Electron backscatter diffraction (EBSD) together with energy dispersive X-ray spectroscopy (EDS) in the scanning electron microscope (SEM) has been used to characterise deformation and interphase relationships in the geological sciences for almost 20 years. However, the analysis of sub-micron scale features in bulk rock samples using EBSD-EDS is very challenging due to limitations in the spatial resolution of the two techniques.

The recent emergence of transmission Kikuchi diffraction (TKD) in the SEM enables characterisation of nanostructured materials and materials with high intragranular dislocation densities [1, 2]. To date, most published TKD analyses have been on metallic samples and have not included simultaneous EDS measurements. Here we demonstrate for the first time the application of combined TKD and EDS to characterise sub-micron scale features in two contrasting rock samples.

The first sample is a focused ion beam (FIB) lift-out section from the Allende meteorite. The section was taken from a region containing Pt group element (PGE)-enriched metal grains. The combined TKD-EDS analysis identified 6 phases including 3 Fe-Ni sulphides, olivine, chromite and the PGE-enriched Fe-Ni. The phase distribution, as indexed using TKD, is shown in fig. 1; note how the high spatial resolution of TKD allows identification of grains as small as 100 nm in diameter. Simultaneous EDS spectra were collected, allowing the generation of individual element maps for the whole section. The EDS spatial resolution is in the range of 25-50 nm and is far superior to that of EDS on bulk samples. A combined element map of the full section is shown in fig. 2.

A second sample is a FIB section from a polycrystalline diamond aggregate taken from the Orapa Kimberlite in Botswana. This particular section contains 2 inclusions of low-Ni pyrrhotite within a single diamond grain. Combined TKD-EDS analyses were carried out at varying magnifications in order to understand the temporal relationship between the diamond and the inclusions. Fig. 3 shows a combined element map of the whole section, showing an iron oxide rim around the pyrrhotite, as well as small Cu-rich domains within the largest inclusion. A higher magnification TKD analysis of part of the main inclusion was carried out, and this identifies the Fe-oxide as magnetite, and confirms the presence of chalcopyrite within the pyrrhotite.

We will discuss the emergence of the integrated TKD-EDS approach and the implication this has on the characterisation and interpretation of sub-micron scale structures in these and other complex geological and meteoritic samples.

References:
Fig. 1: TKD phase map of a FIB section from the Allende meteorite.

Fig. 2: Combined element EDS map from the same area shown in fig. 1

Fig. 3: TKD-EDS results from a sulphide inclusion in diamond from a kimberlite sample FIB section. The left hand image shows a combined element map of the whole FIB section, with the white box marking an area highlighted in the higher resolution TKD phase map on the right.