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Very Large Area Phase Mapping of a Petrographic Thick Section using Multivariate Statistical Analysis of EDS Spectral Images

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There are many applications that require knowing the correct distribution of phases in a sample, such as determining the quality of steel or determining the bulk composition of a sample by modal recombination. Modal recombination is a calculation of a sample's bulk chemistry based on the assumption that the distribution of phases in a cross section of a sample is a good representation of the distribution of phases in the three dimensional volume of the sample. If the abundance of each phase and the composition of each phase are known, the bulk composition of the sample can be calculated. The greater the area of the section from which the distribution of phases is determined, the more accurate the bulk composition calculated from a modal recombination can be. Large area electron imaging, elemental X-ray mapping, and quantitative elemental X-ray mapping have existed for some time. Historically, most modal recombination calculations have been done relying on the phase abundances determined from the analysis of contrast in backscattered electron image which discriminates phases based on average atomic number. However, many phases that commonly occur in the same sample have similar average atomic numbers (e.g., pyroxene and apatite) and may be impossible to successfully discriminate based on electron image contrast. Here, we demonstrate that modern X-ray microanalytical software, if it includes multivariate statistical analysis algorithms, enables phase mapping based on X-ray microanalysis of very large areas (including petrographic thin or thick sections) in practical amounts of time.

The sample analyzed in this study is a 4.5×2.5 cm petrographic thick section of olivine-chromite cumulate from the Stillwater igneous complex [1] in Montana, USA (hereafter, "Stillwater sample"). The section was polished and coated with carbon in preparation for X-ray microanalysis. Analysis was done in an FE-SEM with an accelerating voltage of 15 kV a probe current of ~18 nA. X-rays were detected using dual (a 10 mm^2 and a 30 mm^2 active area) UltraDry EDS detectors. EDS spectral images were acquired in a grid over the sample and processed into a 10808×7587 pixel montage of phase maps using the Thermo Scientific Pathfinder microanalysis system. Each spectral image is 256×192 pixels with a resolution of 2.955 μm per pixel yielding in a phase map montage that covers an area of 3.19×2.24 cm^2. Each spectral image was acquired until it contained ~1000 counts per pixel. Phase maps were extracted from the spectral images using the COMPASS algorithm for multivariate statistical analysis [2,3]. The spectral images were acquired with far more (~100×) data than needed for phase mapping using the COMPASS algorithm [4] because they will also be used for extracting quantitative elemental maps in future work.

The result of the COMPASS-based phase mapping of the Stillwater sample is Fig.1. A cursory inspection of Fig. 1 reveals heterogeneneity, which is most notable in the lower-right corner where there is much more chromite (red) and high-Ca pyroxene (yellow) relative to low-Ca pyroxene (cyan) compared to the rest of the sample. If only a portion of the sample were mapped, different conclusions might be drawn regarding the petrology and bulk composition of the sample. Olivine grains are rounded and typically embayed by low-Ca pyroxene, consistent with the olivine grains reacting with melt to

crystallize low-Ca pyroxene. The serpentine is localized to fractures near olivine and likely formed as a result of olivine reacting with water after the sample fully crystallized.

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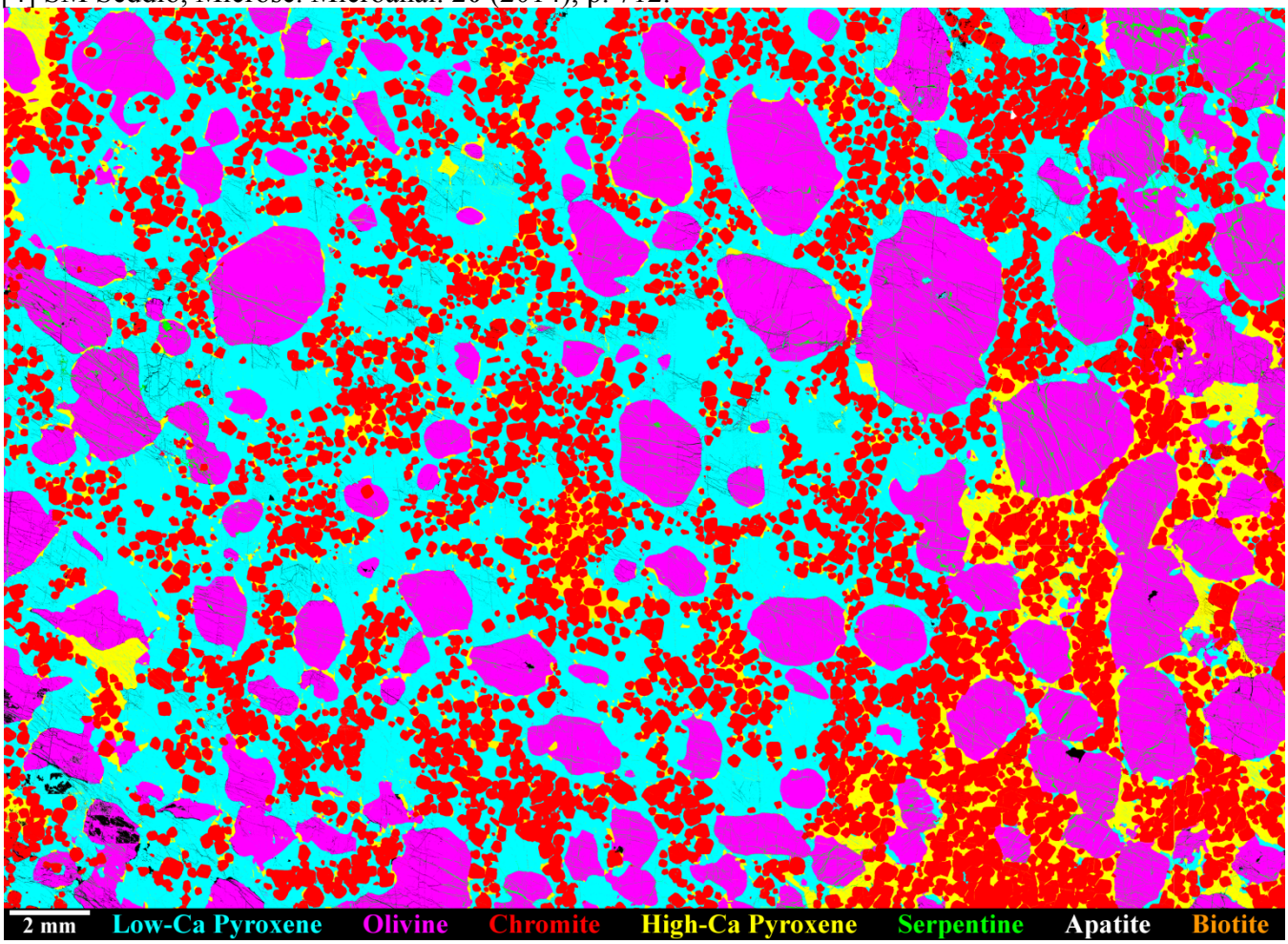

Figure 1. Very large area COMPASS-based phase map.

Table 1.

Phase	Low-Ca pyroxene	Chromite	Olivine	High-Ca pyroxene	Serpentine	Other*	Apatite	Biotite	Total
Pixels	27956832	24234523	23041177	4684864	1305581	661373	2560	1506	81888416
Area%	34.14	29.60	28.14	5.72	1.59	0.81	0.003	0.002	100

*"Other" is black in Fig. 1 and includes surface contamination, charging, and topography mostly associated with fractures.

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