Experiment Brief

Monarc Cathodoluminescence Detector and Octane Elite EDS System

Title

Streamlined microanalysis in the SEM

Instruments Used

Gatan Microscopy Suite® brings together Gatan's Monarc® detector and EDAX's Octane Elite EDS system to capture cathodoluminescence (CL) and x-ray signals simultaneously.

Background

Energy dispersive x-ray spectroscopy (EDS) and CL in the scanning electron microscope (SEM) are critical microanalysis techniques in analytical sciences. With EDS, an x-ray fluorescence spectrum reveals a sample's composition based on characteristic x-rays arising from an element's atomic structure, whereas CL utilizes optical spectroscopy to reveal optical and electronic properties result from the crystal structure and presence of trace elements or defects. Often, a complete analysis requires characterization using both the EDS and CL techniques. For instance, in many minerals, gems, and cultural heritage items, the CL signal reveals the distribution of specific minor and trace elements at concentrations far below the sensitivity level of EDS.

Materials and Methods

Historically, it has been necessary to collect CL and EDS maps sequentially due to the CL detector blocking the x-ray signal. Furthermore, dissimilar analytical working distances often required the sample to be moved between analysis techniques, which lead to extended data collection times, image registration complexity, and increased electron dose. However, an efficient workflow using the Monarc CL detector and EDS detectors from EDAX enables the EDS, CL, and SEM signals to be captured simultaneously within Gatan Microscopy Suite software. A proprietary collection mirror allows a common analytical position to be used, maintaining a line of sight to the EDS detector and exceptional collection efficiency.

An example dataset was captured from a kyanite (Al_2SiO_5) thin-section. Kyanite was chosen due to its importance as a resource for the refractory industry and the ambiguity over the role that minor, and trace elements play in determining the refractoriness.

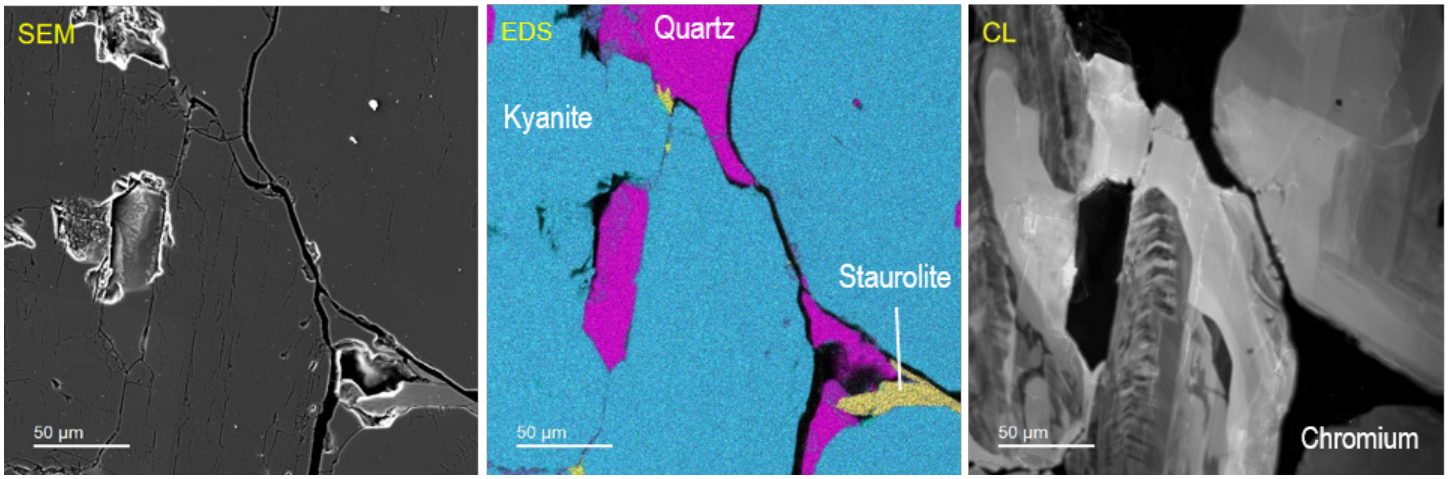

Figure 1. (left) Secondary electron image of kyanite thin-section; (center) EDS map revealing a predominantly coarse-grained quartz-kyanite segregation with small amounts of staurolite; (right) distribution of chromium in the kyanite phase extracted from cathodoluminescence data revealing significant intra-grain segregation indicative of multiple generations of formation—a similar map for titanium was also deduced but is not shown here for clarity.

Summary

By capturing the EDS and CL signals, we were able to collect results that could not be obtained by use of each technique in isolation. We revealed three distinct mineral phases and the distribution of Cr and Ti trace impurities within kyanite. The techniques of CL and EDS clearly complement one another, and the demonstration of simultaneous acquisition presents as a remarkable benefit for specimen analysis.

Credit(s)

A special thanks to Dr. Giulia Degli-Alessandrini of The Open University for providing the specimen used in this study.

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EB-Kyanite-FL1-CA-OCT20