

Using an EPMA to Map Lonsdaleite in Ureilite Meteorites

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Carbon is known to have several allotropes. Of these, the hexagonal diamond structure known as lonsdaleite is the most controversial. First identified in the Canyon Diablo meteor impact site [1] and formed synthetically [2] in 1967, it has since been found in other natural and synthetic samples. However, more recently, it has been claimed not to exist as a discrete material and that prior observations can instead be explained by defective cubic diamond containing twins and stacking faults [3]. We have examined a range of ureilites using EPMA and within these found crystals of lonsdaleite, that we have then confirmed as hexagonal diamond using high resolution imaging and electron diffraction in a Transmission Electron Microscope (TEM) [4].

EPMA mapping was performed on a field-emission gun EPMA (JEOL 8530) with a liquid nitrogen (LN) stage, SXES detector, two SDD EDS detectors (Bruker XFLASH 6010) and a grating cathodoluminescence (CL) system [5]. The LN stage can move while at cryogenic temperature, so we can collect maps in both beam scan and stage scan. It is important to have a stage scanning capability, because in an EPMA the WD, CL and SXES spectrometers are optically aligned to a point and fully focusing. We have developed software to allow collection of the backscatter electron signal, WDS, EDS, SXES and CL spectral data simultaneously. This unique instrument enables us to avoid pixel misalignment and minimising electron beam induced damage artefacts associated with multiple pass mapping, in addition to that of the time saved.

CL is widely used for the study of diamonds with more than 100 centres having been documented, giving rise to luminescence of more than a thousand spectral lines [6], however there is very little published on the luminescence response of lonsdaleite. By mapping samples at liquid nitrogen temperatures, we can improve the resolution of peaks seen in CL spectra, as the cryogenic cooling reduces thermal broadening of CL peaks. Figure 1 shows a map from a region of a ureilite (NWA 7983) that contains both lonsdaleite and diamond. Spectra extracted from diamond and lonsdaleite regions are compared in Fig. 1(b), with the diamond spectrum showing a strong peak N-V⁰ peak at 2.16 eV (575nm), N2V structure at 2.4 eV (517nm) and Band A emission at 2.8 eV (442 nm). The lonsdaleite spectrum shows a sharp peak at 2.36 eV (533 nm) with an associated vibronic structure ($\hbar\omega \sim 0.12$ eV) and a broad peak in the same position as the Band A peak in diamond.

In this same map we collected SXES data, and a comparison of spectra extracted from diamond and lonsdaleite regions is shown in Fig. (c). The 200N grating used in this collection is optimised for carbon reflectivity, and so generates a strong set of higher order reflections. While higher order reflections have less signal intensity, they have the benefit of higher energy resolution, and so Fig. 1 (c) shows the 3rd order reflection of the carbon K α line, which is a good trade-off between signal intensity and increased resolution. A slight narrowing of the carbon K α in the lonsdaleite compared to diamond can be seen, and this is in line with x-ray generated soft X-ray emission by Kurdyumov et al. [7].

A benefit of hyperspectral data collection is that these data sets are amenable to post-hoc examination to find unexpected phases (x-ray data) and defects or centres (CL data) within a sample. There are number of approaches that can be used to examine hyperspectral data sets, and in this work, we have used clustering algorithms to partition and organise the hyperspectral data [8,9].

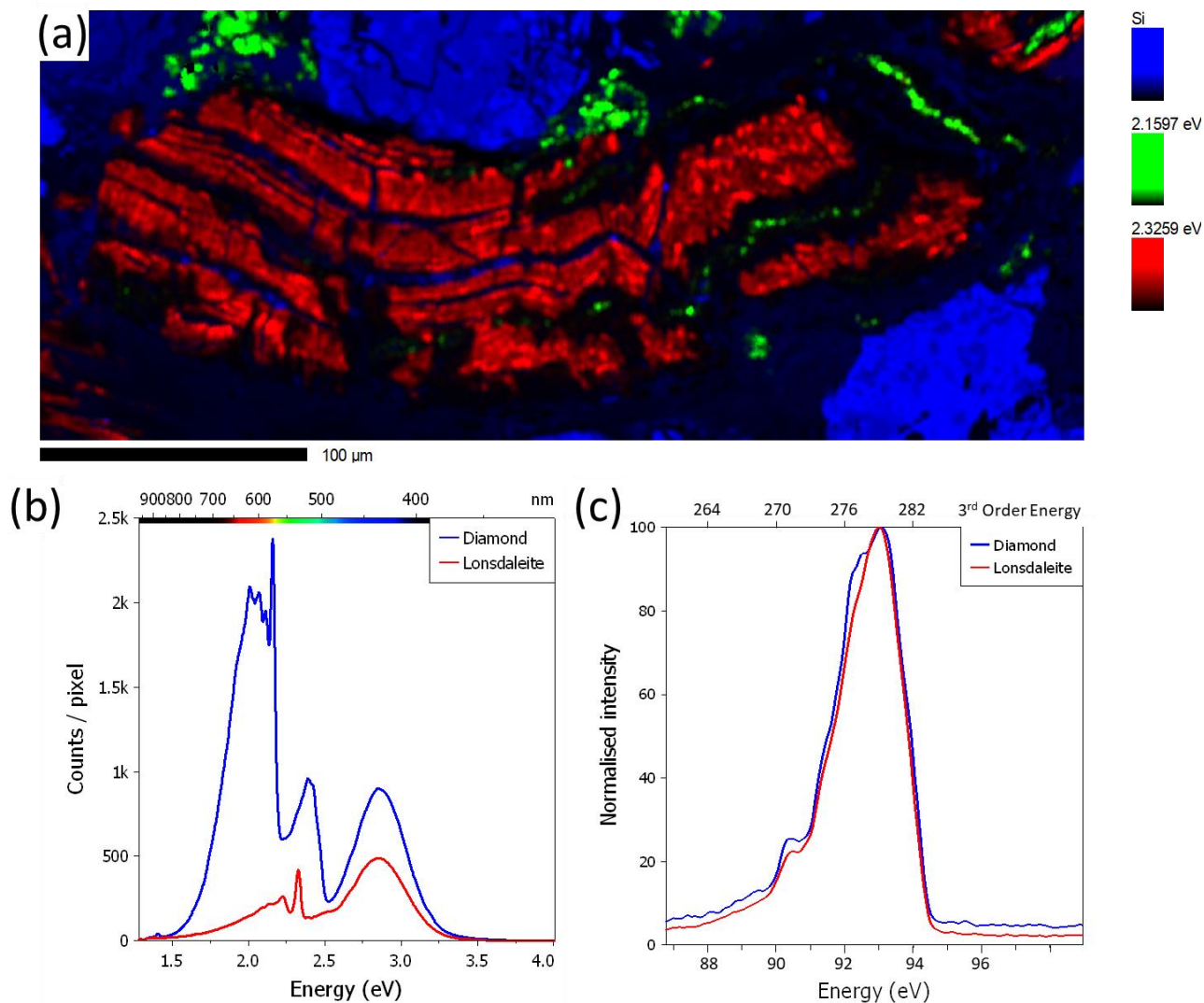


Figure 1. (a) An RGB map collected from ureilite NWA7983 while at Liquid Nitrogen temperature, showing Si in blue, the fitted peak at 2.1597 eV from the N-V⁰ diamond defect in green, and the 2.3259 eV peak fitting the sharp peak in lonsdaleite showing its distribution in red. Spectra have been extracted from diamond and lonsdaleite regions of the map and (b) shows a comparison of CL spectra and (c) shows a comparison of the 3rd order reflection of the carbon K α line collected using the SXES.

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