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Compositional Mapping by EPMA and µXRF

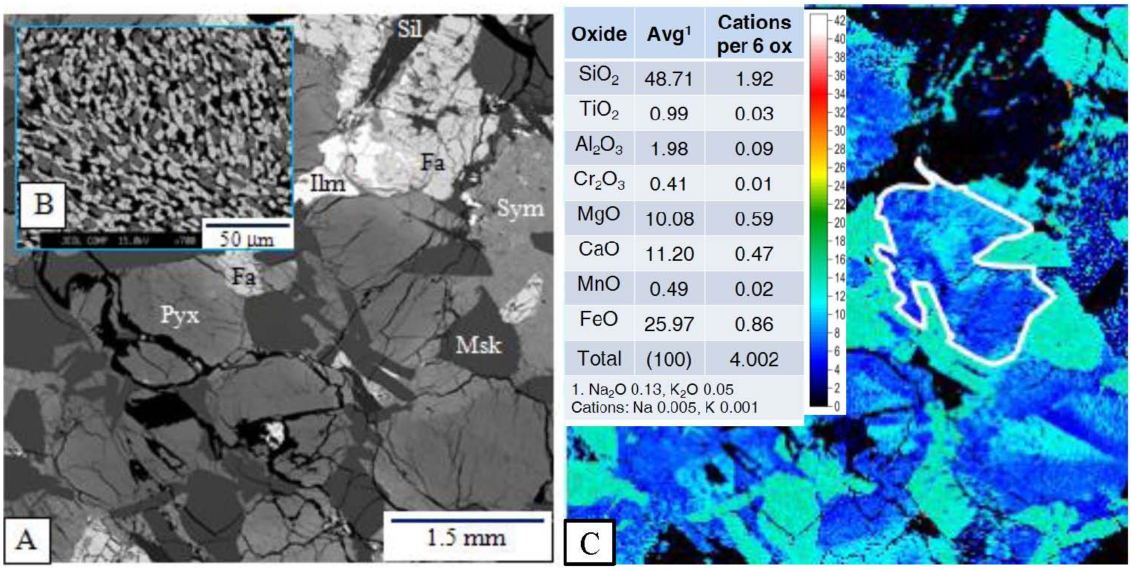
P. K. Carpenter[1], B. L. Jolliff[1], and J. J. Donovan[2]
[1]Dept. of Earth and Planetary Sciences and the McDonnell Center for the Space Sciences, Washington University in St. Louis, Campus Box 1169, Saint Louis, MO, 63130, USA
[2]CAMCOR, University of Oregon, Eugene, OR 97403, USA

We present methods combining backscattered-electron (BSE) mosaic imaging, quantitative spot-mode electron-probe microanalysis (EPMA), and quantitative compositional mapping by EPMA and micro-x-ray fluorescence (µXRF) to provide a framework for detailed analysis of terrestrial and lunar samples. BSE imaging provides a base map for the characterization of samples by EPMA. Recent developments in image stitching provide a convenient method of processing BSE mosaic image sets. Characterizing cm-sized samples by EPMA and µXRF provides complementary information about sample chemistry.

We acquire BSE mosaic and x-ray stage maps on a JEOL JXA-8200 electron microprobe equipped with five WDS spectrometers and a silicon drift EDS (SDD) that are used to acquire major and minor element maps. Quantitative EPMA maps are acquired using Probe Image, Probe for EPMA, and CalcImage software using conventional EPMA standardization, mean atomic number (MAN) background correction, and a full Φ(pz) correction at each pixel in the map. Maps are acquired with 400-1024 pixels at 0.X-15 µm and 10-100 msec per pixel at analytical conditions of 15kV and 50-100 nA. Large areas are covered by selection of appropriate pixel resolution and step size. An EDAX Orbis µXRF equipped with an SDD is used for spectrum image stage mapping using a 30 µm polycapillary optic, similar pixel resolution, and dwell times of 50-500 msec.

Lunar meteorite Miller Range 05035, a low-Ti mare basalt, illustrates the capabilities of quantitative EPMA stage mapping (Fig. 1). It contains complexly zoned pyroxene, maskelynite, and symplectite regions made up of silica, fayalite, and hedenbergite [1]. Maps have been used to calculate per-pixel analyses, cross-sections, and irregular polygon subsets used for the calculation of local bulk composition. We have previously used these data to calculate local average compositions of the symplectite that compare well with the composition of lunar pyroxferroite; this method is more accurate than defocused-beam analysis [2]. A quantitative Ca concentration map is shown in Fig. 1C, with an irregular polygon outlining a zoned pyroxene. The polygon outline is used to define all pixels belonging to the zoned pyroxene and is used to determine the average pyroxene composition. Subsets of the pyroxene polygon can be used to isolate sector zones and core vs. rim compositions. The mineral stoichiometry from the cumulative pixel average is excellent and equivalent to that from conventional EPMA point analysis.

