

To check the analysis reproducibility a rescanning of the same sample was performed. The results of scanning the two short intervals of the sample are presented in Fig. 2. The common feature of these intervals is the existence of narrow singular outliers in the depth profiles. The rescanning of these regions of the sample confirms a natural (not statistical) origin of these outliers as well as shows good reproducibility of the experimental data. Fig. 3 shows the  $650\ \text{mm}$  long profile of Zn concentration during Karga interval (22–58 ky BP) [10]. In spite of the high noise caused by the non-homogeneity of the samples, the signal has clear trends, which can be subjected to a numeric processing (e.g. filtration). Similar profiles were obtained for all elements investigated.

Profiles of some elements display high variability of their contents, sometimes by an order of the magnitude. Nevertheless, there is a clear

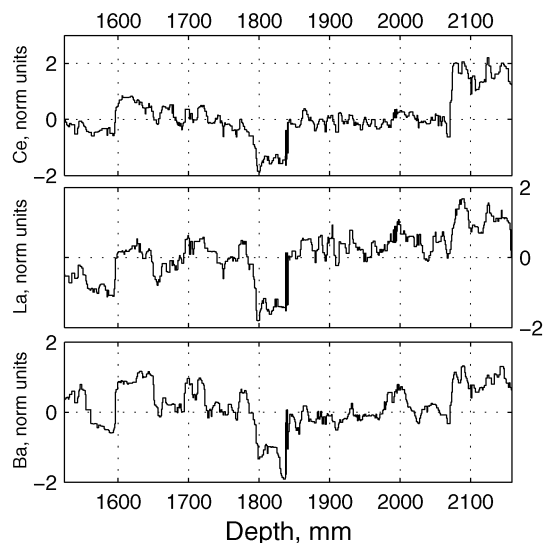


Fig. 4. An example of a good correlation between depth profiles of different elements.

correlation within the groups of some indicator elements: La–Ce–Ba–Nd, Cu–Zn–Sr, Y–Zr, etc. Concentrations of these elements also have strong correlations with other paleoclimate parameters [10], so these data can be used as paleoclimate indicators. The correlation between the “raw” contents of some elements is shown in Fig. 4. One can see from this figure that the element trends are not random, so a correct statistical processing of

these data can improve significantly the observed links.

The same scanning system can be applied also for scanning the wood samples (tree rings). The first scanning of such specimens will be performed in the nearest future.

### Acknowledgements

The authors are very grateful to Drs. V.A. Trounova, N.A. Mezentsev, M.A. Sheromov and Prof. M.A. Grachev for their useful support and for showing great interest in the work. The activity was supported in part by the Russian Foundation for Basic Research: contracts 99-02-17118 and 99-05-64743, CRDF RGI-2075 and by the Integration program of the Siberian Branch of the Russian Academy of Science.

### References

- [1] M.A. Phedorin, V.A. Bobrov, K.V. Zolotarev, Nucl. Instr. and Meth. A 405 (1998) 560.
- [2] E.L. Goldberg, M.A. Phedorin, M.A. Grachev, et al., Nucl. Instr. and Meth. A 448 (2000) 384.
- [3] M.A. Phedorin, E.L. Goldberg, M.A. Grachev, et al., Nucl. Instr. and Meth. A 448 (2000) 400.
- [4] E.L. Goldberg, M.A. Grachev, K.V. Zolotarev, et al., Nucl. Instr. and Meth. A 405 (1998) 584.
- [5] K. Horiuchi, E.L. Goldberg, K. Kobayashi, et al., Nucl. Instr. and Meth. A 470 (2001) 396, these proceedings.
- [6] W. Dansgaard, S.J. Johnsen, H.B. Clausen, et al., Nature 364 (1993) 142.
- [7] G. Bond, W. Showers, M. Cheseby, et al., Science 278 (1997) 1257.
- [8] J.P. Kennet, K.G. Cannariato, I.L. Hendy, et al., Science 288 (2000) 128.
- [9] M.A. Grachev, A.G. Gorschkov, I.A. Azarova, et al., Science, in press.
- [10] E.L. Goldberg, M.A. Grachev, M.A. Phedorin, et al., Nucl. Instr. and Meth. A 470 (2001) 388, these proceedings.