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Re-sampling of SEM-EDS Element Maps to Characterize the Length-Scale of Elemental Heterogeneity

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The degree of mixing can be an important factor affecting the final quality of solid blends from various processes. Various mixing and milling techniques are typically applied to increase the homogeneity of the compound. Bulk analysis techniques such as XRF, ICP and NAA can provide elemental data on the scale of grams to milligrams, but the measurement of heterogeneity at smaller scales is difficult to assess. In addition, those techniques can be labor-intensive and time consuming to acquire a large number of analyses for statistical purposes. This presentation will illustrate the use of SEM-EDS element maps to quantitatively characterize the elemental heterogeneity as a function of length scale in a statistically rigorous fashion.

X-ray microanalysis maps from SEMs or electron microprobes can collect elemental information down to the sub-micron level and up to areas covering millimeters or more. Re-sampling of these element maps is a standard image analysis technique that has not been widely applied in this area. The number of pixels of the map is reduced by either adding or averaging the signal from 4-pixel regions. This procedure is repeated up to the given pixel dimension of the map. The standard deviation of the X-ray counts per pixel for the original and each re-sampled map is calculated. Plots of the standard deviation as a function of the pixel size show the nature of the change in elemental variability as a function of scale.

The method was applied to ceramic mixtures (Figure 1). The effectiveness of milling/mixing techniques can readily be quantified and compared. Results on multiple areas within one sample were reproducible. Counting statistics can be applied to the analysis to identify data that are below the detection limit for the given map. Analysis of the counting statistics compared to the variability signal can be used to tailor acquisition dwell time for the task at hand, minimizing the length of time per map while maintaining the needed level of sensitivity.

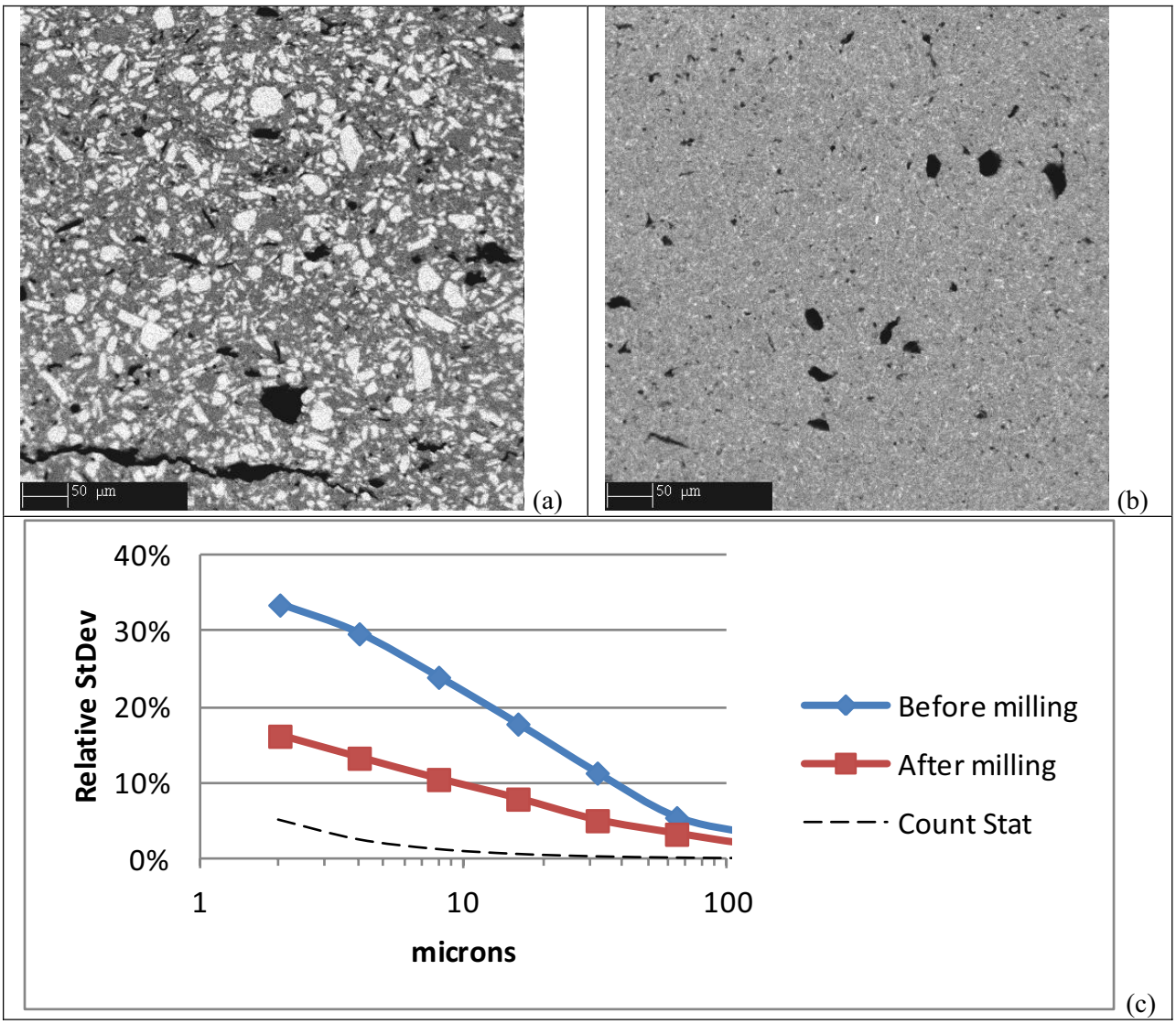

Figure 1. Aluminum element maps for a mixture of alumina and clay before milling (a) and after milling (b). Field of view is 250 µm. Brightness is a function of aluminum content. (c) Heterogeneity calculation as a function of length scale for these two maps. The detection limit based on X-ray counting statistics is also plotted.

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