doi:10.1017/S1431927614005042
Microsc. Microanal. 20 (Suppl 3), 2014

Mineral Classification Using Computer-Controlled Scanning Electron Microscopy

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Computer-controlled scanning electron microscopy (CC-SEM) is an automated technique combining electron microscopy, image analysis, and X-ray spectroscopy to rapidly acquire morphological and chemical information for thousands of individual particles. Measurements are typically performed on polished cross-sections of material embedded in epoxy mounts. Individual particles are first identified from the epoxy matrix based on user-specified criteria (e.g., pre-set grayscale threshold from a representative Back-Scattered Electron image). Following identification, morphologic parameters (e.g., area, perimeter, aspect ratio, etc.) and an X-ray spectrum (EDS) are recorded for each particle. Multiple fields of view can be analyzed, resulting in many thousands of individual particle analyses collected during a single CC-SEM session. The resulting data can then be mined for useful information like particle size distributions (PSDs), phase proportions, and even bulk chemistry.

We used a Hitachi Ultra-High Resolution Analytical SEM SU-70 equipped with a YAG-BSE detector and an Oxford EDS system to perform CC-SEM particle analyses on several samples of loose mineral mixtures. Representative sampling of the mixtures is essential and we developed a sampling strategy that: 1) ensures grains are randomly oriented so that observed area and volume fractions are equivalent [1]; and 2) mitigates the effect of gravitational settling of different minerals during epoxy curing. A mineral classification scheme was then developed using the INCAFeature add-on to the Oxford INCA Energy software package to determine mineral fractions present within the samples. Data were further reduced using Excel-based VBA macros to refine the initial classification and to calculate PSDs and bulk chemistry from EDS data.

Synthetic samples (~ 400 g each) were created using quartz, feldspars, clays, carbonates, and other minerals and were mixed for ~30-45 minutes prior to sampling. Mixtures were mounted and analyzed by CC-SEM to test the accuracy of the mineral classification scheme. Our classification scheme was able to accurately classify these mineral mixtures to within < 6 % (absolute) of expected values.

Particle size distributions were calculated for the bulk samples (i.e., ignoring mineral types) from CC SEM data by binning features according to equivalent circle diameters (ECD) and summing up areas within each bin. Comparison between PSD metrics estimated from the CC-SEM data with those determined by Coulter Multi-sizer (Electrical Sensing Zone method) yield values within ~20% of one another. However, comparisons between the two methods for the synthetic mixtures show significant discrepancies because of clay agglomeration within the epoxy mount. We discuss some potential solutions to this problem.

Finally, we compare bulk chemistries calculated using mineral proportions and averaged EDS compositions with data obtained by ICP-OES and Leico CO2 analyses. Most elements agree to within 3 wt%, with SiO_2 and CO_2 contents showing greater discrepancies of ~5-6 wt%.

Our study demonstrates the utility of CC-SEM analyses for analyzing complex systems composed of many phases. We also demonstrate that PSD and bulk chemistry data obtained by CC-SEM are comparable to more conventional techniques, however this method may be applied in unique situations where conventional analyses are not possible (e.g., *in situ* analyses).

References:

[1] E.E. Underwood in "Stereology", ed. H. Elias, (Springer-Verlag, New York) p.49-60.

https://doi.org/10.1017/S1431927614005042 Published online by Cambridge University Press