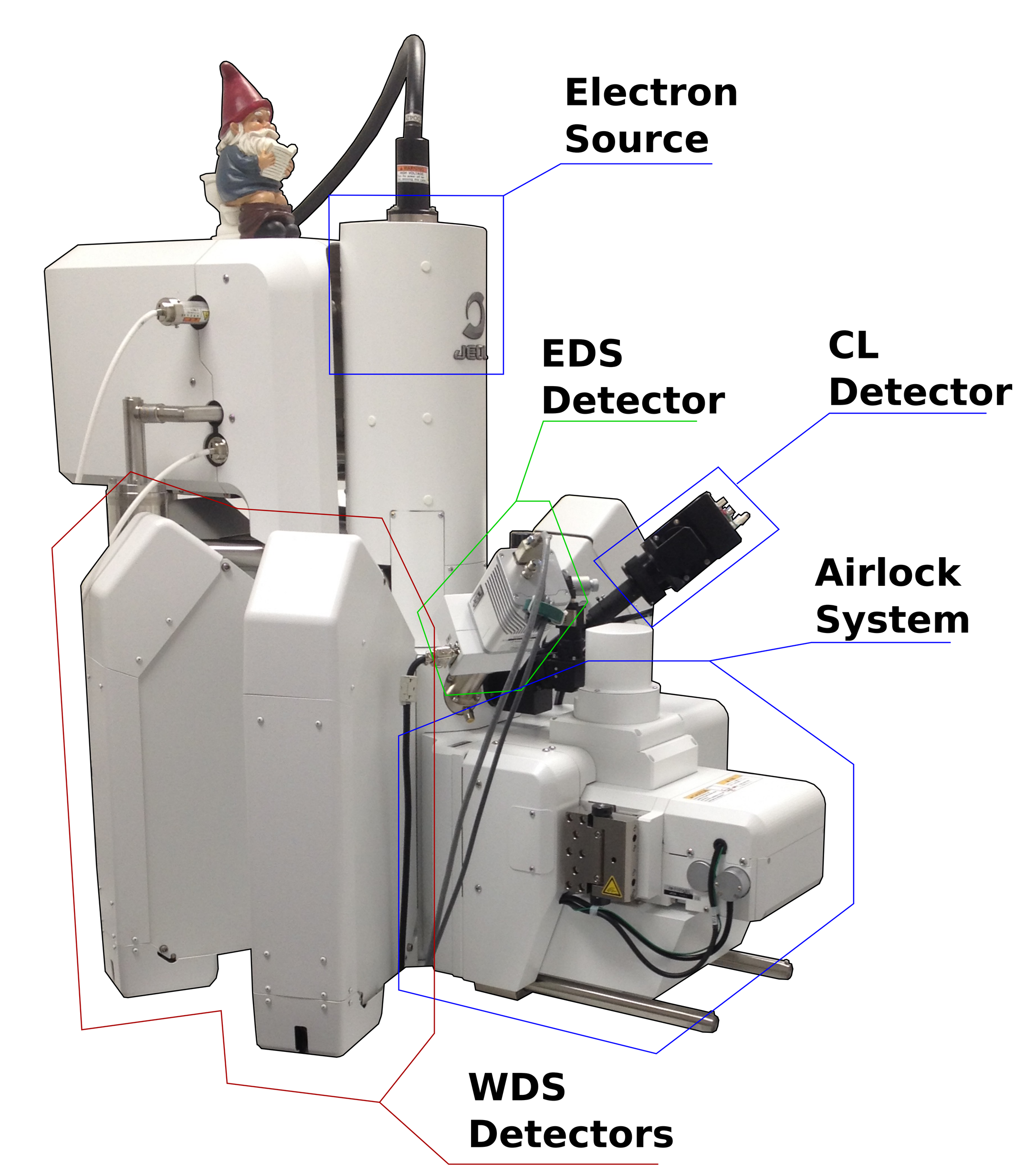
JEOL JXA-iHP200F Electron Probe Microanalyzer (EPMA)

