

Cathodoluminescence Detector for FE-SEM

Authors: X. Liu, M. Kienle, L. Stefan and H. Mathée

Date: February 2010

Introduction

Cathodoluminescence microscopy has become a routine method widely used in material characterization and analysis from semiconductor, nanophotonics, mineralogy and geosciences to biology and life science. To understand the structure composition and properties of luminescent or optical materials, an electron probe beam focused in a tiny spot in a Field Emission Scanning Electron Microscope (FE-SEM) can be applied as a non-destructive excitation source to bombard the sample surface. The generated light from the sample can be collected through a proper cathodoluminescence detector attached to the FE-SEM and electron beam induced cathodoluminescent images can be acquired during raster scanning on the sample.

The electron beam spot of the FE-SEM with a nanometer size is able to excite a cathodoluminescence response from a sample region of about a few hundred nanometers, thus the electron beam induced cathodoluminescence could provide better spatial resolution than optical cathodoluminescence. Not only cathodoluminescent properties but also other information of the sample can be easily

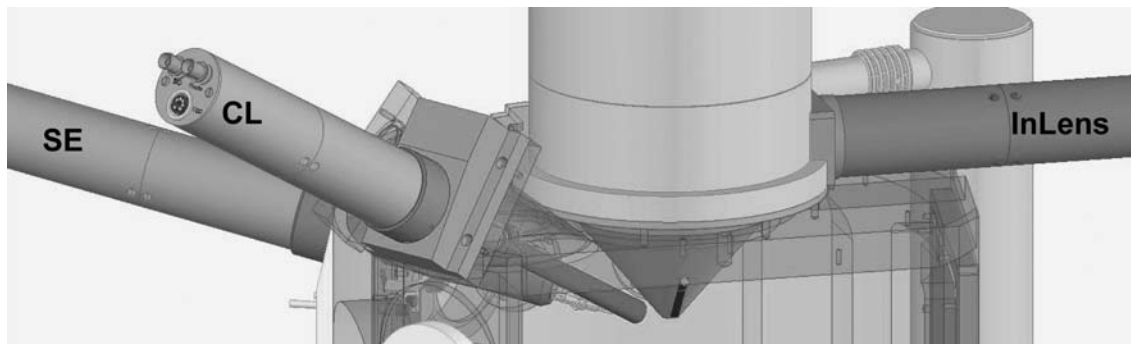
obtained by quickly switching to other detectors (SE, BSE and EDX) in the FE-SEM system.

As reported in many literature not only cathodoluminescence spectroscope and its application are essentially useful for mineralogist and geologists in distinguishing different generation of the same mineral, but also the cathodoluminescence imaging is capable of providing solutions for a number of geological problems such as sedimentological problems, provenance analysis, geochronology and detection of trace elements.

Instrumentation

Carl Zeiss NTS has introduced a new cathodoluminescence (CL) detector for the FE-SEM products which is based on an enhancement of the VPSE (Variable Pressure Secondary Electron) detector. Following Carl Zeiss NTS's philosophy to protect our customer's investments, the CL detector is compatible with the installed base of Carl Zeiss FE-SEM SUPRA®40, SUPRA®55, ULTRA 55 and ULTRA PLUS series. The corresponding SmartSEM® User Interface software requires at least the version 5.04.

Fig. 1: Schematic drawing of the detectors positioning in SEM presenting the Everhart-Thornley SE, in-lens and CL detectors.



We make it visible.

Figure 1 illustrates the detectors positioning on the chamber of a FE-SEM. The CL detector can be selected by a simple and easy switch from the menu of the SmartSEM® User Interface software "Tools / Goto Control Panel / Detectors" as shown in Figure 2.

The imaging condition of the CL detector works under vacuum of about 10^{-6} mbar. The recommended working distance is from 4 mm to 40 mm. Depending on the activator concentration of the specimen, variable beam current from a few pA to 40 nA could be used by rationally selecting different aperture sizes and acceleration energies from 2 kV up to 30 kV.

