A NEW KRAKOW SCANNING NUCLEAR MICROPROBE: PERFORMANCE TESTS AND EARLY APPLICATION EXPERIENC

S. Lebed[1], J. Lekki[2], M. Paszkowski[3], W. Polak[2], A. Potempa[2], Z. Stachura[2], J. Styczen[2]
[1] Institute of Applied Physics (IAP), UR-44030 Sumy, Ukraine, salmp1@yahoo.com
[2] Institute of Nuclear Physics (INP), PL-31342 Krakow, Poland, Janusz.Lekki@ifj.edu.pl
[3] Institute of Geological Sciences, Polish Academy of Science, PL-31002, Krakow, Poland

A new scanning nuclear microprobe (MP) with a short-length probe forming system was designed, installed and tested at the 3MV Van de Graaff accelerator in Krakow. The MP resolution of 3.3μm was reached for a 2.4 MeV proton beam in the high-current mode (≥100pA). The MP facility provides a local, non-destructive, quantitative elemental microanalysis using a Proton Induced X-ray Emission (PIXE) technique. As example of possible applications an analysis of a geological sample containing monazite crystals investigated by PIXE method is presented.
PACS numbers: 29.27.Eg, 32.30.Rj, 91.65.Dt, 91.70.-c

1 INTRODUCTION

The new scanning nuclear microprobe (MP) with a short probe forming system (Fig.1) of 2.3m length was designed, constructed and installed in the Institute of Nuclear Physics (IFJ) in Krakow, Poland, during the period from 1995 to 1999 [1-4].

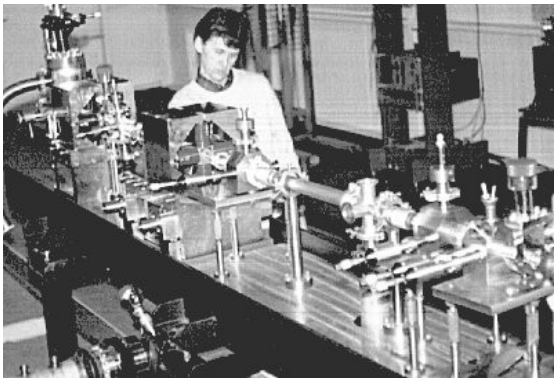

Fig. 1. Microbeam line of the Krakow scanning nuclear microprobe.

The accelerating and beam transporting systems of the Krakow MP were improved significantly last years. The MP parameters are maintained stable over many hours, with a relatively high ion microbeam current (up to 3nA at ≤10μm beam spot size) permitting measurements to be performed within a reasonable time. With such a current the microprobe proton induced X-ray emission (micro-PIXE) technique can be used in many applications of the geological sciences. The micro-PIXE is now accepted in many laboratories throughout the world as a highly sensitive method for elemental analysis. The method was developed at the Krakow MP [4,5].

The subject of this paper is to demonstrate the Krakow MP performance and micro-PIXE technique capabilities in geological research.

2. EXPERIMENT

2.1 Accelerator

The Krakow MP is based on a Van de Graaff (HVEC type K-3000) accelerator. Performance of the set-up has been improved by updating the accelerator during the year 2000: a new high-brightness RF ion source and a new belt charge power supply have been installed.

2.2 Beam transport system

The previous beam transport system of the Krakow MP was based on a quadrupole doublet of magnetic quadrupole lenses [3]. This was changed by installing an additional magnetic quadrupole doublet [4]. The doublet (Fig. 2) was manufactured in the IFJ mechanical workshop.

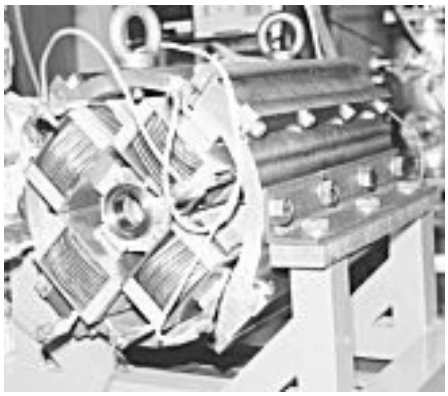

Fig. 2. New magnetic quadrupole doublet.