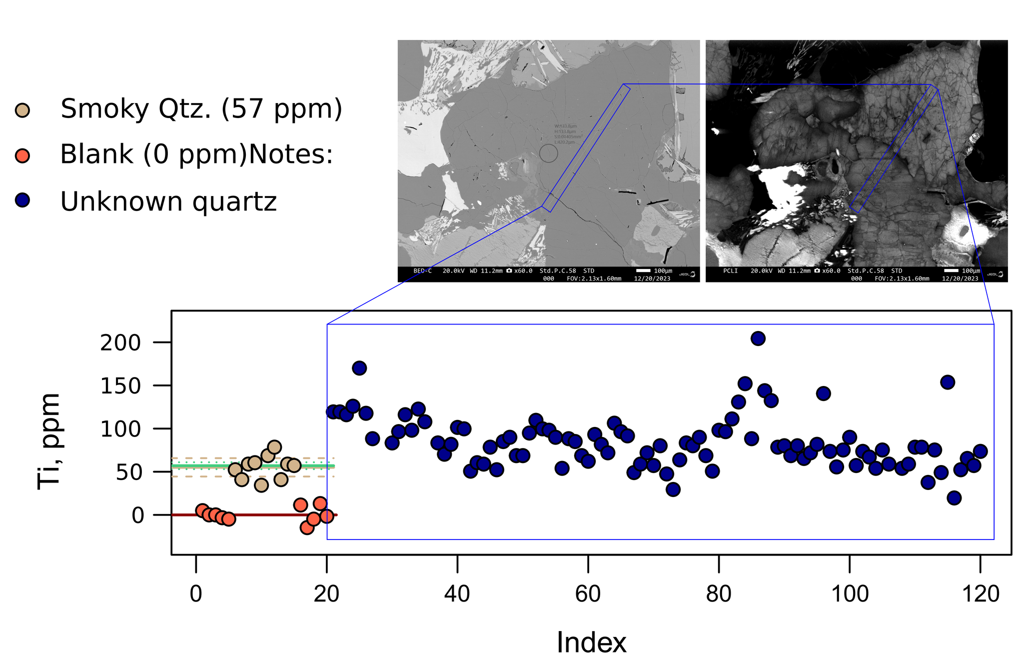


Electron microprobe hardware

The electron probe microanalyzer (a.k.a. "electron microprobe", or just "microprobe") is a specialized scanning electron microscope (SEM) for use cases which prioritize the elemental composition of a sample. In addition to the energy-dispersive X-ray spectrometer (EDS) common on most SEMs, the microprobe is equipped with five wavelength-dispersive X-ray spectrometers (WDS) which provide superior energy resolution and detection limits compared to EDS. In addition to secondary electron (SED) and backscattered electron detectors (BED), the microprobe is also equipped with a panchromatic cathodoluminescence imager (PCLI) for observing chemical zonation in certain geological materials or fluorescent compounds.

The microprobe uses a field emission X-ray source to produce a bright, highly focused electron beam, which allows for excellent spatial resolution and high X-ray count rates over a small area. The system also has a fully automated airlock system used to introduce samples with minimal atmospheric contamination. JEOL software allows for automated WDS standardization and quantification as well as integration with the EDS detector for simultaneous WDS+EDS acquisition (e.g. EDS for major elements and WDS for trace elements).

