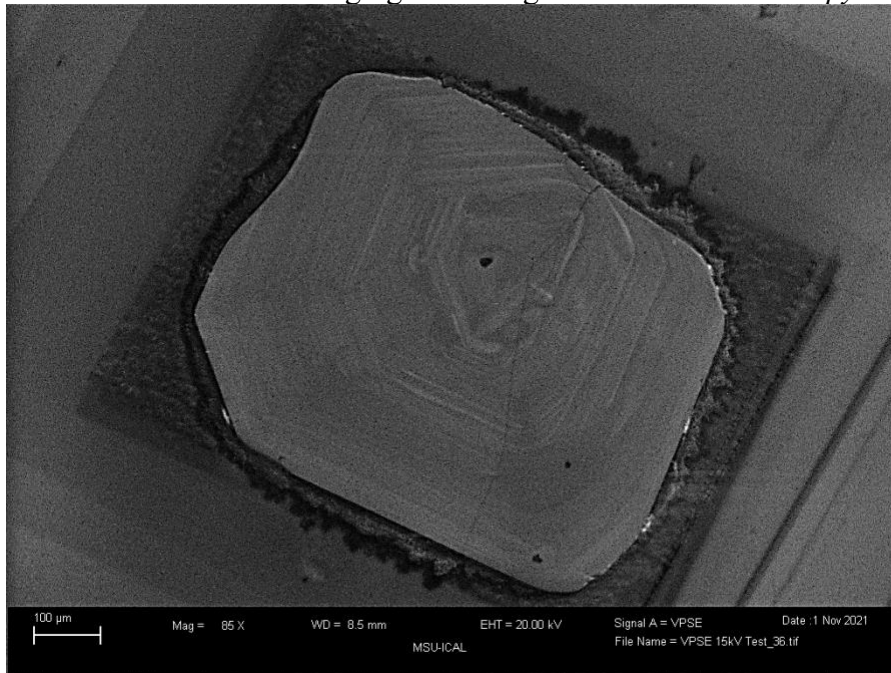


Cathodoluminescence Imaging and Spectroscopy

David Mogk, Assistant Director ICAL, October 13, 2025

Cathodoluminescence (CL) imaging and spectroscopy extends instrumental capabilities beyond traditional SEM and BSE imaging and EDS elemental mapping and analysis. The CL signal derives from excitation of transition metals (with d-orbitals) and Rare Earth Elements (with f-orbitals) under an electron beam with sensitivity down to the ppm level. CL images can be obtained on the Zeiss Supra 55 FESEM using the Variable Pressure Secondary Electron detector (VPSE), and on the Zeiss Ultra 55 FESEM using the dedicated Delmic SPARC CL detector system with spectrometer.

Zeiss Supra 55 VPSE: Panchromatic (gray level) CL images can be obtained on the Zeiss Supra 55 FE-SEM using the variable pressure secondary electron (VPSE) detector. Recommended instrument parameters for obtaining these CL images are 20keV beam voltage, working distance of 7-10 mm, and largest aperture. Relatively large format CL images can be rapidly acquired on a millimeter scale. In addition BSE, EDS, and EBSD data may be acquired in VP mode. The advantages of obtaining CL images in this mode are: very rapid instrumentation set up (simply change from SE2 to VPSE mode), large imaged area format, and ease of integration with SEI and BSE images. For details, see: Williams, T. and Bailey, F., 2006. The Photon Detector and the Variable-Pressure Scanning Electron Microscope an Alternative Method for Cathodoluminescence Imaging of Geologic Materials. *Microscopy and Microanalysis*,

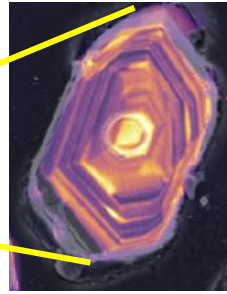
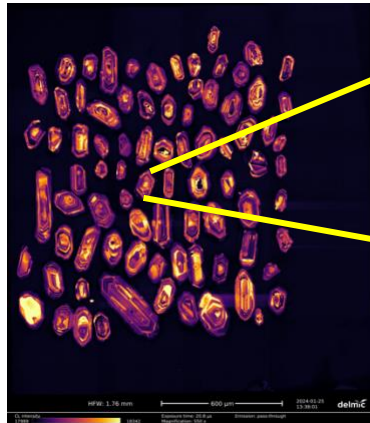


CL image of quartz crystal from Yellowstone volcanics taken in VPSE mode on the Zeiss Supra 55 FESEM.

