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Advanced EDS and µXRF Analysis of Earth and Planetary Materials using Spectrum Imaging, Computer-Controlled SEM and an Annular SDD

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Introduction: An analytical approach using both energy-dispersive X-ray spectrometry (EDS) on scanning electron microscopes (SEM) and benchtop micro X-ray fluorescence spectrometry (µXRF) enhances data generation for Earth and Planetary science samples.

Methods: Improvements for EDS and µXRF are a result of silicon drift detector (SDD) technology which reduces measurement time. Furthermore, data processing has advanced by spectrum imaging techniques. µXRF allows the distribution of elements with $Z>10$ and trace elements (down to 20 µg/g) with a spatial resolution >25 µm to be displayed. Samples with sizes up to 20x16 cm can be rapidly analyzed with the M4 Tornado µXRF system to locate regions of interest for further high resolution SEM studies. Using SEM-EDS, minerals in large areas can be classified by feature analysis with stage control, a combination of morphological classification with chemical analysis. Analyzing only features of interests by selecting grey scale thresholds in the BSE micrograph significantly reduces measurement and evaluation time. All grains <4 µm in diameter can be classified in a whole thin section within <2 hours using a BSE pixel resolution of ~1 µm (Table 1). The spatial resolution for element analysis can be enhanced to the sub-µm scale by using low electron beam energy (HV<7). Extended atomic databases included in the EDS software improve the identification and quantification of low energy X-ray lines. A new type of EDS detector, the annular four channel SDD (XFlash QUAD 5060F) is placed between the SEM pole piece and the sample. It covers a very large solid angle (1.1 sr) and allows sufficient data collection at low beam currents on beam sensitive samples with substantial surface topography. Even at the lowest beam current (<10 pA), a sample can be investigated without carbon coating under high vacuum. Compared to low vacuum analysis, this approach reduces hydrocarbon contamination and avoids beam skirting effects.

Applications: (1) Sudbury Igneous Complex (SIC): The existence of high demand elements in samples from the offset dike can be determined by µXRF (Figs. 1a,b). The analysis of features at the sub^m scale by SEM-EDS (Fig. 1c) provides new insights for sulfide, telluride and arsenide deposit models. (2) Mocs historic meteorite: µXRF studies reveal lead enrichment along cracks (Fig. 2a). SEM-EDS indicates contamination of lead as a result of old polishing and soot by heating with coal-fired furnaces (Figs. 2b,c). (3) Tissint Martian meteorite: SEM-EDS studies display a thin coating and local enrichment of carbon and nitrogen (Fig. 3). It may be attributed to processes during the ejection from Mars. Further examples obtained by the XFlash QUAD 5060F on micrometeorites at the radiator panel of Hubble space telescope, Stardust analogue crater experiments and fossil arthropods will be discussed.

Conclusions: Improvements in SDD technology, advanced data processing and the possibility to analyze samples in a close to natural state with little preparation will stimulate new approaches for Earth and Planetary sciences as well for biological and cultural heritage sciences.

Table 1. Classified minerals using a bright BSE threshold. The area shown in Fig. 1a was analyzed by automated feature analysis in 875 fields. (XFlash 6|10, 25 kV, 80-160 kcps, and 0.5 s per spectrum).

Class	CoNi-arsenide	Pt-arsenide	Pd-Bi-Telluride	Bi-Telluride	Monazite	Th-phase	Zircon	Baryte	All
Count	9	6	8	2	19	8	50	1	103
Area (%)	0.000117	0.000098	0.000294	0.000011	0.000082	0.000034	0.000389	0.000004	0.001029