Trace element quantification

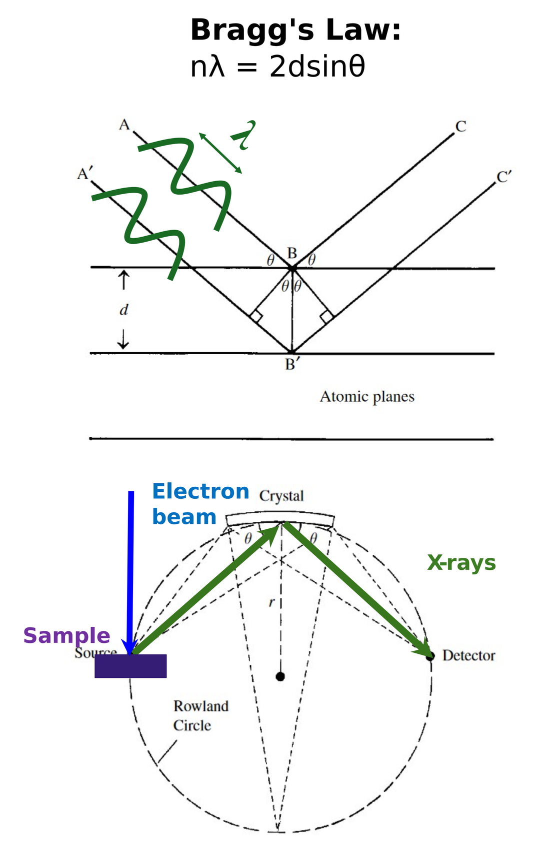


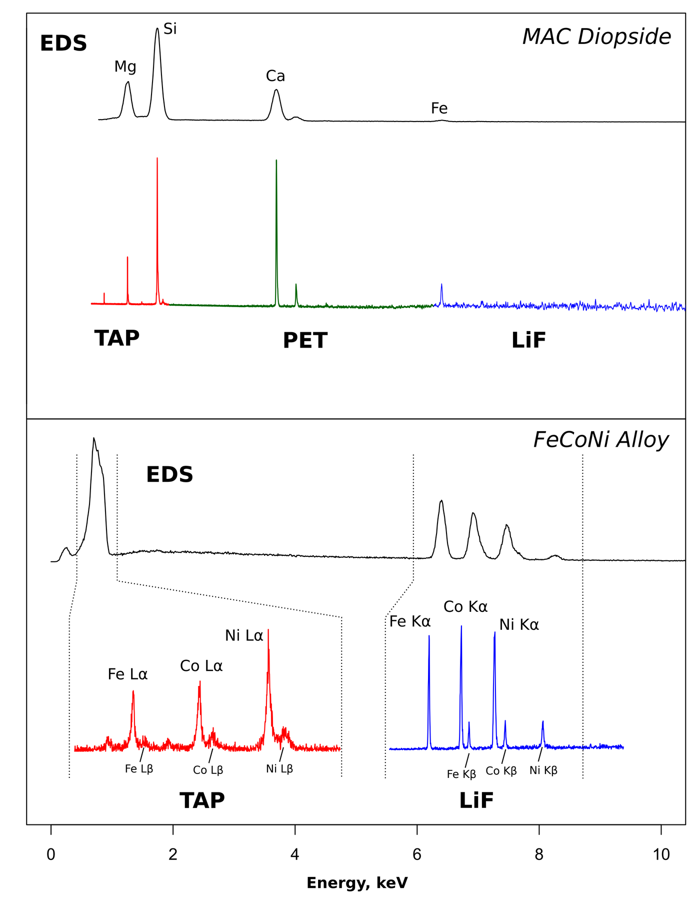
When conditions are favorable, the microprobe can quantify the composition of an analyte down to 10s of ppm. For example, the concentration of trace Ti in the mineral quartz (SiO2) is used by geologists to estimate formation temperatures in igneous and metamorphic rocks. We calibrated our WDS detectors using TiO2 and SiO2 standards and then used four of our five WDS spectrometers to simultaneously count on the Ti Kα X-ray to maximize the count rate.

The images above show analyses of a synthetic quartz blank (0 ppm) and a smoky quartz reference material (57 ppm) used to check for accuracy. Analyses were collected along a transect of 100 points spaced 10 microns apart (5-micron beam diameter). The transect location is shown in the secondary electron (SEI) and cathodoluminescence (CLI) images above. The Ti concentration is correlated with the CL brightness, which suggests a complex temperature and alteration record in the rock.

What is WDS?

