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SEMantics for High Speed Automated Particle Analysis by SEM/EDX

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Automated particle analysis by scanning electron microscope (SEM) with energy dispersive x-ray spectrometer (EDX) has been available since soon after laboratory-scale computers were first introduced[1-2]. These systems were much slower than the current state-of-the-art, yet they were still a significant advance over what the most dedicated analyst could do by hand. Particle analysis is different from classic x-ray microanalysis. In classic x-ray microanalysis, the emphasis is on collected high precision data on a small number of representative samples. In automated particle analysis, the emphasis is on collecting moderate precision data on thousands or tens-of-thousands of microparticles. Because of particle geometry effects collecting high precision spectra is a waste of time and ultimately, the utility of the analysis is determined by the precision of the particle population statistics or the ability to find rare particles. Even a slow automated system can collect an order-of-magnitude more particle size and spectrum data items than a dedicated manual operator.

The early automated systems were limited in their ability to search and find particles by slow scan control electronics, slow backscatter detectors, large spot sizes, slow stages and long CPU cycle times. The chemical typing of the particles was limited by slow Si(Li) detectors with analog pulse processors and slow CPUs performing quantitative analysis. We have come a long way. Today a high brightness electron beam from a Schottky field-emission source can be rastered with sub-100 ns dwell times and a well designed backscatter detector and amplifier can both keep up and produce near count statistics limited signal-to-noise. Stages are much faster and more precise. Modern CPUs and digital signal processors are capable of making decisions in nano-seconds rather than microseconds. Finally, large area (30 mm^2) silicon drift detectors are capable of approximately two orders-of-magnitude increased output count rates and can be multiplexed to further the advantage.

We set out to design the fastest possible automated particle analysis system given commercially available 2011 technology. A TESCAN MIRA 3 Schottky field-emission microscope was used to provide a high brightness (small diameter at high probe current) electron beam†. We estimated that a probe current of 1 to 5 nA would be a sweet spot to balance probe diameter, backscatter signal and x-ray signal. We paired this with a high solid angle backscatter detector with fast, near signal-to-noise limited electronics. A probe current of 1 nA on Cu ($\eta = 0.31$) will produce about 2,000 $e^-/\mu s$. A detector with a collection efficiency of 20 % will collect around 400 μs^{-1} for a count statistics limited signal-to-noise of 20:1. This is adequate for use in a threshold mechanism.

The Schottky SEM was paired with four PulseTor 30 mm^2 silicon drift detectors. At 1 nA of probe current these detectors summed together produce a output count rate of over 200 kHz on Cu at 25 keV. At this rate, it is not necessary to spend more than a hundred milliseconds per particle to get adequate count statistics to identify elements at the major (>0.10 mass fraction) and minor (>0.01 mass fraction) levels. Furthermore, the four spectra per particle can be compared to help ameliorate common issues such as shadowing and differential absorption.

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TESCAN has implemented a highly optimized algorithm for searching and measuring particles in real-time. The microscopy will be automated using a NIST-developed Java library called *SEMantics* to control the TESCAN MIRA 3 through the TCP/IP-based SharkSEM API. SEMantics is being implemented as an extension to NIST DTSA-II to take advantage of its x-ray processing algorithms. SEMantics will record all images, spectra, x-ray spectrum images and measurement data to a PostgreSQL structured query language (SQL) database. Associated data like standard spectra and calibration data will also be stored in the database. The SQL database will make it easy to reanalyze data offline at any time immediately or in the future using the chronologically appropriate standards and calibrations. Each data set will be associated with a project and a sample facilitating reporting. Archiving all the particle data in a single database will facilitate studying relationships between samples and projects. A hashing algorithm has been developed to facilitate quickly identifying particles with similar composition even when the database grows to contain millions of particle records. The database structure will allow the individual microparticles to be assigned to multiple classes simultaneously which can occur either as the result of successively more precisely defined classes or as the result of classification using various schemes or rule sets.

Our goals for this system are to be able to find, size and compositionally type 10,000 particles per hour or approximately 3 particles per second under optimal conditions. This throughput is beneficial to search large particle samples and to extract either meaningful population statistics or to find rare particle classes. We believe we have put together the hardware necessary to accomplish this goal. What remains is careful system design to optimize the flow of information from the imaging subsystem, the x-ray sub-systems, through the SEMantics library and into the SQL database.

[1] M. F. Hoover, E. N. White, J Lebiedzik, G G Johnson, *Automated characterization of particulates and inclusions*. Microbeam Analysis (1975) 54A-54B.

[2] S. Ekelund, T. Werlefors, *A system for the quantitative characterization of microstructures by combined image analysis and X-ray discrimination in the scanning electron microscope [with minicomputer control]*. Scanning Electron Microscopy I (1976) 417-424

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