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Using Peakaboo* XRF Spectral Analysis and Imaging Software with VESPERS Microbeams: Mineralogical and Metallurgical Applications

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Introduction

Synchrotron X-ray Fluorescence (SXRF) spectroscopy provides unparalleled sensitivity to trace elements in solids. SXRF is now primarily used in the generation of “maps” showing the spatial distributions of trace elements in materials, thus revealing important local differences in chemical or structural properties. To be fully effective in this, the technique requires an exciting photon beam of micrometres down to sub- micrometre dimensions, stable sample scanning stages, fast detectors and efficient software. The VESPERS beamline is making good progress in all these directions and provides a beam with a spot size down to 1.5 x 1.5 μm (measured in November 2009). All spectrum data shown in this report were collected using the VESPERS beamline 07B2-1 at the CLS.

A key development has been the “Peakaboo” SXRF freeware that has been developed as a part of the Science Studio project [1]. It is designed to analyze the detailed structure of XRF spectra, including escape peaks, and yields total integrated intensities of K, L and M series lines of all detectable elements. The software provides a number of mathematical filters that are used in noise reduction, background attenuation, or removal. Noise filters include moving average, low pass FFT, Savitsky-Golay, and wavelet transform. The reach and order of most of these filters can be altered to suit a particular spectrum.

To create a map, a composite spectrum is fitted and the resultant elemental intensities are plotted in a map whose dimensions are provided by the scan coordinates saved during the acquisition. In Figure 1, the main page of Peakaboo is shown during the analysis of a composite XRF spectrum in preparation for the creation of a map of elemental intensities. The software can be downloaded from www.sciencestudioproject.com.

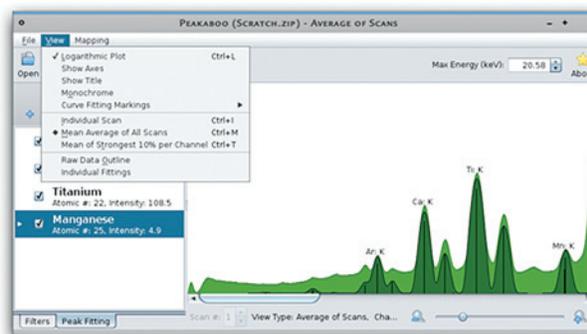


Figure 1: Screenshot of Peakaboo showing the analysis of a composite spectrum. The View tab is open to show the choices of spectral display available to the user.

Science

In this Report, we show two examples of SXRF mapping from VESPERS obtained in the past six months. The spectra were acquired using 10% bandpass radiation centred at 19 keV. The first is a sample taken from a uranium mine tailing deposit in the Athabasca Basin of Northern Saskatchewan.

The distributions of over 25 elements were able to be mapped in the specimen, but only eight are shown here. Four are of particular interest. All eight maps are shown in Figure 2(a-h). Arsenic is widely distributed and the map shows its variation from highest to lowest concentration. The apparent dominance of iron and absence of arsenic in some regions is somewhat exaggerated due to the intensity scaling properties used. However, some iron-containing phases (ferrihydrite; $\text{Fe}(\text{OH})_3$) are indeed localized with reduced arsenic concentrations, in addition to localized regions containing chromium and potassium. Nickel-arsenic phases are identified in (2b) and are likely primary minerals such as gersdorffite (NiAsS). Similar localized elemental associations of Fe, As and Ni have been demonstrated by electron microscope X-ray maps [2]. Uranium remnants are found at the periphery of the iron-containing phase while thorium was located in another area entirely. The identification of localized uranium and thorium occurrences is made more certain by the use of multiple L lines, all of which must be fitted to the spectrum.

The second example is a cross-section of an ataxite (iron-nickel) meteorite from Bali, Indonesia (Figure 3(a-d)). All SXRF data was obtained using white beam radiation. Troilite (iron sulphide) globular nodules are evident in the taenite (nickel-iron) matrix. Cr is associated with the troilite globules. A Ni enriched area, likely awaruite, is apparent in the upper right quadrant. A number of other elements can be mapped: Al, Ga, Zr, Mn, Ti, and Ca.

Combined with a photon beam size in the micrometre range, X-ray maps with that resolution and a 200 micrometre field, could be acquired in 2-3 hours. Typical detection limits in a map for mid-Z elements such as copper and arsenic will be in the order of 30 ppm when white beam radiation is used for excitation.