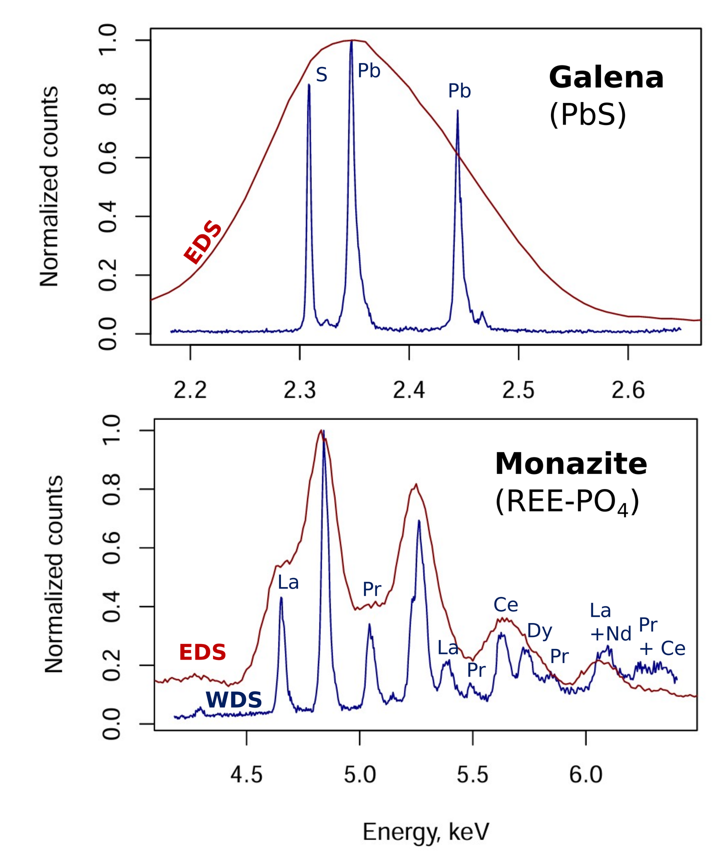
WDS detectors use a crystal monochromator to isolate and count X-rays using the principle of Bragg diffraction (analogous to X-ray diffraction; XRD). If an X-ray satisfies Bragg's Law (nλ = 2dsinθ), it is able to travel from the sample to the detector and be counted. The WDS system allows the user to select one of several crystals with known interatomic spacings (d) and control the incidence angle of the X-ray path relative to the crystal and detector (θ). This process of X-ray filtering produces superior energy resolution and signal-to-noise ratios compared to EDS.



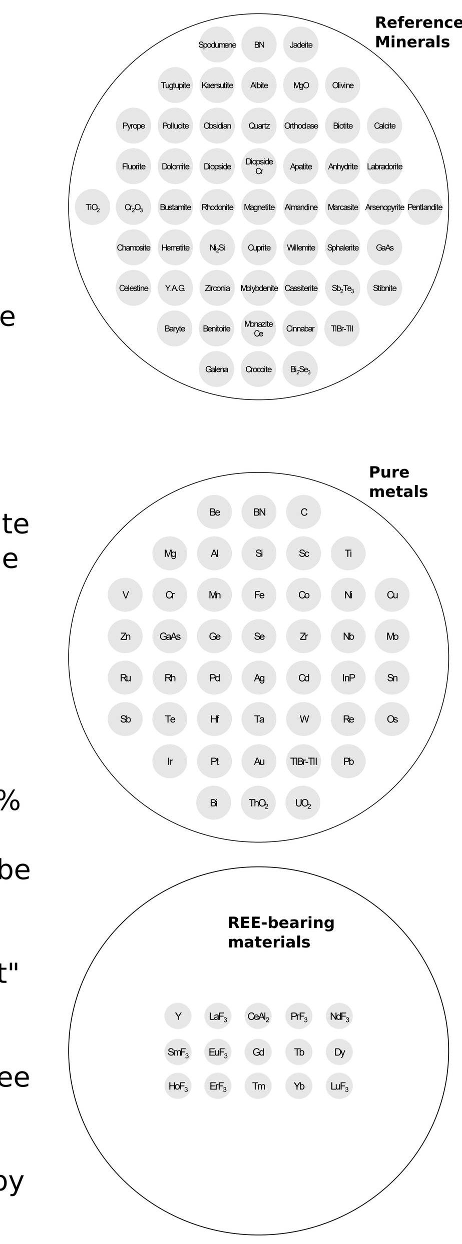


Diffraction crystals

Crystal		Used to analyze	2d, Å
LiF	Lithium Fluoride	Ti → Br	4.026
PET	Pentaerythritol	S → Ti	8.742
TAP	Thallium Acid Phthalate	F → P	25.9
LDE1	*Layered dispersion element*	N, O, F	60
LDE2	*(not technically a crystal)*	B, C, N	100



Standards-based quantitative analysis



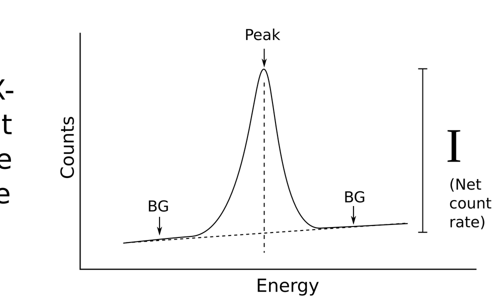
The electron microprobe lab has a suite of over 100 reference materials used for method development, calibration, and quality control. While standardless analysis is a common practice used for quantitative EDS analysis, a major drawback of this approach is that elemental concentrations must be normalized to 100 wt%. This masks errors associated with "missing" elements that were not quantified and shape effects associated with variations in the count rate of X-rays due to irregularities in the surface of the analyte material.

