Orientation Information measured by Soft X-ray Spectroscopy Mapping

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A FEG-EPMA equipped with wavelength and energy dispersive spectrometry, together with soft x-ray emission spectroscopy (SXES) and cathodoluminescence (CL) spectrometery has been used to investigate a geological sample from a Tanzanian deposit containing graphite, quartz and a number of other minerals. One of the aims of this study was to understand the crystallinity of the graphite, as the geology indicated the ore has been subjected to a heating event up to 800°C. At these conditions the carbon should be fully crystalline graphite. The SXE spectrometer equipped with a Princeton CCD [1, 2] and was used to investigate whether different graphite or carbonaceous forms were present while the CL together with trace Ti analysis, was used to determine the maximum heating temperature using the Ti in quartz geothermometry [3]. The sample was mounted in a 25mm round, polished and the surface was finished by ion beam milling at 2kV, 5°, for 10 minutes using a Technoorg Linda model SEM Prep2.

For the SXE investigation the mapping was performed at 10kV, 70nA, 600ms dwell with the SXE spectrometer utilising a 200N grating that was configured to show from 1st through to 4th order C Ka reflections. In Fig. 1 we show an elemental map with a central graphite grain within a mineral assemblage. C Ka spectra from across a number of carbon rich grains clearly showed the graphite structure compared to previous spectra [4, 5]. The second order reflection of C Ka was found to be a good compromise between resolution and sensitivity. By selecting several energy regions across the second order reflection of C Ka and projecting these across the mapped area, Fig. 2, the grains are seen to be composed of graphite with different orientations. Using a scatter plot of the energy regions 135-138 vs. 140-141eV and selecting the end-member clusters the C Ka spectra are shown in Fig 3. The C Ka is generated by an electron transition from a 2*p* orbital to an unfilled 1s orbital and theoretical electronic density of states (DOS) calculations by Srbinovsky et al. [6] have shown that the 1s DOS contains no structure while the 2*p* atomic orbitals do have structure therefore the 2*p* DOS reflect the shape of the C Ka emission spectra. This study showed the graphite has two distinct spectroscopic forms the $2p_{(x, y)}$ - 1s and the $2p_z$ - 1s, Fig. 4. The theoretical spectral shapes are good agreement with C Ka spectra measured using SXE spectrometer. EBSD maps of the graphite grains revealed similar grain shapes to those observed with SXE mapping.

References

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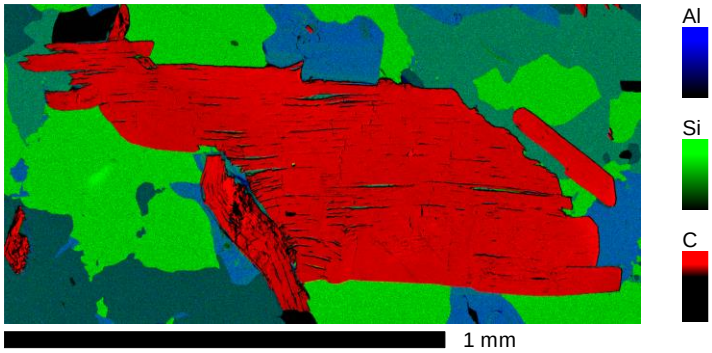

Figure 1. Three element map showing graphite grains within a mineral complex.

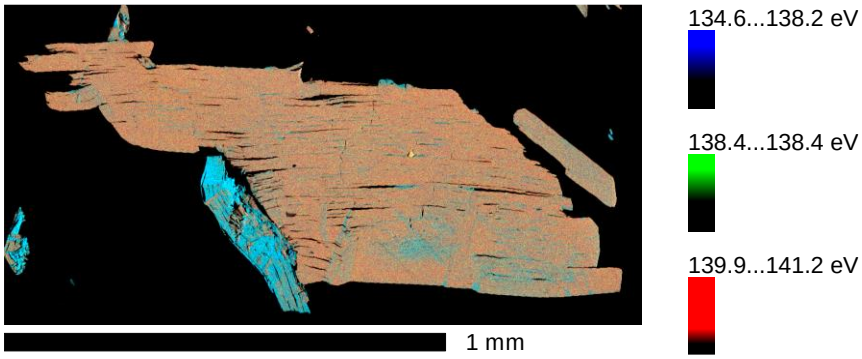

Figure 2. Map showing three ROIs across the 2nd order reflection of C Ka peak revealing different grains within the graphite.

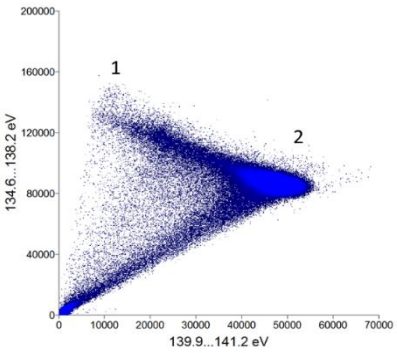

Figure 3. Scatter plot of two C Ka ROIs. The two marked regions associated spectra are given Fig. 4.

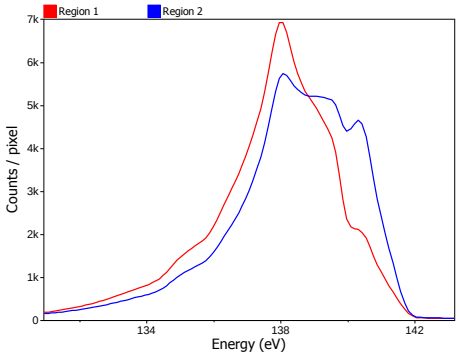

Figure 4. 2nd order C Ka spectra from the end-member clusters. Region 1 is [C Ka 2*p*(x+y)] and Region 2 is [C Ka 2*pz*].