A new electron microprobe facility for teaching and research in West Virginia

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CONCORD UNIVERSITY

1 Abstract

In 2010, Concord University, a 2800-student, predominantly undergraduate institution in southern West Virginia, installed an ARL SEMQ microprobe formerly located at the University of Kentucky as the first stage of a plan to establish a regional user facility that is open to outside commercial and academic users, with emphasis on undergraduate teaching and research. The West Virginia Research Trust Fund provided major support for initial operation of this, the only electron microprobe in West Virginia. The instrument is equipped with four wavelength-dispersive (WDS) spectrometers containing LIF/PET, PET/RAP, ADP/LIF, and TAP/OV60 analyzing crystals which enable quantitative analysis of elements from carbon through uranium. Quantitative analysis functions (beam, spectrometers, and stage) are automated using Probe for EPMA running on modern PC hardware. With support from a West Virginia EPSCoR Innovation Grant, we are currently upgrading the existing Si(Li) energy-dispersive (EDS) detector to a state-of-the-art LN-free SDD detector. This major upgrade provides significant enhancements to our analytical, X-ray mapping, and automation capabilities, including the ability to perform combined WDS+EDS analyses. Ancillary equipment includes a carbon coater and a separate sample preparation and polishing laboratory. The microprobe is further complemented by a Horiba XGT-5000 micro-X-ray fluorescence (XRF) analytical microscope for qualitative elemental mapping of areas as large as 100 cm^2 with a resolution of 0.01-0.1 mm.

We are incorporating this instrument into the curriculum at all levels from introductory general education to advanced major courses in multiple disciplines, including the earth sciences, chemistry, and physics/material science. Among the first courses to use the instrument are petrology and introductory geology in Spring 2011. The microprobe will also be used in student independent study and thesis research as well as faculty research. To serve off-site users, we are developing the capability for remote operation of most functions needed for quantitative analysis and X-ray mapping. We anticipate use of the microprobe by other universities for both undergraduate- and graduate-level teaching and research.

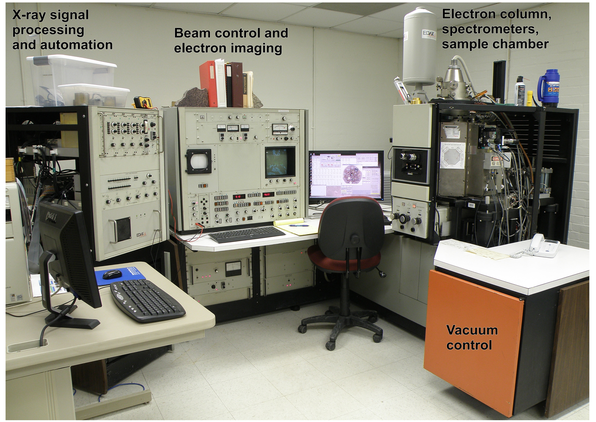
2 ARL SEMQ Design

The SEMQ design has a number of advantages. The higher take-off angle (52.5° vs. 40°) increases light-element sensitivity through reduced absorption of X-rays exiting the sample, reduces matrix corrections for quantitative analysis, and reduces the sensitivity to minor surface topography. The instrument and spectrometers are ruggedly built and therefore durable.

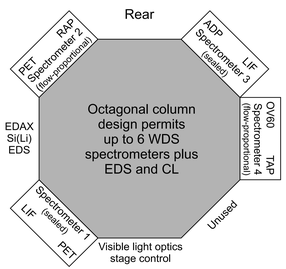
Up to 6 fully-tunable wavelength dispersive (WDS) spectrometers can be installed when the energy-dispersive spectrometer (EDS) is placed in a rear port. Some SEMQ instruments were configured with as many as 9 WDS spectrometers (6 fixed, 3 tunable). In this arrangement, 2 fixed-position spectrometers fit in the same space as 1 tunable spectrometer.

3 Instrument Configuration

http://academics.concord.edu/microanalysis/



The instrument is configured with 4 WDS spectrometers, 1 Si(Li) EDAX EDS spectrometer, and both secondary electron (SEI) and backscatter electron (BSE) detectors. Currently, separate computer systems are used for (1) quantitative WDS analysis and (2) EDS analysis, X-ray mapping, and electron image capture. When the new SDD EDS system is installed, these functions will be integrated into a single computer system. Besides the much higher EDS count rates afforded by the SDD detector, this upgrade will also provide the capability for improved X-ray mapping, improved electron imaging, and combined WDS+EDS quantitative analysis. This will also allow for greater analytical throughput and/or increased precision as well as improve the remote operation capability.