When standards-based analysis is used, the concentration of each element is calculated independently. The concentrations of the elements can then be summed to yield an analytical total, which should be close to 100 wt% (98.5-101.0 wt%). If the analytical total is unsatisfactory, then the source of this error can be determined by further investigation.

Our electron microprobe is equipped with a "split" sample holder, in which the bottom 9 x 6 cm portion of the holder holds samples being analyzed, and the top 6 x 3 cm portion holds three 1-inch diameter blocks of reference materials (shown to the right) which are never removed from the sample chamber and can be analyzed by the user at any time.

Quantification procedure

WDS quantitative analysis is done by counting X-rays on- and off-peak to determine the net count rate (i.e. above background). The net count rate (I) of the standard is initially quantified using the ratio of the unknown (unk) net count rate compared to that of a standard with a known concentration [C (std)]. The concentrations of each elements are then iteratively corrected for matrix effects (e.g. ZAF correction)



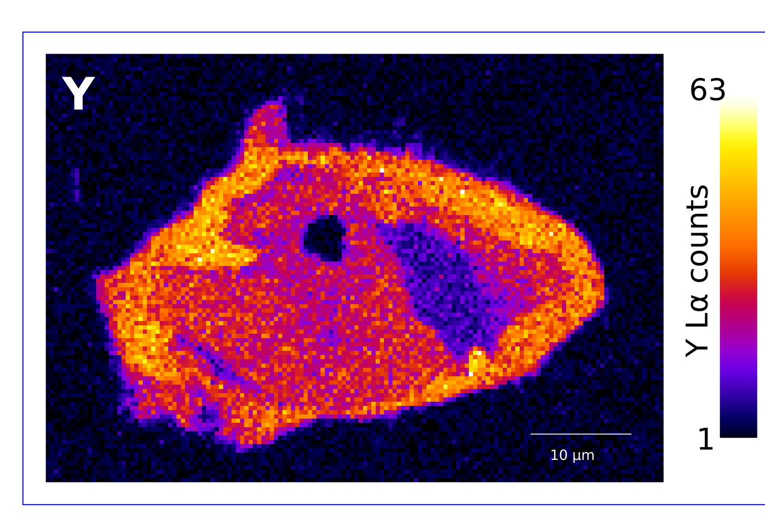
C (unk) = C (std) x I (unk) / I (std)

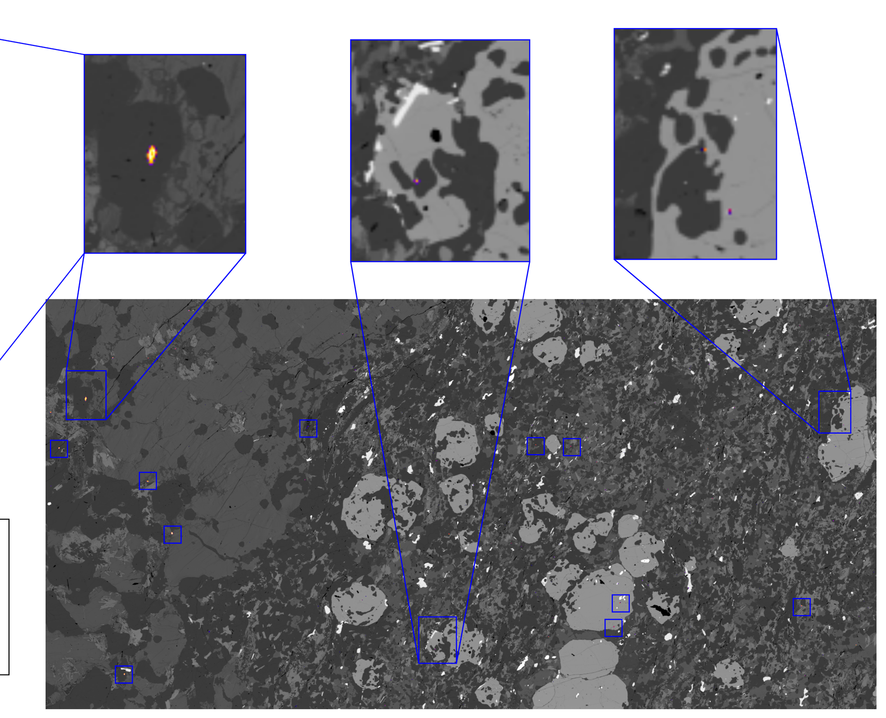
Elemental mapping

The microprobe collects element maps by scanning samples with the electron beam and simultaneously counting X-rays using the EDS and/or WDS detectors. The sample is scanned in one of two modes:

1) in the "**beam scanning**" mode, the sample is stationary while the beam is deflected to scan over a rectangular area. This mapping mode is appropriate for small areas (FOV up to ~50 microns wide for WDS maps or ~1mm for EDS maps).