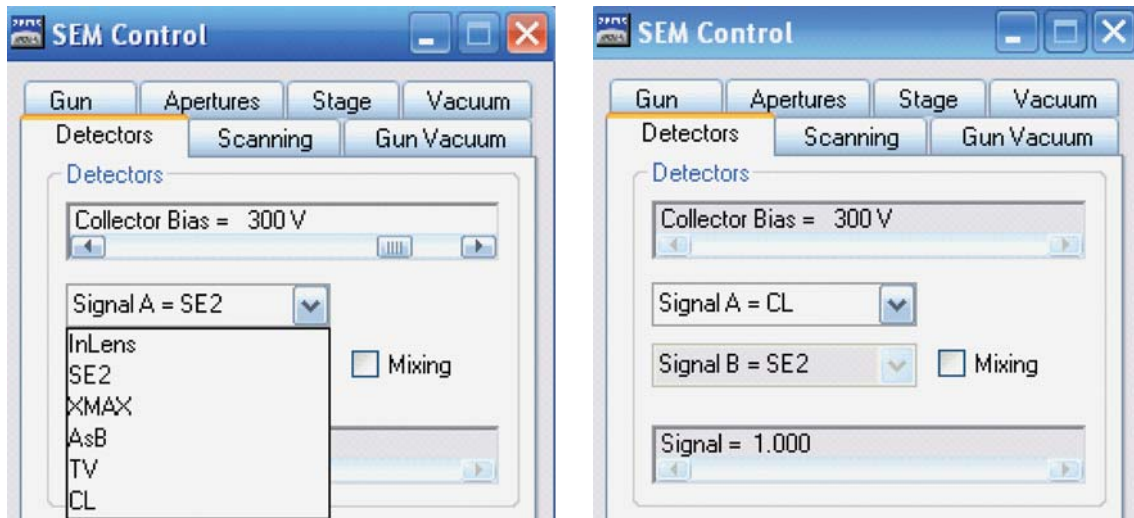


Fig. 2: The SEM control panel for switching detectors option.

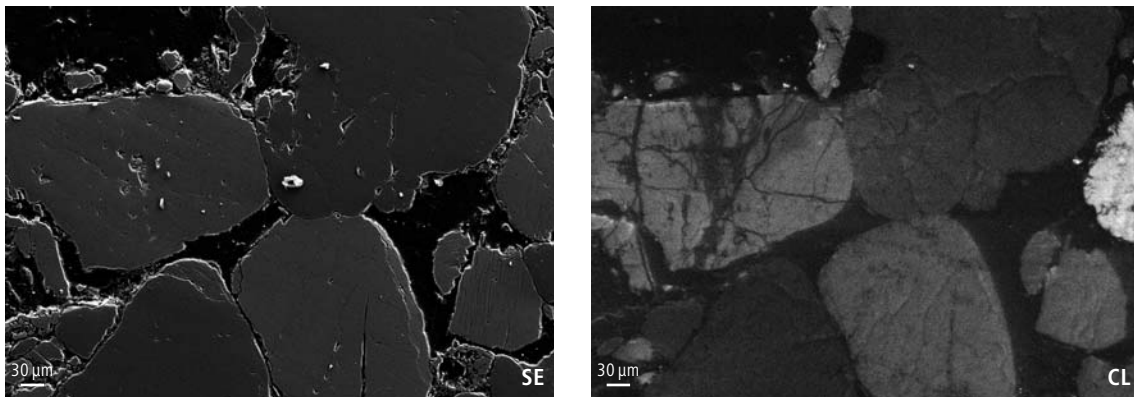


Fig. 3: The secondary electron image from ET detector vs. cathodoluminescent image from CL detector of Feldspathic Sand. The secondary electron image shows the topography structure. Due to the different mineral phases (chemical composition) the CL image presents different contrast intensity between the different grains, whereas such information is unavailable from the SE image. The dark phases are the quartz (SiO_2) and the bright phases are from Calcite (CaCO_3) and K-Feldspar (KAlSi_3O_8). The dark trails of lower luminescence in some bright grains in the CL image are the features of the weathering of the K-Feldspar.

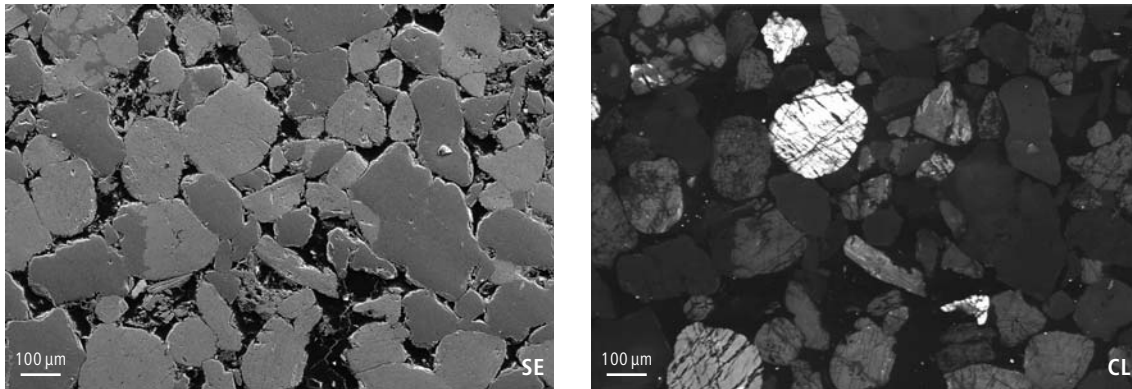


Fig. 4: The secondary electron image from ET detector vs. cathodoluminescent image from CL detector of Sandstone from Elbtal region. The secondary electron image shows the surface structure. The bright phases in the CL image are K-Feldspar (KAlSi_3O_8) and the dark ones are quartz (SiO_2). The dark trails in bright grains reveal the features of weathering of the K-feldspar grains.