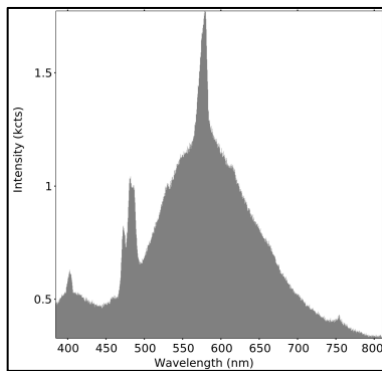
DELMIC SPARC CL Detector. Much more comprehensive CL data can be acquired on the Zeiss Ultra 55 FESEM. This includes the ability to acquire high resolution CL images (sub-micron scale), true color CL images using a series of RGB filters, large area CL images may be acquired by stitching a mosaic of individual high resolution images, and spectroscopic analysis to identify the residence of trace elements and their chemical state. Attributes of the DELMIC system include:

- Micron-scale spatial resolution is available to map very discrete differences in composition of materials.
- The Delmic CL detector on the Zeiss Ultra Field Emission SEM at ICAL can obtain either panchromatic (gray scale) total emission images, or true color images can be obtained obtaining sequential images using RGB filters.
- The spectrophotometer of the Delmic CL instrument can detect luminescence in the range of 400- 900 nm. The spectrometer can be set to obtain optical spectra of areas of interest (spot analysis) to determine what the elemental activator is. Conversely, if an element of interest is known to have emissions in a specific range of wavelengths, the window can be set to create a map of areas where only those wavelengths are observed.
- In addition, the wavelengths of certain elements are sensitive to their chemical state (e.g., Mn^{+2} vs Mn^{+3}) so it is possible to identify and map areas of different microchemistry.
- Flat, polished samples work best for CL imaging, but grain mounts, engineered materials, etc. can also be used.
- Large area CL images can easily be produced by obtaining a series of overlapping CL images and using the Delmic software to stitch the images into a composite file.
- CL is widely used in imaging geologic materials (minerals, rocks, fossils) to demonstrate changing geochemical conditions during crystallization, distinguish discrete generations of crystal growth, demonstrate complex compositional zoning patterns or cross-cutting relations (overgrowths, numerous vein stages), taphonomic history of fossil bones and teeth etc. These differences in composition registered by CL may be entirely transparent to traditional BSE imaging or EDS elemental mapping. CL images are commonly used as guides for follow-on analytical work such as LA-ICPMS analysis of zircons and apatites for radiometric age dating or for trace element analysis at the ppm level).
- CL is also widely used in material science, particularly in studies of semiconductors and nanomaterial characterization (particularly materials doped with REEs). Typical applications are trace element detection, chemical analyses, and defect mapping of materials as part of QA/QC procedures.
- CL is also being used to characterize biological samples that are tagged with luminescent stains or with luminescent nanoparticles.
- For more detailed information about CL, visit https://serc.carleton.edu/research_education/geochemsheets/semcl.html

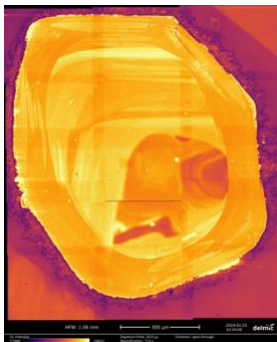
Applications of CL Imaging and Spectroscopy Using Delmic CL



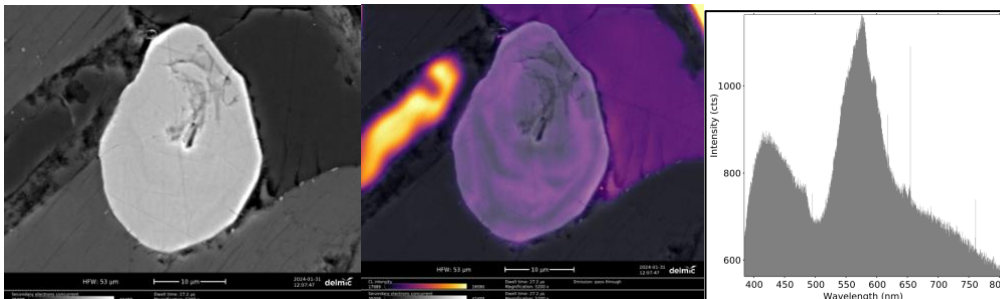
CL image with false color applied; high resolution images were acquired in a 4x5 array and stitched to form this composite image; pixel size is 488 nm; acquisition time was ~15 minutes; Samples from Devon Orme, images by Sara Zacher



Optical spectrum was obtained from the bright central zone; peaks correspond to numerous REE elements



Intricate compositional zoning patterns are evident in this false color CL image of quartz phenocrysts from the Yellowstone Volcanics. Image was obtained by stitching a 3x5 array of CL images. Each image was acquired with a resolution of 1236 pixels, and acquisition time was ~1 minute/frame. CL images may be obtained with a field of view up to ~500 microns. Sample from Madison Myers, image by Sara Zacher



SEM image, CL image and optical spectrum of an Apatite grain

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