2) in the "**stage scanning**" mode, the beam is stationary, and the sample is moved back and forth under the beam. This mapping mode is appropriate for large areas, which can be as large as the entire sample (up to approximately 6 cm x 9 cm).

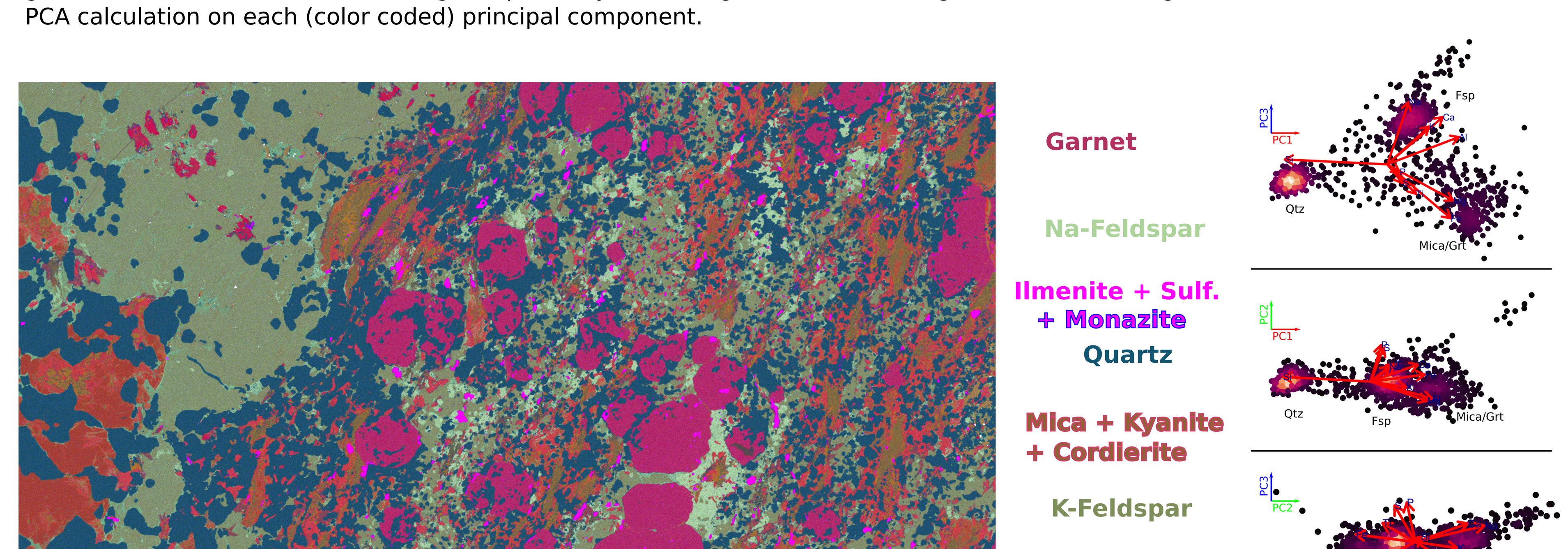




Above: X-ray map of Y in monazite obtained using beam mapping.
Right: Stage map of a metamorphic rock containing monazite accessory mineral grains; grayscale = BED image; blue boxes = monazite grain locations identified using Ce X-ray map.