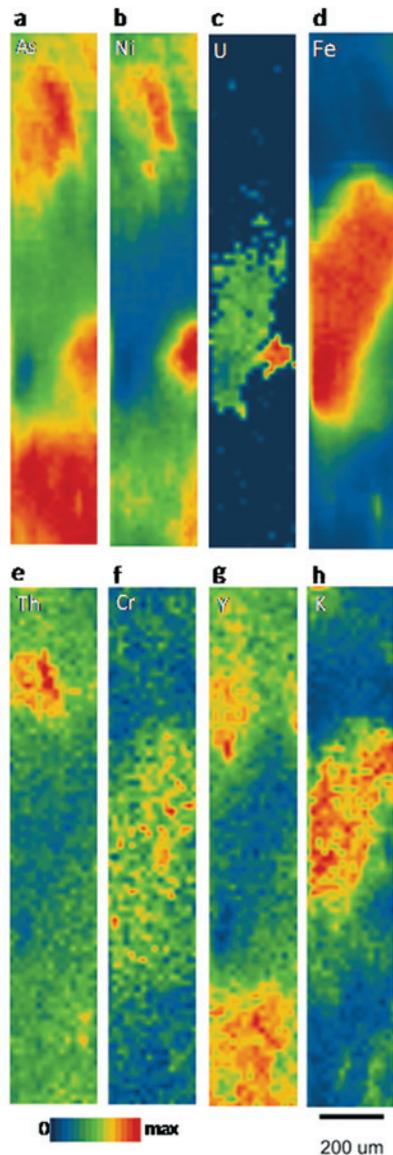


Figure 2: SXRF maps of (a) As K, (b) Ni K, (c) U L, (d) Fe K, (e) Th L, (f) Cr K, (g) Y K and (h) K K in an Athabaska Basin mine tailing. The intensity range shown does not infer the absence of an element in the lowest intensity range.

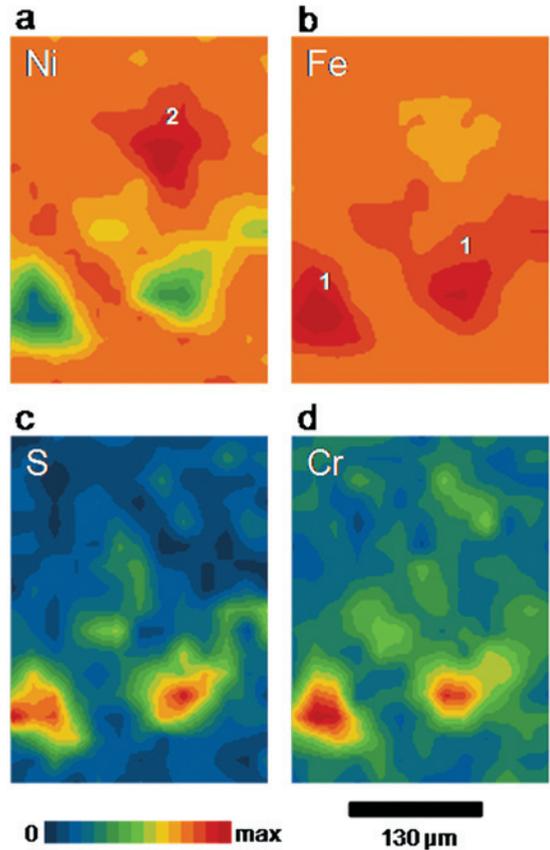


Figure 3: SXRF maps of (a) Ni K, (b) Fe K, (c) S K and Cr K lines showing troilite (iron sulphide) globular nodules (1) in the meteorite cross-section. A Ni enriched area (2), likely awaruite, is apparent in the upper right quadrant.

References

1. Science Studio is a web services platform designed for remote operation of scientific experiments and processing of large data sets.
2. Essilfie-Dughan, J, Hendry, J, Pickering, I, George, G, Warner, J, Kotzer, T, 2009. Mineralogical characterization of arsenic, selenium, and molybdenum in uranium mine tailings. 24th International Applied Geochemistry Symposium, June 1-4, 2009, Fredericton, New Brunswick, 4 pages.