2180
doi:10.1017/S1431927617011564
Microsc. Microanal. 23 (Suppl 1), 2017

Requirements for a Complete Geological Analysis Solution with EDS

M Hiscock

Oxford Instruments NanoAnalysis, Halifax Road, High Wycombe HP12 3SE, UK

The automated analysis of geological materials presents unique challenges which are not seen elsewhere in microscopy. Whilst such materials are typically stable under an electron beam and can sustain higher energy analysis enabling high throughput rates, the information that is required from them is needed in very specific forms and care must be taken in the analysis process to ensure that the best results are achieved in a timely fashion. This must be combined with a high degree of ease of use. Many operators of systems of this kind are not microscopists and simply want the right answer to be found in as short a time as possible with the minimum level of complexity.

In order to achieve the goals listed above, a system must be optimized on many fronts. This is because when running automated analyses we must rely on the results produced by the system without feeling the need to review each particle/feature which is measured. As such, we must have well characterized detectors which allow a high throughput whilst maintaining a good resolution. These must be paired with dedicated electronics which have undergone a similar characterization process in order to handle the data from the detector appropriately. The next step in the chain is to have software algorithms which are precisely matched to the rest of the measurement chain to capture the digital data on the detected x-ray counts and turn it in to something that we can make use of. Only when we have this level of hardware and software can we then build on top of it to make a truly reliable and fast automated mineralogy system.

Our next requirement is automated algorithms to process and correct for any artifacts in our data. This should include (for example) pulse pile-up, back ground corrections or peak overlap deconvolutions. We then need to perform a quantification of our data. All of this needs to be done near instantaneously in order to achieve the throughput we desire of our system. Finally, on top of these algorithms, we can build our particle analysis platform and its dedicated implementation for mineral samples. Depending on the sample type we may chose a straightforward feature analysis, identifying features from the backscattered electron image (BSE) grey level, automatically obtaining a spectrum from each feature, classifying it according to a user defined classification and reporting the results for the entire area. We can enhance this process and potentially improve throughput by utilizing morphology filters to only analyze the features with the morphology we are interested in or EDS filters to control the duration of EDS acquisition based on the results of a fast, prior acquisition. We can make use of termination criteria to stop when we have discovered a certain amount of a given phase (or lack thereof) and we can choose to reconstruct any features which extend over field boundaries to ensure that the correct morphology and quantified composition is reported for them.

Depending on our requirements we could go further and record specific, mineralogical parameters such as degrees of liberation for each phase in particulated samples (see Figure 1 which shows liberation curves for sphalerite), or particle associations – determining for each phase if they are associated within particles on a primary, binary or tertiary basis. We can interrogate data, including combined datasets from different size fractions, for chemical or morphological properties – perhaps breaking them down into size bins as shown in Figure 2. For solid samples we can measure parameters such as common

perimeter associations to learn about the relationships between grains sitting next to each other. Visualising data is important for an intuitive understanding and full field views of data colored to the users' requirements (Figure 3) are particularly useful for this. We can also take advantage of mineral databases when setting up classification schemes to guide us in identifying the phases that we have measured. Figure 4 shows the relative proportions of the different phases in this sample as determined by automatically interrogating such a database.

Here we demonstrate an optimized workflow for an unknown mineral sample that takes advantage of multiple silicon drift detectors to go from a state of knowing very little about the sample through to a full characterization. This workflow takes advantage of basic point & ID analysis, single field EDS mapping, phase mapping, particle analysis (including automated phase detection for phases which cannot be separated from one another by grey level alone) (Aztec & AztecFeature) and dedicated mineralogical analysis in modal and particulated samples (AZtecMineral). We will also discuss how the addition of other analytical techniques can complement the dataset for an even greater degree of characterization.