A comb-layered xenolith from a cinder cone in the San Francisco volcanic field of Arizona illustrates combined EPMA and µXRF mapping capabilities. This sample contains multiple layers of mm-length dendritic plagioclase, sector-zoned Ca-pyroxene, euhedral to granular olivine, and quenched glass. It represents directional solidification during rapid cooling from a basaltic melt. Quantitative EPMA stage maps highlight olivine, pyroxene sector zoning, and glass chemistry (Fig. 2A). The µXRF maps permit a larger mapping area on a polished slab of the xenolith, and highlight both major and trace element zoning (Fig. 2B). For example, the variation in Ti, Cr, and Mn in the sector-zoned pyroxene and interstitial trapped glass is displayed in a RGB composite image. The increase in plagioclase Sr content is seen by µXRF using the Sr Kα line, but was not observed by EPMA using the Sr Lα line. The high spatial resolution of the EPMA maps and spectrum imaging data cube obtained using the µXRF provide complementary data to analyze texturally and compositionally complex materials.



Oxide	Avg[1]	Cations per 6 ox
SiO_2	48.71	1.92
TiO_2	0.99	0.03
Al_2O_3	1.98	0.09
Cr_2O_3	0.41	0.01
MgO	10.08	0.59
CaO	11.20	0.47
MnO	0.49	0.02
FeO	25.97	0.86
Total	(100)	4.002

1. Na_2O 0.13, K_2O 0.05
Cations: Na 0.005, K 0.001

Figure 1. (A) BSE stage map of MIL05035. Zoned Ca-pyroxene (Pyx), maskelynite (Msk), fayalite (Fa), ilmenite (Ilm), silica (Sil), and symplectite (Sym). **(B)** BSE of symplectite with dark silica, gray hedenbergite, and bright fayalite (mag. 700x). **(C)** Quantitative Ca wt. % map with concentration scale and average analysis of Ca-pyroxene from irregular polygon (shown in white).

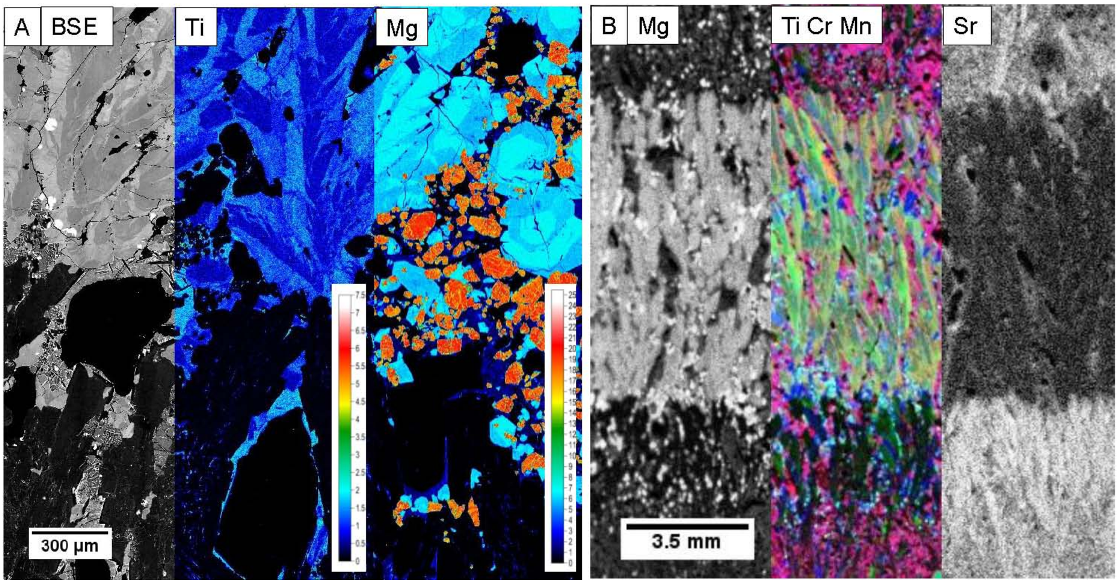


Figure 2 A and **B** Comb layer xenolith. **(A)** Sample from 1" polished microprobe mount with EPMA images acquired at layer boundary over 2.25 mm square area. Images are BSE and quantitative Ti and Mg wt. % maps with wt. % scale. EPMA map: 800x800 pixels, 3 µm and 50 msec per pixel, 100 nA at 15kV. **(B)** Sample from 21 mm by 12 mm area of polished slab from same xenolith. Images are µXRF x-ray intensity maps for Mg, RGB map using Ti, Cr, and Mn, and Sr map using Sr Kα. The µXRF map: 512x400 pixels, 41 µm and 500 msec per pixel, 600 µA tube current at 30 kV. Note difference in scale as µXRF area is factor of 10 larger. Each figure shows three different signals superimposed on the total base map area.

References:
[1] Zeigler *et al*, LPS XXXVIII (2007), #2110.
[2] Carpenter *et al*, LPS XXXXIV (2013), #1827.