The implementation of a DSSSD in the upgraded boron analysis at LIBAF for applications in geochemistry

LIBAF — Lund Ion Beam Analysis Facility

P. Kristiansson[1]*, M.Borysiuk[1], U. Hålenius[2],
J. L. Mosenfelder[3], L.Ros[1], H. Skogby[2],
N. Abdel[1], M. Elfman[1], E.J.C. Nilsson[1] and J. Pallon[1]

[1]Division of Nuclear Physics, Department of Physics, Lund University, Lund, Sweden
[2]Swedish Museum of Natural History, Department of Geosciences, Stockholm, Sweden
[3]Caltech, Division of Geological and Planetary Sciences, Pasadena, CA, USA

Introduction

Boron and boron related analyses have been performed at LIBAF (Lund Ion Beam Analysis Facility) for almost 20 years. For the analysis the nuclear reaction p+[11]B is used with beam energy just below 700 keV where the reaction has a broad resonance. This reaction emits three alpha particles with energies much higher than the elastically scattered proton, which can easily be discriminated and counted as a function of beam charge.

In the original set-up an annular surface barrier was used to detect the alpha particles[1,2]. Then an analysis currents of 100-500 pA could be used together with the focused ion beam. This kept the count rate below 10 kHz, which was considered acceptable for this kind of analysis. In this paper we will describe and evaluate the upgrade of the system from a single detector to a DSSSD (double-sided silicon strip detector)[3].
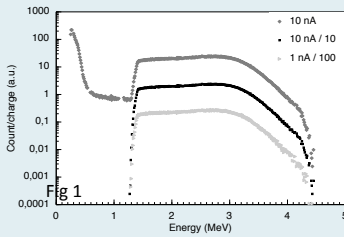
Experimental System

Experimental site: Lund Ion Beam Analysis Facility[4,5]

Beam energy: 700 keV and 610 keV protons

DAQ: Combined CAMAC (scan) and VME (energy)

Scan: Typical 128×128 with 10 μm step (smaller for high-resolution runs)

Detector: Double sided silicon strip detector with 64 sectors on the front side and 32 rings on the back side.

Trigger: Low threshold on rings (CR) and high threshold on sectors (B) in coincidence.

Charge normalization: Internal B-standard in sample. Charge and CR used for normalization.

Boron as a geochemical tracer

The interest in high-resolution boron analyses from a geochemical perspective is due to the recognition of boron as an important tracer of chemical recycling in the Earth, as a result of the high solubility of boron in aqueous fluids and silicate melts. Although boron is not a nominal component in common silicates (e.g. pyroxene, olivine, sillimanite) they may contain enhanced B-concentrations (≤1 wt% B_2O_3) and hence constitute important boron reservoirs in the deeper parts of the Earth. The nature of B-substitution mechanisms in these minerals is not fully understood and partly contradictory models have been suggested. Isotopic fractionation of boron is strongly dependent on how boron is coordinated by its nearest anions[7].

The Samples

In the experiments two sets of samples have been analyzed, one set mounted on an In-backing and one set on a epoxy backing. Each set had between 15 and 20 crystals and also one tourmaline crystal was included on each sample for internal normalization.

Results

Apart from analyzing two sets of crystals regarding boron concentration and distribution the performed experiment was intended as an evaluation of the upgraded system with the new DSSSD detector.
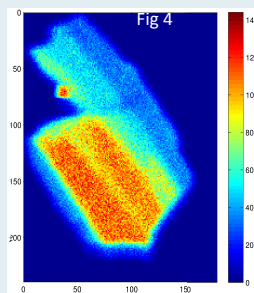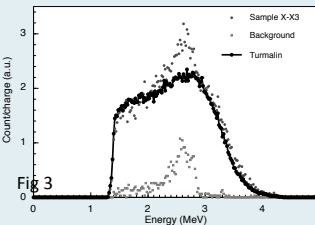
In figure 1 energy spectra from analyzes of an tourmaline standard are shown. The upper curve is the raw spectrum taken with a current of 10 nA, the second curve (scaled a factor of 10) is the same set of data but gated on the multiplicity condition 1. The third curve is a spectrum taken with 1 nA and shows the same shape as the high current spectrum.

Kmax Run no	VME Run no	Sample	comment	I (nA)	B-counts	no of pixel B	Q [B-area]	B/Q	live time [%]	B/Q - bkg	err	Norm (ppm)	stat err
16	544	Tourm.	Reference		533106	3774	45686	11,669	96	11,6506	0,0160	101800	140
17	545	20425X2		1,9	38132	4407	165979	0,230	97	0,2114	0,0012	1832	11
18	546	20425X1	mean	3,5	27660	8012	182717	0,151	96	0,1331	0,0010	1166	8
		20425X1	high B part		7384	1494	34140	0,216	96	0,1980	0,0026	1734	23
19	547	20441X2		2,3	29181	2312	240602	0,121	90	0,1030	0,0008	958	7
20	548	20441X2		3,6	38374	5740	328941	0,117	95	0,0984	0,0006	867	6
21	549	36034X1	no Boron		4303	14882	223091	0,019	97	0,0010	0,0004	9	4
22		36034X2	no Boron	3,7	3446	3922	190212	0,018	97	-0,0002	0,0004	-2	4
23		27122X1		1,2	13296	2355	65496	0,203	97	0,1847	0,0018	1602	16
24	552	27122X2		1,8	10418	3639	82866	0,126	97	0,1074	0,0013	928	11
25	553	90123X2	no Boron	2,3	4049	5952	237318	0,017	95	-0,0012	0,0004	-11	3
26		74591X1	no Boron	3,6	12643	3718	724674	0,017	95	-0,0008	0,0002	-7	2
27	555	74591X2	no Boron	3,0	4695	4864	257522	0,018	96	-0,0001	0,0004	-1	3
28	556	99011X2		2,8	22657	9479	444452	0,051	96	0,0327	0,0004	286	3
29	557	99011X2		2,7	7380	8954	160725	0,046	97	0,0276	0,0006	240	5
30	558	X-X3		2,9	190750	6060	813929	0,234	97	0,2161	0,0006	1881	5
31	559	X-X2		3,1	81370	5827	339848	0,239	96	0,2211	0,0009	1929	8
32	560	X-X1		2,9	35767	4549	150026	0,238	94	0,2201	0,0013	1961	12