The new optimised beam transport system is based on a "divided Russian quadruplet" geometry providing a minimum contribution of the intrinsic and parasitic aberrations to the degradation of the beam brightness in the system. The system optics and quadrupole doublet design were developed by S.Lebed. These innovations and other above-mentioned VdG modernisation have laid the foundations of a significantly improved MP performance.

2.3 MP performance

The Krakow MP resolution is tested by measuring the beam profile and beam spot size (full width at half-maximum (FWHM)) at the target [4]. For this purpose a copper electron microscope grid is used. Secondary electron profiles from the grid are measured. The profiles from linear beam scanning over the grid are analysed. FWHM reaches 3.3μm (Fig. 3) in the high current (PIXE) mode and less than 10μm at 3nA proton beam

current (I_P). Detectors used are: a channeltron for secondary electron imaging and a Si(Li) detector for PIXE measurements.

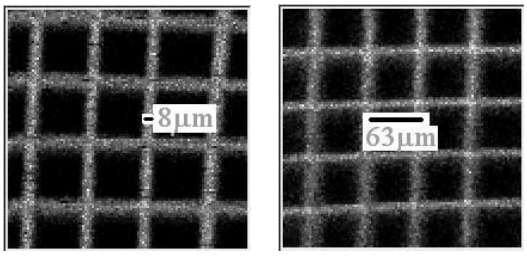

Fig. 3. Calibration copper grid (400mesh/inch) imaged by PIXE (2.4 MeV protons) in K_α and K_β X-ray lines. Left: spot size is 3.3μm at I_P =100pA. Right: spot size is 8μm at I_P =3nA.

2.4 Data acquisition system

A new data acquisition and evaluation system was installed [5]. The system collects detector events in the list (event–by–event) mode, storing the number of counts, energy pulse height and the current *x* and *y* coordinates of the beam. This data allows detailed off–line sample analysis. The MP scanning mode also makes possible a dynamic on–line two–dimensional (2D) analysis of the elements (trace, minor and major) in the sample by indicating the regions of interest (ROI).

3 RESULTS AND DISCUSSIONS

The geological micro–PIXE investigations were carried out on a sample of monazite crystals mounted in epoxy glue and coated with carbon (Figs. 4-7). The sample was prepared by processing the uppermost carboniferous sandstone from Kwaczawa village, Gródek gorge, Poland.

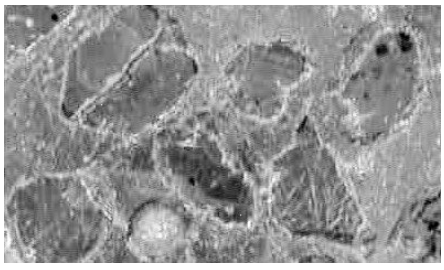

Fig. 4. Light microscope image (600x400 μm^2) of the sample.

In order to improve the minimum detection limit of heavy trace elements, a 200μm Al filter was placed in front of the Si(Li) detector to absorb most of the intense low energy X-rays emitted from the matrix materials of the sample. The relative intensities of the characteristic lines of the U, Th and Pb elements evaluated in the present experiment (Figs. 5, 6) can be used to calculate the chemical age of the geological sample, applying the Montel method [8].

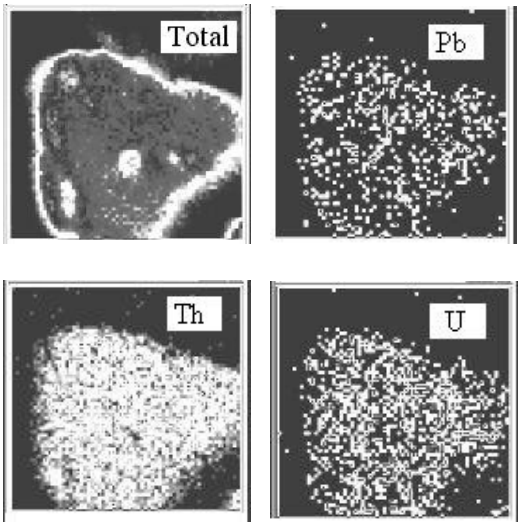

Fig. 5. Total and trace micro-PIXE elemental maps of a large monazite crystal. Measurement time is 15 minutes, scan area is 256x256 μm^2. Images show fairly homogeneous distribution of Pb, U and Th in the sample.

