

## Practical Aspects of X-Ray Mapping in Electron Probe Microanalysis of Minerals

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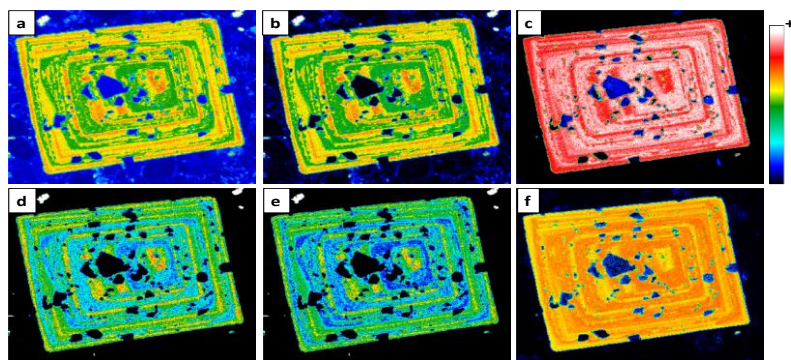
X-ray element distribution maps acquired by wavelength-dispersive spectrometry (WDS) on electron probe microanalyzers (EPMA) are widely used to visualize small-scale spatial variations of the chemical composition in rocks and minerals. Often only qualitative x-ray intensity maps are presented where real compositional features are superimposed with instrumental artefacts such as x-ray continuum variations and spectral interferences. To provide reliable, quantitative information, WDS maps should be processed in the same way as point measurements in terms of background, interference and matrix corrections [1].

Natural perovskite ( $\text{CaTiO}_3$ ) crystals from the Mark kimberlite (Lac De Gras, Canada) were mapped on a JEOL JXA-8530F Plus instrument at 15 kV accelerating voltage, beam current of 50 nA (Ca, Ti, Mg, Si, Fe, all  $K\alpha$ ) or 300 nA ( $\text{Na}K\alpha$ ,  $\text{Nb}L\alpha$ ,  $\text{Ce}L\alpha$ ,  $\text{Nd}L\alpha$ ,  $\text{Th}M\alpha$ ), beam diameter of 0.2  $\mu\text{m}$ , step size of 0.5  $\mu\text{m}$  and dwell time of 200 ms. Oxygen was calculated by stoichiometry and the mean atomic number (MAN) background correction was employed [2]. They show complex oscillatory zoning with bands bright in BSE enriched in elements like Na and Th, or Nb, Ce, and Nd [3]. Fig. 1 shows the effect of background correction, quantification, and Ti interference correction on the Ce map.

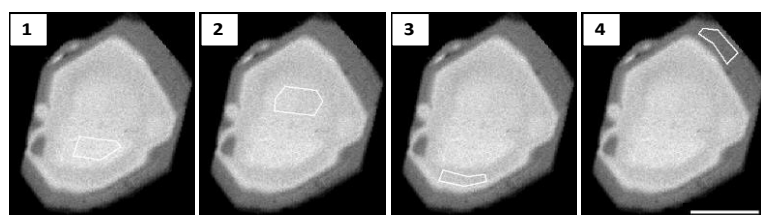
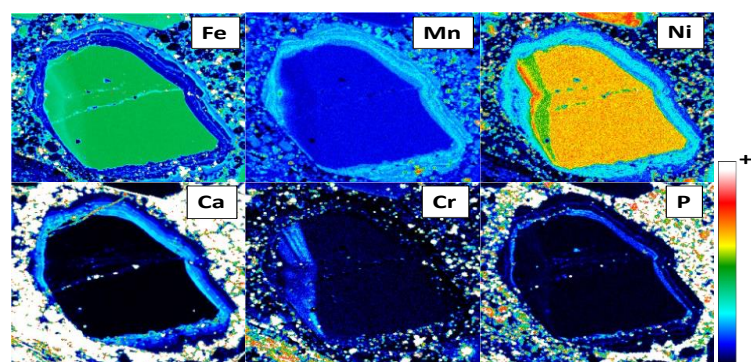
The same approach was used for the mineral olivine,  $(\text{Mg,Fe})_2\text{SiO}_4$ . Its high resistance to damage by the electron beam makes it suitable not only for high precision trace element point analysis [4], but also trace element mapping [5]. Fig. 2 shows selected element maps for an olivine grain from the Mark kimberlite, mapped at 15 kV, 70 nA for Mg, Si, Fe and 300 nA for Na, Al, P, K, Ca, Cr, Mn, Ni (all  $K\alpha$ ). The precision of the element data can be dramatically improved by combining multiple pixels in areas with uniform composition and extracting area compositions, see example in Fig. 3 for an olivine grain from the Udachnaya-East kimberlite, Russia [6]. A comparison of the extracted compositions with point analyses in the same regions in Fig. 4 shows generally good agreement. The Cr data points plotting significantly high above the 1:1 line could potentially be due to buried Cr-spinel inclusions which can easier be avoided in the area extraction.

### Figure 1. Perovskite map.

a)  $\text{Ce}L\alpha$  raw intensity, b)  $\text{Ce}L\alpha$  net intensity, c) Ti quantified, display range 4-31 wt%, d) Ce quantified, range 2.5-6.0 wt%, e) as before, with Ti on Ce interference correction, f) amount of Ti-Ce interference correction, range 0-0.25 wt%. Horizontal field of view = 170  $\mu\text{m}$ .

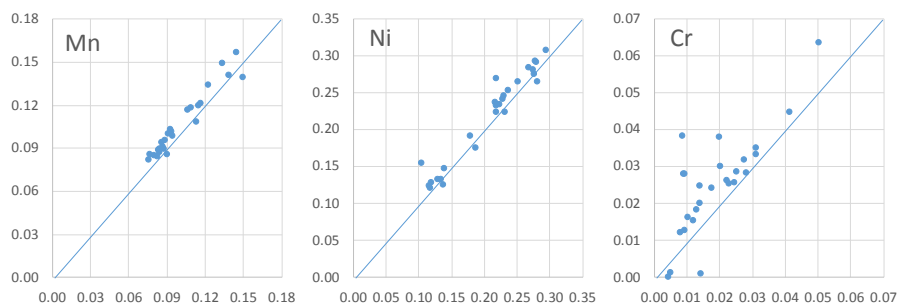


**Figure 2.** Olivine map for selected elements, display ranges: Fe 1-15 wt%, Mn 0-1 wt%, Ca 0-6 wt%, Ni, Cr, P 0-0.4 wt%. Horizontal field of view  $\approx 620 \mu\text{m}$ .



wt%	Area 1	+/-	Area 2	+/-	Area 3	+/-	Area 4	+/-
Fe	6.08	0.01	7.51	0.01	7.87	0.01	8.19	0.01
Ca	0.0205	0.0002	0.0270	0.0007	0.0228	0.0003	0.0607	0.0005
Cr	0.0104	0.0003	0.0141	0.0003	0.0229	0.0015	0.0311	0.0010
Ni	0.2685	0.0006	0.2523	0.0005	0.2375	0.0008	0.1340	0.0006
Mn	0.0844	0.0004	0.0939	0.0004	0.0873	0.0006	0.1340	0.0007
pixels	445		632		263		321	

**Figure 3.** Data extraction from Olivine map, with extraction areas highlighted on quantified Ni map. Corresponding area compositions are listed for selected elements with errors and numbers of combined pixels listed in table. Non-Olivine pixels blanked. Scalebar =  $100 \mu\text{m}$ .



**Figure 4.** Extracted area compositions in wt% (x axis) for a range of Udachnaya-East Olivine grains plotted against point analyses of same areas (y axis).

## References:

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