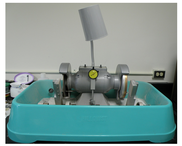


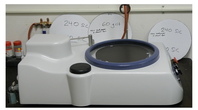
Potential upgrades include additional WDS spectrometers, a large-crystal spectrometer, solid-state WDS detectors, cathodoluminescence (CL) imaging, video camera on the visible light optics, dual CRTs for simultaneous display of SEI and BSE images, a higher precision stage, and modern fully-automated SEM electronics.

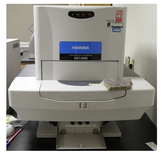
4 Supporting and Complementary Equipment and Materials

An extensive collection of calibration standards and reference materials is under development, including: minerals, metals, alloys, glasses, and synthetic compounds.

Equipment not shown includes saws for large and small samples and petrographic microscopes


Hillquist thin section machine


Buehler grinder/polisher with magnetic quick-change system


Horiba XGT-5000 micro-XRF


Denton Vacuum DV-502 high-vacuum coater

Acknowledgements

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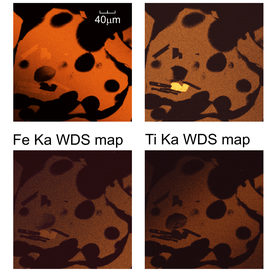
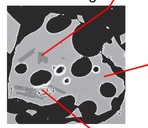
National Science Foundation
West Virginia Research Trust Fund
West Virginia EPSCoR
Concord University

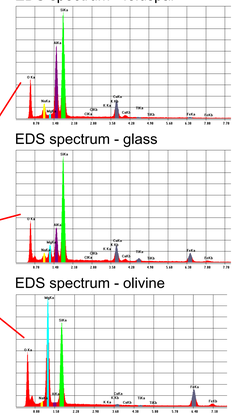
5 Sample Results



15 kV, 20 nA beam, 2 hours total time
field of view approximately 300x300 μm

Laki 1783 AD volcanic ash





WDS Quantitative Analyses

Sample		SiO_2	TiO_2	Al_2O_3	FeO_T	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Cl	Total	n
Laki 1783 (glass)	**Mean**	**49.55**	**3.12**	**12.93**	**14.05**	**0.22**	**5.39**	**9.87**	**2.76**	**0.45**	**0.35**	**0.017**	**98.70**	**44**
	StDev	0.36	0.04	0.17	0.18	0.02	0.08	0.11	0.10	0.04	0.08	0.011	0.48	
INTAV Reference value:		49.69	3.08	12.97	14.14	0.23	5.40	9.69	2.87	0.46	0.34	0.019	98.90	
BHVO-2G (glass)	**Mean**	**50.02**	**2.78**	**13.65**	**10.83**	**0.16**	**7.26**	**11.49**	**2.18**	**0.51**	**0.26**	**0.010**	**99.15**	**23**
	StDev	0.34	0.05	0.23	0.10	0.02	0.06	0.14	0.08	0.02	0.06	0.012	0.47	
USGS Reference value:		49.80	2.75	13.50	10.80	0.17	7.23	11.40	2.18	0.51	0.27		99.14	
Edziza tephra (glass)	**Mean**	**61.28**	**0.23**	**17.60**	**4.67**	**0.13**	**0.11**	**1.05**	**8.20**	**5.31**	**0.046**	**0.21**	**98.81**	**27**
	StDev	0.73	0.03	0.20	0.09	0.02	0.03	0.04	0.19	0.09	0.045	0.03	0.75	
INTAV Reference value:		61.55	0.24	17.55	4.55	0.13	0.12	1.09	8.21	5.30	0.04	0.21	98.99	

Analyzed 26-29 January, 2011 Analytical conditions: 15 kV, 15 nA current, 10 μm defocused beam, 4.5 minute total analysis time
Calibration standards are: Albite (Na, Si, Al), Sanidine (K), Kaersutite (Ti, Mg, Ca, Fe), Tugtupite (Cl), Spessartine (Mn), Apatite (P)
Time-dependent-intensity (TDI) correction turned on for Na, Al, Si on Edziza tephra glass; TDI turned off for basaltic glasses and calibration standards
INTAV Reference values from: Kuehn, S.C., Froese, D.G., and Shane, P.A., in press, The INTAV intercomparison of electron-beam microanalysis of glass by tephrochronology laboratories, results and recommendations, *Quaternary International*.
Edziza reference based on EPMA (27 labs), XRF, and ICP-AES. Laki reference based on EPMA (27 labs).