



## How can we characterise graphite via electron microscopy?

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Graphite is one of the most electrically-conductive and mechanically weak minerals commonly encountered in crustal rocks, so its presence affects geophysical properties and rheology. It also behaves as a buffer for oxidation reactions, can record peak temperature through crystallinity if undeformed, has been encountered as a minor 'pinning' phase affecting grain size, and can impact the ability of dissolved species to permeate along grain boundaries.

However, because our typical rock sample preparation methods involve use of resins containing carbon, and because we typically coat samples with carbon to ensure conductivity in electron beam instruments, there have been comparatively few attempts to map and describe its distribution in metamorphic rocks.

We have attempted to characterize graphite using a wide variety of sample preparation and analytical methods, and have found:

(i) Specimens for scanning electron microscopy (SEM), electron microprobe (EMP), and transmission electron microscopy (TEM) analyses should be prepared as polished billets rather than thin sections without use of carbon-containing compounds. Analyses should target freshly polished surfaces where there would be no residual/smeared epoxy/plastic.

(ii) Mechanical polishing, even with colloidal silica, does not yield a sample surface suitably crystalline to be analysed using EBSD but broad ion beam (BIB)-polishing does.

(iii) Element maps made using a TEM as well as an EMP with wavelength and energy dispersive spectroscopy (WDS and EDS) detectors optimized for light elements, and soft X-ray detectors (SXES) can reliably demonstrate the presence of C even if it is disseminated on grain boundaries in layers only tens of nm thick. If SXES is to be employed, we found it useful to first map a wider variety of elements as well as using a large area WDS pseudocrystal optimized for light elements (e.g. the JEOL LDE6L) to narrow down the target regions and then acquiring SXES data from the same areas. This is because SXES map acquisition is currently several times slower than WDS in stage mapping mode and still slightly slower using beam scanning. High vacuum Ir-coating is preferred; a thin 1 nm coat does the job well and allows BSE imaging with only slight extraneous peaks in the spectra. Field emission source X-ray mapping at lower kV (e.g. 10 kV and lower) and lower current (e.g. 10 nA) reduces the C Ka X-ray range and keeps the electron beam compact.

(iv) The crystalline and molecular-scale structure of carbon can be characterized using electron loss spectroscopy (EELS) on a TEM.

(v) Raman spectroscopy provides a good alternative to electron beam methods to map C at grain scale.

These methods have allowed us to demonstrate that carbon fills quartz triple junctions and lies along grain and phase boundaries in <50nm thick layers in quartz-feldspar mixtures typically encountered in Alpine Fault Zone mylonites.