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Micro-Focused Five Dimensional X-ray Imaging with the Color X-ray Camera

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Addressing a micro-focused X-ray beam to a sample and recording the X-ray fluorescence spectrum using an energy dispersive detector has long been used as a microanalytical technique, known commonly as μXRF. Similarly, if the same focused X-ray beam is addressed to a sample in the forward scattering geometry (Bragg-Brentano) and a $\theta - 2\theta$ is performed, a diffraction pattern can be recorded, and the technique is known as μXRD. In both cases, an image can be created by recording data at an array of equally spaced points on the sample, which is typically accomplished by moving the sample with a motorized stage. However, if both diffraction and fluorescence data are required, then these measurements are typically made in separate instruments. The technique of μXRF imaging can be quite fast, but μXRD imaging, because it requires a $\theta - 2\theta$ scan at each point, can take days. Energy dispersive X-ray diffraction (EDXRD) enables the analysis of a wide range of d-spacings without the need for a $\theta - 2\theta$ scan, but this technique is difficult to use if the sample is compositionally complex, since the fluorescence and diffraciton peaks frequently overlap. This problem can be mitigated through the use of an imaging spectrometer, such as the pnCCD [1]. The pnCCD is integrated into the Color X-ray Camera (CXC), and it is capable of recording the position and energy of each X-ray event that hits the detector, with such data being recorded in a three-dimensional X-ray spectrum image (XSI) [2]. The CXC is able to collect both the diffraction and fluorescence data in as little as 1 ms with no moving parts, and then separate the XRD and XRD data analytically [3]. This method can be further enhanced by addressing a micro-focused X-ray beam to an array of points on a given sample and recording an XSI at each point, producing a five-dimensional dataset.

To demonstrate the usefulness of this new micro-focused five-dimensional imaging (μ5D) technique, a set of one hundred different mineral samples were mounted in epoxy and polished. The minerals represented a wide compositional range, coming from a number of common mineral groups such as silicates, feldspars and carbonates, some of which were polymorphs, differing only in crystal structure. The μ5D dataset was collected by scanning a polychromatic, $100\,\mu\mathrm{m}$ beam across an area measuring 18 mm x 14 mm with a pixel spacing of $100\,\mu\mathrm{m}$ per point. Each point only required 200 ms of acquisition time (approximately 2 hours total mapping time), with an average count rate of approximately 100 000 counts per second. The 180 x 140 point μ5D X-ray image contained 6.6 TB of data. The raw data were then reduced to just over 1 GB of data contained in two separate XSIs. The first XSI contained the fluorescence data with dimensions (X_{sample}, Y_{sample}, Energy), while the second contained the diffraction data with dimensions of (X_{sample}, Y_{sample}, θ). The data reduction process is shown in Figure 1. These two XSIs were further processed using an unsupervised machine learning algorithm to identify each of the minerals present, as shown in Figure 2. This is accomplished by first clustering the data based only on elemental images from the XRF data. Once clustered, the XRD spectra from each of the identified clusters are themselves clustered again to separate the spectra based on the similarity of the diffraction patterns. In this example, 9 XRD based clusters were created from 25 XRF clusters, leading to 225 potential materials. The final result is a database of materials identified through both the XRD and XRF data, requiring only two hours of data collection and data processing.

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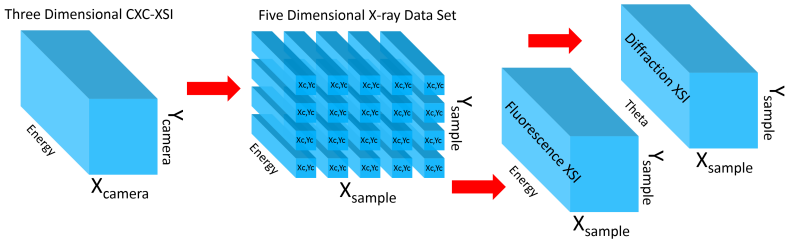

Figure 1: The basic data reduction strategy for a μ5D dataset. A focused X-ray beam is addressed to a single point on a sample and a full XSI is recorded. This process is done at each point in an equally spaced array on the sample, resulting in a five dimensional dataset. These data are reduced by separating the diffraction and fluorescence data and storing the resulting spectra in two separate XSIs.

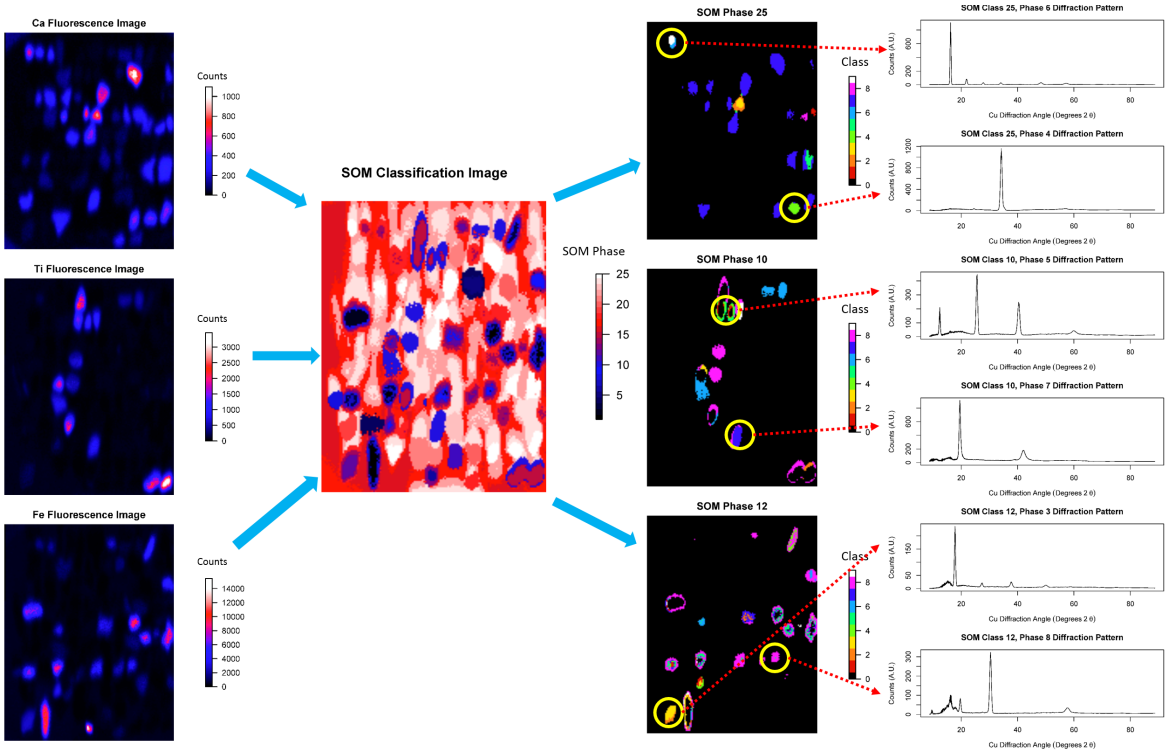

Figure 2: A summary of the unsupervised material classification technique used. The elemental images derived from the fluorescence XSI are provided to a self organizing map classifier, which identifies 25 separate phases. The diffraction patterns from the diffraction XSI belonging to each phase are then classified again, resulting in a unique material identification for each mineral in the sample.

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