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Bringing cathodoluminescence microscopy to your desk

Cathodoluminescence (CL) is the emission of light from a material in response to excitation by incident free electrons. In the last decades CL has become a widely adopted technique in microscopy to study optical and material properties at the nanoscale in a variety of materials and structures in the fields of geology[1], materials science[2], and nanophotonics[3]. A wide range of CL imaging modalities have been developed including spectroscopy, angle, polarization, and time-resolved CL imaging[4,5]. Currently, the majority of CL systems are integrated in large floor-model scanning electron microscopes (SEMs) or even larger transmission electron microscopes. Although such systems can attain very high performance, they are costly, require a large, dedicated laboratory space with special infrastructure and highly trained personnel.

In contrast, desktop SEMs have gained interest due to their ease-of-use, low operating cost, and compact form but until now CL imaging was unavailable as a technique on such systems. With the support from the ERC (POC H2020 grant) we have developed and built a proof-of-concept table-top CL system based on a Phenom XL desktop SEM. In our study we have implemented two approaches for the collection of CL in the vacuum chamber:

1 Light collection through a graded-index lens connected to an optical fiber
2 Light collection through a paraboloid mirror

Approach 1 is a simpler form of CL collection in which no optical alignment is required, but the collection efficiency is lower compared to approach 2. A graphical render of the intended future product design and schematic representations of the different CL collection approaches are shown in Figure 1. The experimental results shown here, were acquired using the first approach. For the second approach experiments are in currently in progress.

To test the capabilities of the table-top CL system we analyzed three sample types. The first sample is a polished zircon ($ZrSiO_4$) crystal embedded in an epoxy plug. Imaging crystal zonation of zircons is a well-established application of CL in geology and is used for radiometric geochronology studies amongst other things[6]. The CL emission was analyzed by a spectrometer which a spectrum is recorded for each scanning pixel (hyperspectral CL imaging). A wavelength averaged map of this data is shown in Figure 2 together with the corresponding backscattering electron (BSE) image which was acquired in parallel. The CL map clearly reveals the internal zonation in the crystal with good contrast. As is common in many zircons the CL contrast is inverted with respect to the BSE contrast[6]. From the same CL data, we also extracted spectra for different regions on the zircon. The emission bands observed in the spectra correspond to radiative intrinsic defect and impurities in the zircon crystal matrix. Next, we studied a YAG:Ce^{3+} powder and a GaN/InGaN semiconductor wafer. Both materials are used in optoelectronic applications such as LEDs. Characteristic CL spectra for these materials are shown in Figure 3. Again, good CL imaging contrast and signal-to-noise ratio in the spectra was achieved.

The results demonstrate that performing CL imaging is possible on table-top SEMs and they are in line with what is needed for many CL applications. As such we see a bright future ahead for desktop CL microscopy and are determined to continue in this direction.

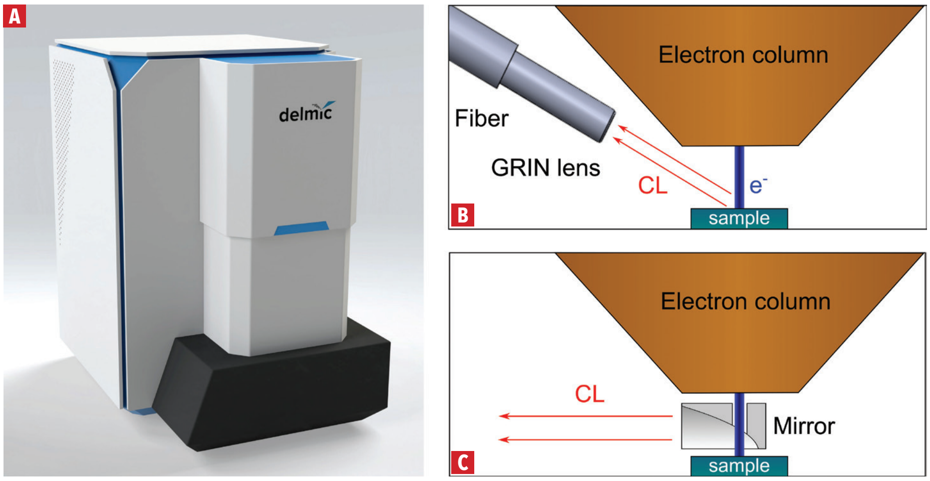

FIGURE 1 *(a) Graphical render of intended table-top CL system product design. (b) Schematic representation of fiber-based CL collection system. (c) Schematic representation of paraboloid mirror CL collection system.*