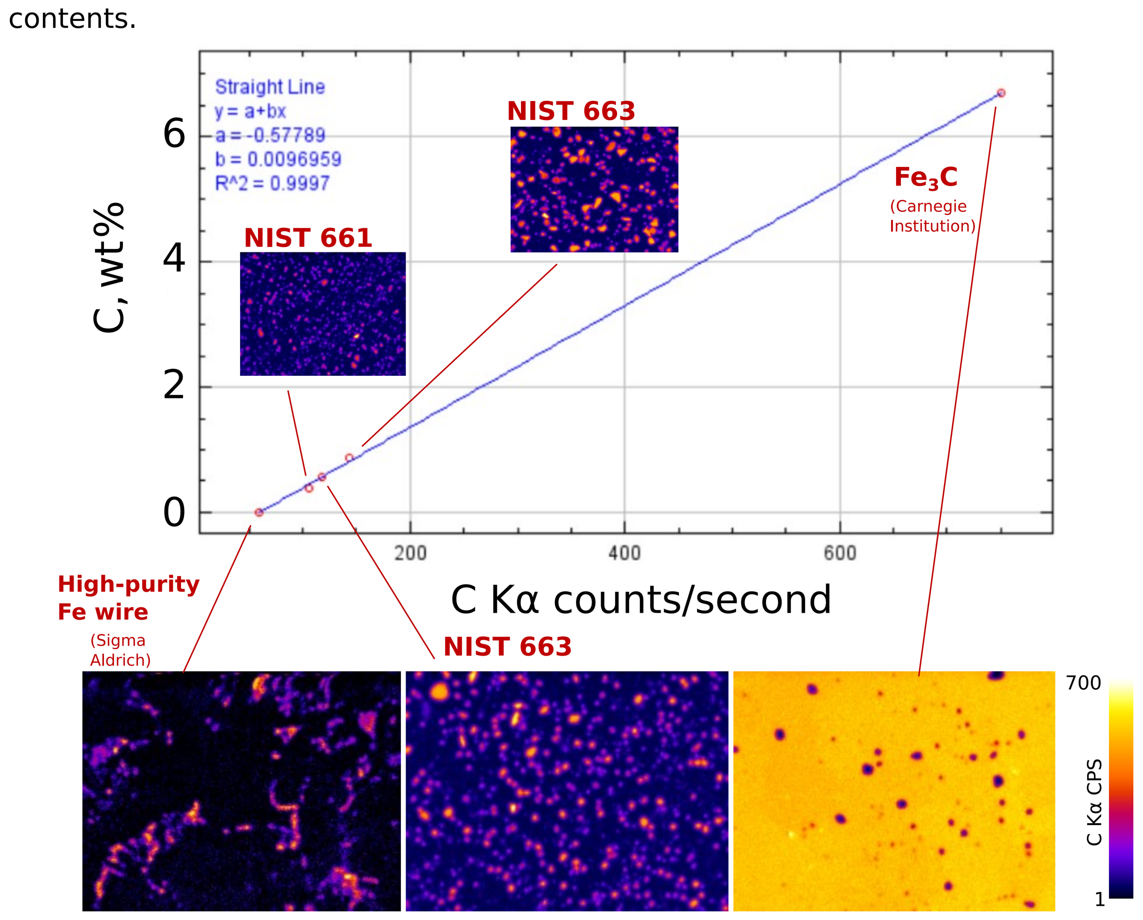
Sometimes finding accessory phases in a large sample can be like "trying to find a needle in a haystack." Stage scanning is an effective strategy for locating accessory phases when they can be identified based on their elemental composition. For example, geologists use the mineral Monazite, an accessory REE-phosphate mineral present in many rock types, to determine the ages of igneous and metamorphic rocks using radiometric dating. The images above show a standard-sized petrographic thin section (a 27 mm x 46 mm polished slice of rock) which has been mapped to locate areas with high concentrations of Ce, which is present as a major element (>10 wt%) in monazite. The right image is a composite grayscale backscattered electron (BSE) image with a colored overlay indicating the Ce count rate (i.e. yellow/orange pixels = monazite).

Element maps are also an effective tool for determining which phases are present in a chemically heterogeneous sample. For example, the images below show how principal component analysis (PCA) may be used to identify different mineral phases present in a metamorphic rock. The left image is a composite map of principle components 1, 2, and 3, which are displayed using the red, green, and blue channels of the image respectively. The images shown to the right show the loading of the elements used for the PCA calculation on each (color coded) principal component.



Light element analysis

The electron microprobe can be used for light element analysis, such as quantifying C and mapping its distribution in steel. Two of the WDS detectors on the electron microprobe are equipped with LDE2 crystals, which have a large interplanar spacing and can detect elements as light as B. The images below illustrate the empirical process for determining the C content of steel using reference materials with known C contents.



Contact

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