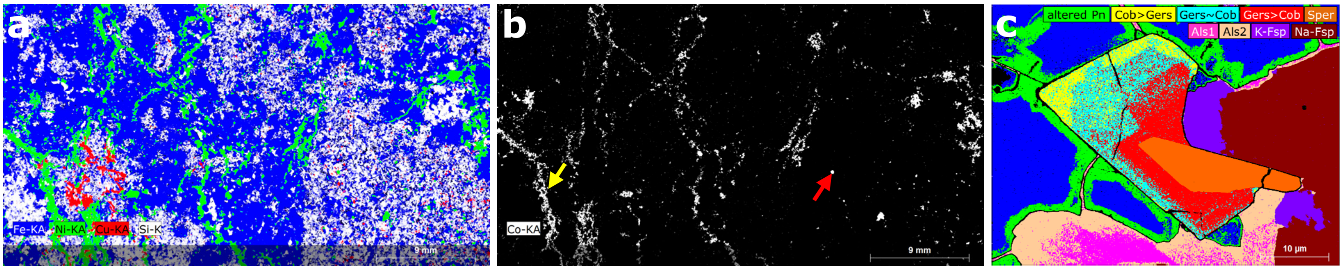

Figure 1. Sample from the Sudbury Igneous Complex; (a) Composite µXRF map of a thick section (44 mm x 24 mm, 2x 30 mm^2 SDD, 50 kV, 600 µA, 310 kcps, 2h, 20 mbar chamber pressure, 2210x1185 pixels, 20 µm pixel size, and 2 ms dwell time). (b) µXRF map of Co-K. Note the association of Co and Ni at pentlandite (yellow arrow) and arsenides (red arrow). (c) Chemical phase map showing the distribution of pentlandite (Pn), cobaltite (Cob), gersdorffite (Gers), sperrylite (Sper), alumosilicates (Als) and feldspars (Fsp) under consideration of Fe-L, Co-L, Ni-L, As-L, S-K, Al-K, Si-K, Pt-L, K-K, Na-K and Mg-K. (XFlash 6|10, 7kV, 22 nA, ~97 kcps, 187 min, 1024x768 pixels, and 45 nm pixel size).

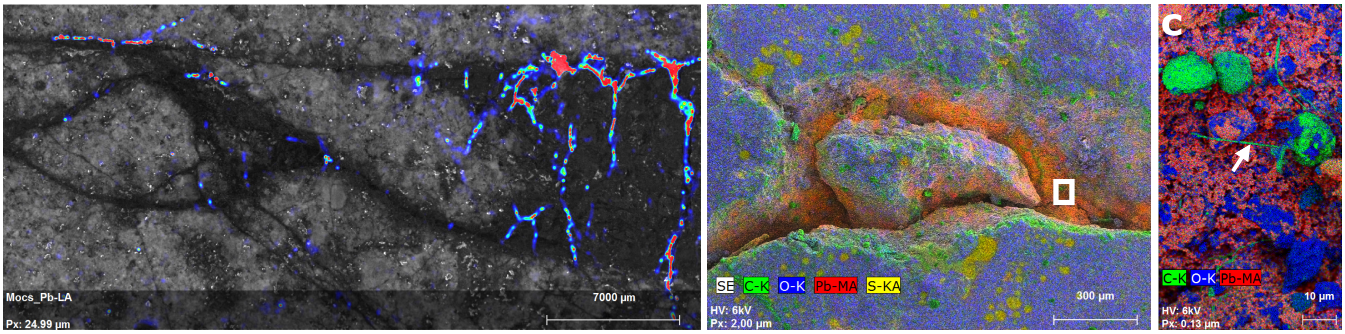

Figure 2. Mocs meteorite; (a) Video micrograph overlain with µXRF map of Pb-L (1472x676 pixels, 25 µm pixel size, and 33 min). (b) Composite EDS element map overlain with SE micrograph. Overlapping peak intensities (Pb-M, S-K) were deconvolved with an automatic routine. (XFlash QUAD 5060F, 6 kV, <10 pA, 2 kcps, 800x600 pixel, 2 µm pixel size, and 17 min). (c) Composite EDS map of the area shown in (b). Arrows show carbon features <300 nm in size (130 nm pixel size, and 5h 10 min).

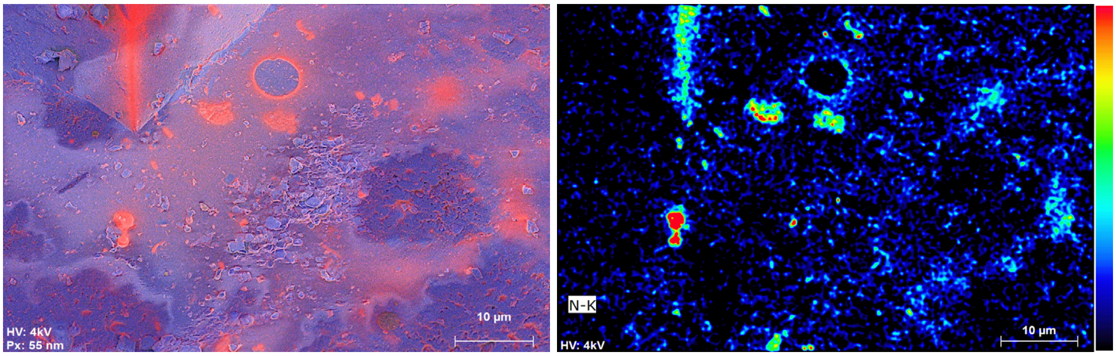

Figure 3. Tissint Martian meteorite; (a) Composite EDS map of carbon (red) and oxygen (blue) overlain with SE micrograph. (b) Nitrogen distribution in false color display. (XFlash QUAD 5060F, 4 kV, 1.8 kcps, 1280x853 pixel, and 17 h 32 min).