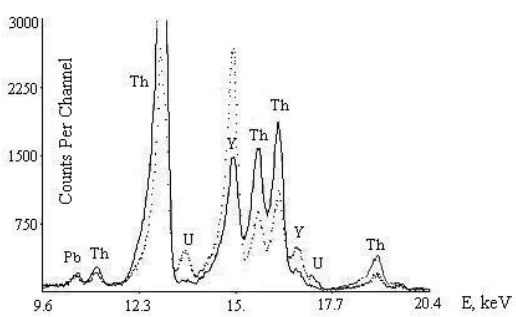

Fig. 6. Micro-PIXE spectra of the two monazite crystals. Measurement time is 70 minutes, scan area is 16x16μm^2.

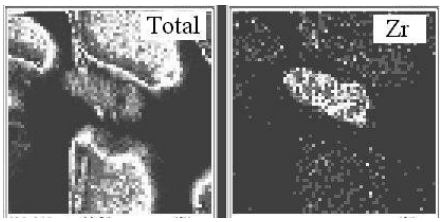

Fig. 7. Total and trace (with ROI set to Zr-K_α line) PIXE elemental maps of the monazite samples. Image resolution is 64×64 pixels, scan area is 256x256 μm^2. A small zirconium inclusion (~50x100 μm^2) is clearly seen in the central part of the image.

The micro-PIXE method delivers high detection sensitivity (1–10 ppm) for a broad range of geochemically important elements with Z>11. An effective depth of the analysis is 10–35μm. It allows the analysis of specific micro–inclusions (e.g. relics of mineralising fluids) in the minerals [9], containing important genetic information. In industrial geology, trace element distributions in minerals are used in prospecting for diamonds, platinum group minerals as well as in petrology, mining, ore genesis, etc. [7–9].

4. CONCLUSIONS

The spatial resolutions achieved by the Krakow MP are currently 3.3μm at beam current of 100pA and 8μm at beam current of 3nA for a 2.4MeV protons. The micro-PIXE technique is developed at the Krakow MP. The average U-Th-Pb trace elemental data obtained will be used to calculate the chemical age of a geological sample.

Most MP applications in geology are connected with the use of micro-PIXE in trace element geochemistry of rock forming minerals and their inclusions. The Krakow MP is well suited for these purposes.

The results obtained on the Krakow MP show the micro-PIXE technique to be highly sensitive, local, fairly fast, non-destructive, quantitative elemental microanalysis. The MP will be applied as an analytical tool in geology, biophysics, medicine, environmental and materials sciences, etc.

ACKNOWLEDGEMENTS

The work has been supported by the State Committee of Scientific Research (KBN) in Poland (contract No.1438/IA/620/95) and the Ministry of Education and Science in Ukraine (contract No. 2M/289-99).

REFERENCES

1. S.Lebed, Z.Cioch, A.Rys, Z.Stachura, L.Zrodlowski, M.Cholewa, D.Jamieson, G.Legge // *Ukrainskij Fizicheskij Zhurnal*. 1999, v .44, N 8, p. 937.
2. S.Lebed // *Nucl. Instrum. and Meth*. 1999, B 152, p. 145.
3. S.Lebed // *Nucl. Instrum. and Meth*. 1999, B 155, p. 322.
4. S.Lebed, Z.Stachura, M.Cholewa, G.J.Legge, J.Lekki, S.Maranda, A.Potempa, C.Sarnecki, Z.Szklarz, J.Styczen, B.Sulkio–Cleff // *Nucl. Instrum. and Meth*. 2001, B 181 (1-4), in print.
5. J.Lekki, R.Hajduk, S.Lebed, A.Potempa, T.Pieprzyca, Z.Stachura, M.Zieblinski, J.Styczen, Report No.1856/AP (2000), Inst. of Nuclear Physics Krakow, www.ifj.edu.pl/reports/2000.html.
6. S.Lebed, T.Butz, J.Vogt, T.Reinert, D.Speemann, J.Heitmann, Z.Stachura, J.Lekki, A.Potempa, J.Styczen, B.Sulkio–Cleff // *Nucl. Instr. and Meth.* 2001, B 181 (1-4), in print.
7. C.G.Ryan // *Nucl. Instrum. and Meth*. 1995, B 104, p. 377.
8. R.Frei, V.M.Prozesky, W.Przybylowicz // *Nucl. Instrum. and Meth*. 1997, B 130, p. 676.
9. S.H.Sie // *Nucl. Instrum. and Meth*. 1997, B 130, p. 592.