Fig 1

In figure 2 the energy spectra from a tourmaline are shown for two different beam energies (above and below a [18]O resonance). The difference in yield is about a factor of two.
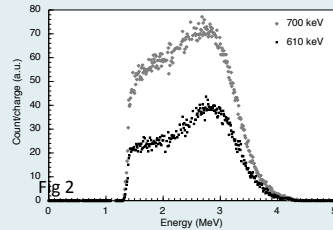
In figure 3 a background spectrum and one spectrum from a low boron concentration crystal are shown (X-X3 from the table). The solid line is a the tourmaline yield normalized to the boron in X-X3. The overlay of background on the distribution is clear. Here the background corresponds to roughly 150 ppm. The background will be lower below the [18]O resonance energy, but so will the yield.

Fig 2

Fig 3

Fig 4

An illustration of what can be achieved with a high resolution run with good statistics. The zonation pattern in the crystal is nicely shown in figure 4 .

Quantitative analysis is the primary goal of the presented technique. In the table the different steps going from counts to ppm are illustrated for the epoxy sample. First a suitable area from a scan is selected and the number of B-counts is extracted from this area. The corresponding charge is extracted and the data is normalized. The background is subtracted and the concentration is normalized to the tourmaline standard.
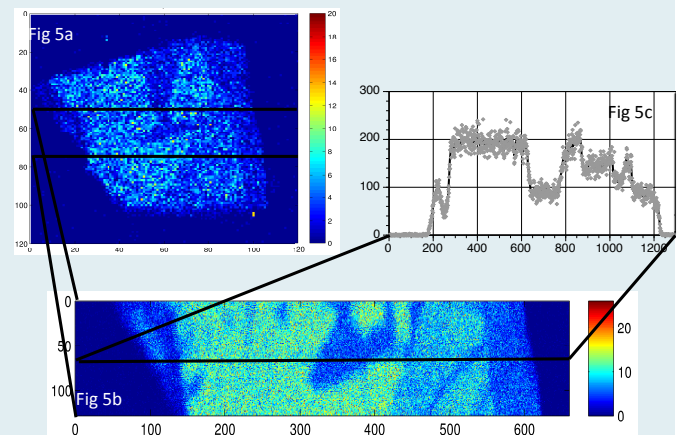

Fig 5a

Fig 5b

Fig 5c

Figure 5 demonstrates another application of the scanning method. A fast scan gives the gross features of the crystal (a). An area is selected and the step is decreased and a high resolution map over the interesting part of the crystal is collected (b). Finally a line scan is performed with optimal resolution (c).

References

[1] Per Kristiansson, Ulf Hålenius, Henrik Skogby, Mikael Elfman, Klas Malmqvist, Jan Pallon, Boron distributions in single crystals investigated with nuclear reaction micro analysis, Nucl. Instr. Meth. B 158(1999)562

[2] Henrik Skogby, Per Kristiansson and Ulf Hålenius, An assessment of nuclear microprobe analyses of boron in silicate minerals, American Mineralogist 2003 88, 1601-1604

[3] P. Golubev, P. Kristiansson, N. Arteaga-Marrero, M. Elfman, K. Malmqvist, E.J.C. Nilsson, C. Nilsson, J. Pallon and M. Wegdén. First results from the Lund NMP particle detector system. Nuclear Instruments and Methods in Physics Research Section B267 12-13 (2009)2065-2068

[4] A Shariff, C. Nilsson, V. Auzelyte, M. Elfman, P. Kristiansson *, K. Malmqvist, J. Pallon, M. Wegdén, The Lund nuclear microprobe sub-micron set-up. Part II: Beam line, focusing system and scanning, Nucl. Instr. Meth. B231 (2005) 7-13

[5] M. Elfman, J. Pallon, V. Auzelyte, P. Kristiansson, K. Malmqvist, C. Nilsson, A. Shariff, M. Wegdén, The Lund nuclear microprobe sub-micron set-up. Part III: Sample stage, optical imaging and detector configuration in the experimental chamber, Nucl. Instr. Meth. B231 (2005) 14-20

[6] Chaussidon M. and Libourel G. (1993) Boron partitioning in ther upper mantle: an experimental and ion probe study. Geochim Cosmochim Acta 57, 5053-5062.

[7] Hervig R., Moore G.M., Williams L.B., Peacock S.M., Holloway, J.R. and Roggensack K. (2002) Isotopic and elemental partitioning of boron between hydrous fluid and silicate melt. American Mineralogist 87, 769-774.

Conclusion

➢ New detector set-up tested for boron analysis - improved the system significantly.
➢ Can be operated with very high count-rate – up to 200 kHz or higher.
➢ Good detection limits – depends on sample composition and beam energy.
➢ High resolution mapping possible with good result.

Questions? Per Kristiansson, Div. of Nuclear Physics, Lund University, Lund; Sweden. per.kristiansson@nuclear.lu.se
21st International Conference on Ion Beam Analysis
IBA2013, June 23-28, 2013 – Seattle, WA USA