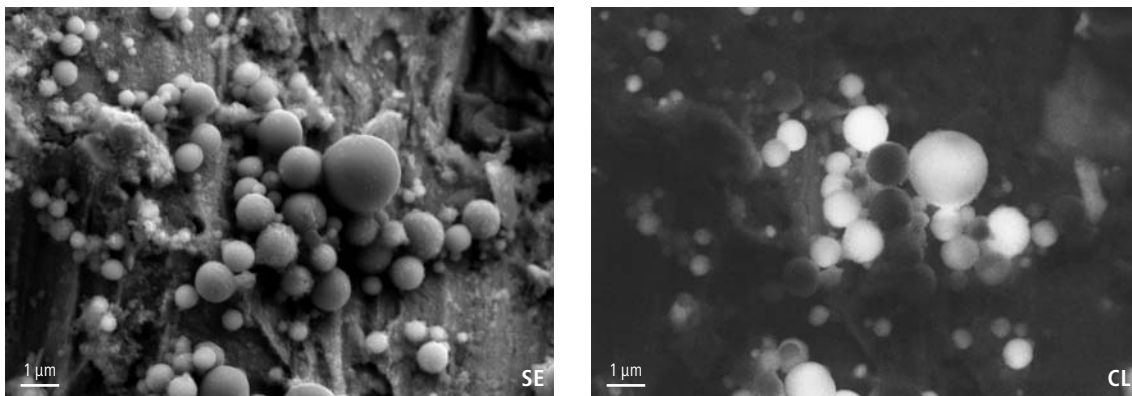


Fig. 5: The secondary electron image from ET detector vs. cathodoluminescent image from CL detector of $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$ (hydroxyapatite) doped with Mn. The small spherical beads on the surface of the hydroxyapatite result from the high temperature plasma-spray process during the sample processing in the SE image. The different contrast intensities of the spherical beads are due to different amounts of doped Mn in CL image since Mn^{2+} is the main activator of the cathodoluminescence in the hydroxyapatite. The more the Mn^{2+} inside the spherical beads, the brighter they are.

Conclusions

The CL detector rapidly provides cathodoluminescence information of from a large area of the specimen. Compare to the secondary electron image, the cathodoluminescence imaging is a powerful method in provenance studies of petrology, in distinguishing mineral phases and in detecting trace activators (Mn^{2+}) and their distribution in biogenic carbonates. It provides useful solutions for ceramic industry, mineral industry, petrochemistry, pharmaceutical industry, biopetrology and Oil gas reservoirs sandstones analysis.

Acknowledgment

We appreciate Prof. J. Götze, TU Bergacademy Freiberg for providing us the samples.

Maximum Information – Maximum Insight

More than 160 years of experience in optics has laid the foundation for pioneering electron and ion beam microscopes from Carl Zeiss. Superior integration of imaging and analytical capabilities provides information beyond resolution, unlocking the best kept secrets of your sample.

With a broad technology portfolio Carl Zeiss provides instruments both tailored to your requirements and adaptable to your evolving needs. With our highly versatile application solutions we endeavor to be your partner of choice.

Superbly equipped, regional demo centers provide you with access to our applications expertise developed in collaboration with world-class partners in industry and academia. Global customer support is provided by the Carl Zeiss Group together with an extensive network of authorized dealers.

Our mission at all times: Maximum Information – Maximum Insight.

Carl Zeiss NTS GmbH

Carl-Zeiss-Str. 56
73447 Oberkochen
Germany
Tel. +49 73 64 / 20 44 88
Fax +49 73 64 / 20 43 43
info@nts.zeiss.com

Carl Zeiss NTS, LLC

One Corporation Way
Peabody, MA 01960
USA
Tel. +1 978 / 826 1500
Fax +1 978 / 532 5696
info-usa@nts.zeiss.com

Carl Zeiss NTS Pte. Ltd.

50 Kaki Bukit Place #04-01
Singapore 415926
Singapore
Tel. +65 65 67 / 30 11
Fax +65 65 67 / 51 31
info.sea@nts.zeiss.com

Carl Zeiss NTS Ltd.

511 Coldhams Lane
Cambridge CB1 3JS
UK
Tel. +44 12 23 41 41 66
Fax +44 12 23 41 27 76
info-uk@nts.zeiss.com

Carl Zeiss NTS S.a.s.

Zone d'Activité des Peupliers
27, rue des Peupliers -
Bâtiment A
92000 Nanterre
France
Tel. +33 1 41 39 92 10
Fax +33 1 41 39 92 29
info-fr@nts.zeiss.com

www.zeiss.com/nts



We make it visible.