Pushing the Limits of Lab X-ray (Micro)Diffraction

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In the Department of Mineralogy, we have coupled a high brightness X-ray source (Bede MicroSOURCE) to a rapid X-ray detector (INEL CPS120). This combination has allowed us to trade off some of the brightness and use a smaller beam for microdiffraction-type applications or use the full intensity of the beam to investigate very rapid phase transitions at non-ambient temperatures.

Lab microdiffraction is not a new concept; Debye-Scherrer and Gandolfi methods have been routinely carried out for decades. However, the main drawback with these techniques is that data collection times are long (many hours-days). Data collection times with the Bede MicroSOURCE/Inel Position Sensitive Detector are short; only a few minutes are required.

This presentation summarises a few of the problems tackled with this set-up:

Within a broken sample of Carbonado Diamond, a patch, approximately 300µm in diameter, of delicate crystals (10µm or less in diameter) with different morphologies, was discovered. Electron Microprobe analysis found the crystals contained varying amounts of **Ag,Ti,Cu** and **N**. In order to identify the phases present, *in situ* XRD was carried out; five phases were identified in this specimen three of which were found to be new minerals. This demonstrates how particles, or grains can be analysed *in situ* as a specific part of a polished block/rock sample.

Dilute microdiffraction was carried out to analyse particulates collected on a polycarbonate filter, used to monitor the amount of Pb in contaminated air. Elemental analysis was carried out using SEM-EDS, but XRD was required to identify the phases present. The SEM analysis determined the approximate amount of particulates on a $1cm^2$ piece of filter to be 147µg. By rotating the sample non-centrosymmetrically, a large area of the filter was irradiated by the X-ray beam.

The Bede MicroSOURCE/Inel Position Sensitive Detector system can also be coupled to an Anton Paar Heating Stage. This allows very rapid phase transitions of materials to be investigated at non-ambient temperatures over subtle changes in temperature by taking “snapshots” of the diffraction data every few seconds. The dehydration stages of Melanterite, $FeSO_4.7H_20$, have been investigated using this set-up. At temperatures close to ambient (25-40°C), very rapid structural changes to other hydrated iron sulphates, e.g. Rozenite, $FeSO_4.4H_2O$, have been detected.