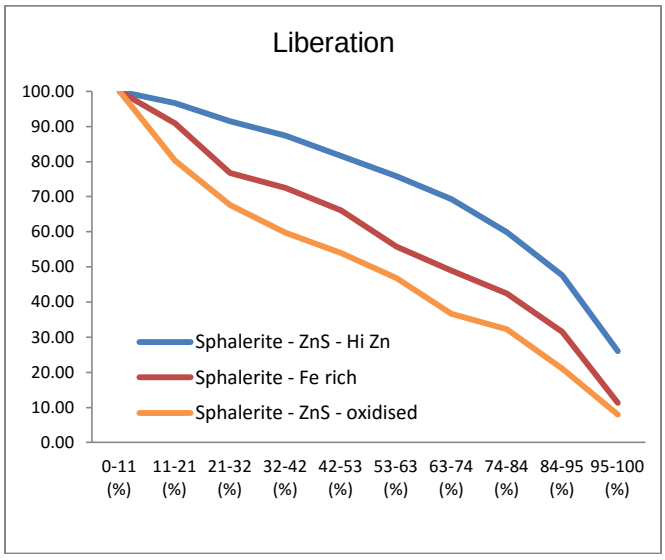

Figure 1: Example of Data Required from a Liberation Analysis of a Particulated Sample

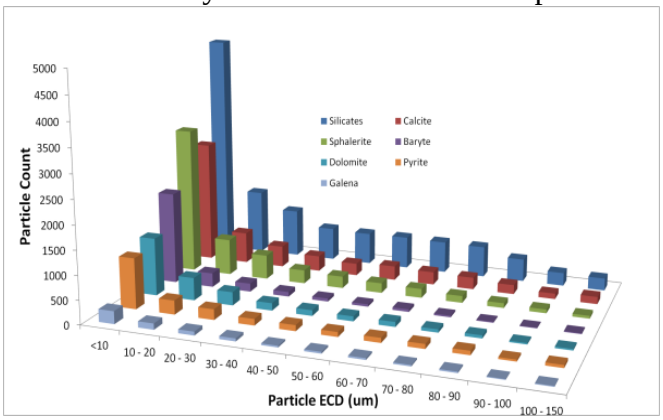

Figure 2: Grain Size Distribution for Selected Phases

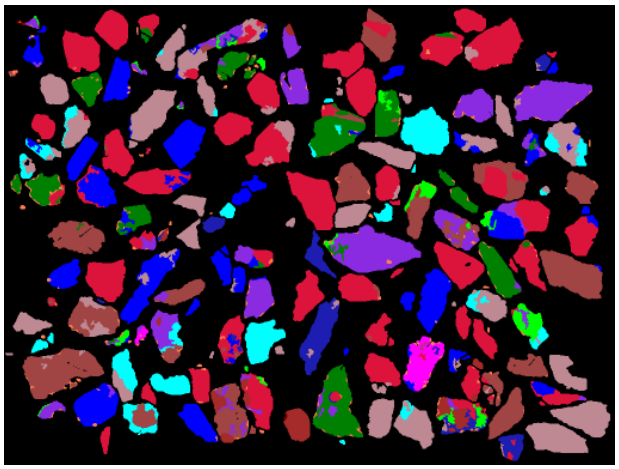

Figure 3: Full Field view of Measured Particles, Colored to User Specification

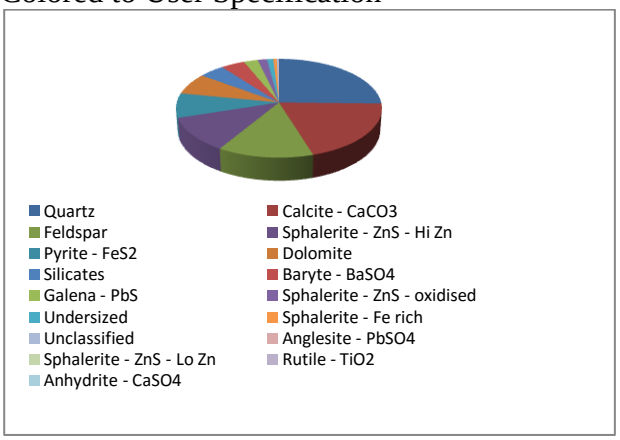

Figure 4: Phase Abundances as Calculated with AZtecMineral

https://doi.org/10.1017/S1431927617011564 Published online by Cambridge University Press