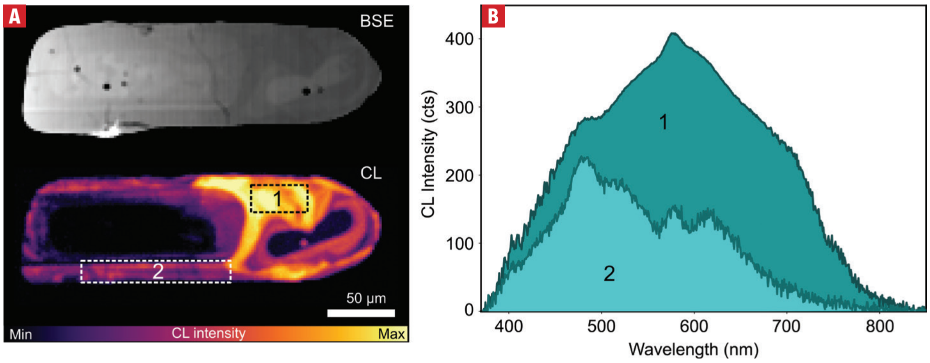

FIGURE 2 *(a) BSE image of a zircon grain (top) and corresponding false-color CL intensity image of the same grain obtained by averaging the hyperspectral CL data in the 400 – 800 nm spectral range (bottom). (b) Spectra obtained from different spatial regions on the zircon crystal as indicated in (a). Data was acquired using 15 kV acceleration voltage and 500 ms dwell time per pixel.*

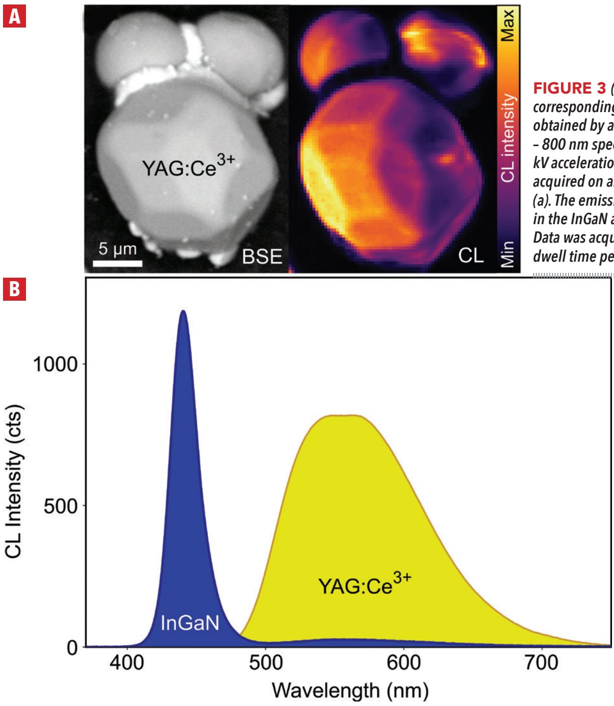

FIGURE 3 *(a) BSE image of a YAG:Ce^{3+} particle (left) and corresponding false-color CL intensity image of the same grain obtained by averaging the hyperspectral CL data in the 400 – 800 nm spectral range (right). Data was acquired using 10 kV acceleration voltage and 200 ms dwell time. (b) CL spectra acquired on an InGaN LED wafer and on the YAG:Ce^{3+} particle in (a). The emission peaks correspond to band-edge recombination in the InGaN and emission from the Ce3+ ions in the YAG:Ce^{3+}. Data was acquired using 10 kV acceleration voltage and 200 ms dwell time per pixel.*

For more information see: delmic.com/cathodoluminescence

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