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Re-sampling of SEM-EDS Element Maps to Assess 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 (for instance XRF, ICP and NAA) can provide elemental data on the scale of grams to milligrams, but heterogeneity at smaller scales is difficult to assess. In addition, these techniques can be labor-intensive and time consuming to acquire a large number of analyses for statistical purposes. Some chemical reaction processes have length-scales of transport phenomena smaller than this. This presentation will illustrate the use of SEM-EDS element maps to quantitatively assess the degree of elemental heterogeneity down to the micron 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, as applied here to gray-scale images, is a standard image analysis technique whereby 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 average and standard deviation of the X-ray counts per pixel for the original and each re-sampled map are 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 effectiveness of various milling techniques can readily be quantified (Figure 1). Counting statistics can be applied to the analysis to exclude data that are below detection 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. Insight can also be gained from fractal analysis of the data sets.

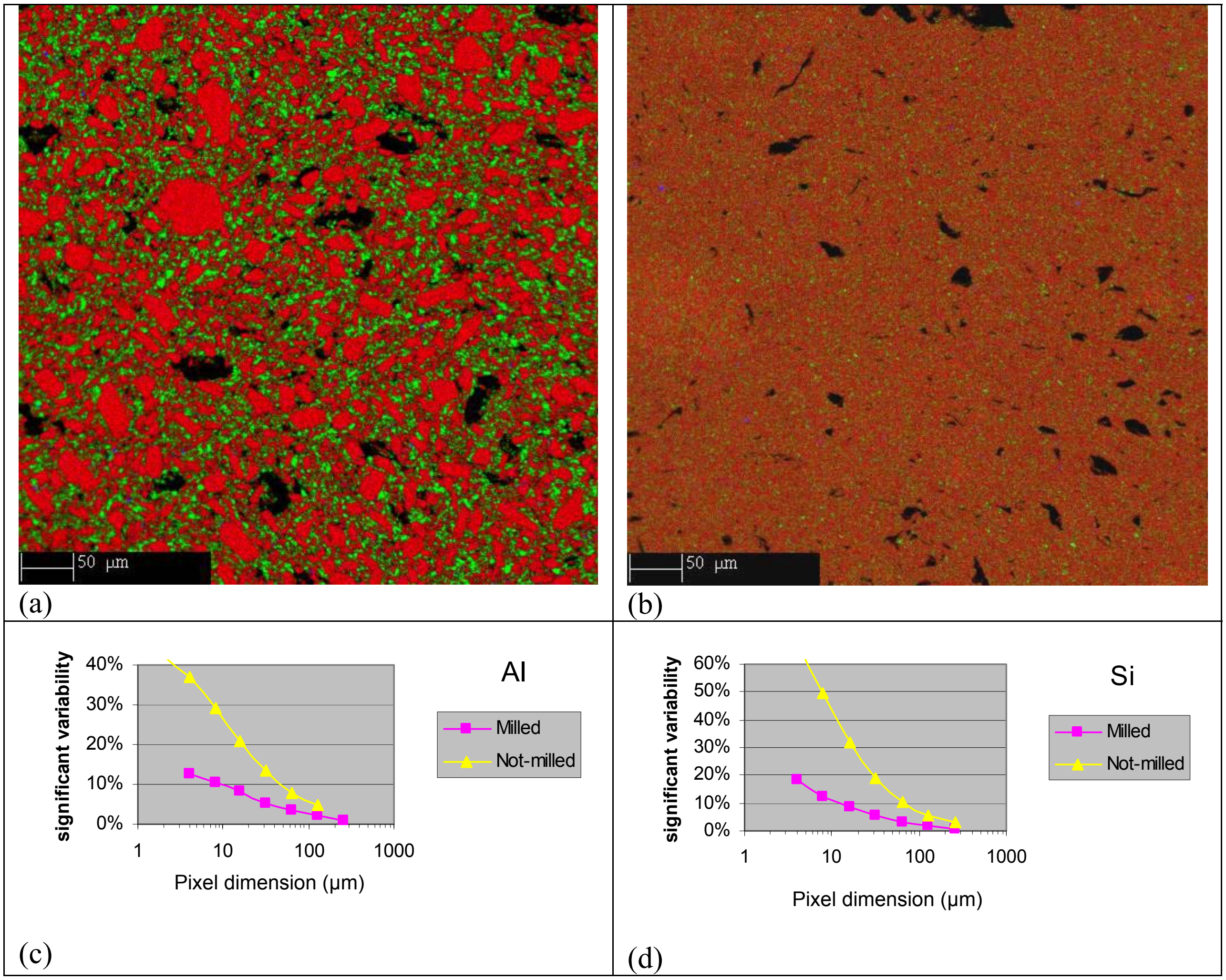

Figure 1. False-color combination element maps for (a) not-milled and (b) milled mixtures of aluminum oxide and silicon oxide. Red = Al element map; Green = Si element map. Re-sampling plots for (c) Al and (d) Si; the Y-axis is the standard deviation of the averages of the re-sampled maps as a percentage of the average of the element signal for the entire map. The decrease in elemental variability due to milling can be quantified. There is about four times less variability at a given length-scale. Alternatively, the length-scale is decreased by a factor of four for a given level of variability.

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