



Scanning electron microscopy cathodoluminescence of quartz: Principles, techniques and applications in ore geology



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ABSTRACT

Scanning electron microscopy cathodoluminescence (SEM-CL) of quartz has been a prevalent research technique in porphyry and epithermal systems for the past two decades. Quartz from specific geological environments reveals unique textures in SEM-CL, which can be used to constrain the evolution of these ore-bearing systems when complemented by fluid inclusion, hyperspectral mapping, and trace element studies. We review SEM-CL principles and instrumentation, sample preparation and handling, and experimental conditions of quartz SEM-CL imaging that result in the high quality CL images. The effects of sample polishing, accelerating voltage, beam spot size, working distance, vacuum conditions, image acquisition, and post-processing were examined through experimental trial. For the XL 30 ESEM and the attached Gatan PanaCL detector used, the optimum experimental conditions to obtain high quality panochromatic SEM-CL images of quartz at high vacuum mode for carbon-coated conductive samples are as follows: 15 kV accelerating voltage, relative beam spot size 6 (approximately 500 nm in diameter), HT – 570 V to – 580 V photomultiplier tube (PMT) voltage. Low vacuum mode (with chamber H₂O vapor pressure from 0.1 to 1.0 Torr) working conditions are similar to the conditions at high vacuum mode except the PMT voltage should be reduced to – 550 V to – 560 V. Working distances vary based on the position of user's retractable CL detector. The sample surface should be as close as possible to the CL detector, but a 1 mm clearance between the detector and the sample surface is recommended to prevent detector from possible damage by the sample. Several minutes of beam exposure prior to image acquisition at 320 second scan speeds at 50×–1500× magnifications is recommended to generate the greatest CL emission. Monochromatic CL imaging requires three scans over the same area using red, green, and blue optical filters that can be merged to produce a "true color" image. The red and green filters require stronger PMT voltages to produce sufficient CL emissions by an increase of – 200 V to – 300 V and – 150 V to – 200 V, respectively, from the PMT voltage used for panochromatic imaging. Special attention is given to the challenges associated with imaging hydrothermal quartz veining in ore deposits and the value of CL data as a foundation for geochemical studies. SEM-CL imaging of vein quartz is explored through case studies of the Red Hills Porphyry Cu–Mo Deposit, Texas, USA, and the Ertsberg–Grasberg Cu–Au District, Papua, Indonesia to aid in vein paragenesis. The most common application of quartz SEM-CL in ore geology is to reveal the relative timing of mineral precipitation, mineral dissolution, and inherited structural features. Understanding of temporal relations among these events makes it possible to select specific generations of quartz within a vein for further studies such as the TitaniQ thermometry and fluid inclusion microthermometry in order to establish T–P–X fluctuations throughout the development of a hydrothermal system.

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1. Introduction

When an electron beam bombards a sample in a scanning electron microscope (SEM) or electron probe microanalyzer (EPMA), many signals, such as secondary electron (SE), backscattered electron (BSE),

characteristic X-ray, visible light, etc., are produced. Crookes (1879) first observed the phenomenon of cathodoluminescence (CL), the emission of light by high energy electron bombardment of a mineral, in the 1870s. Since Crookes' observation, CL has been used in numerous petrologic studies of quartz from a variety of geologic environments (Seyedolali et al., 1997), particularly in sedimentary rocks and hydrothermal ore deposits, because CL can illuminate textures which are not observable by other methods (transmitted light, SE, BSE, etc.). Common minerals utilized for CL petrography include quartz, zircon, diamond, corundum, apatite, anhydrite, fluorite, carbonates, etc. (e.g., Götze et al., 2001; Machel and Burton, 1991; Pagel et al., 2000).

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The geologic applications of quartz CL include crystal growth, dissolution, replacement, deformation, and provenance (Boggs et al., 2002; Landtwing and Pettke, 2005; Matter and Ramseyer, 1985; Penniston-Dorland, 2001; Rusk, 2012; Rusk and Reed, 2002; Seyedolali et al., 1997). In sedimentary petrography, Sippel (1968) used CL to determine texture, sediment source, degree of compaction, diagenetic history, ratio of authigenic and detrital minerals, stratigraphy, siliciclastic components, and cementation history in quartz sandstones. Owen and Carozzi (1986) also deduced stratigraphy, siliciclastic components, and cementation history of the Jackfork Sandstone, Arkansas. Studies using CL by Müller et al. (2003a) in conjunction with trace element variations in igneous quartz were used to correlate CL zoning patterns with genetic processes such as magma mixing, velocity of ascent, growth and diffusion rates of the melt, and large scale convection within a magma chamber. Metamorphic petrography by Spear and Wark (2009) also used CL fabrics of plastically deformed quartz for Titanium thermometry, whereas Sprunt (1978) determined that the luminescence color of metamorphic quartz can be correlated with metamorphic grade (red: low temperature, blue: high temperature) in addition to deformation mechanisms by mechanically induced defects.

Early studies of quartz CL examined the color variations using optical-CL by either cold- or hot-cathode systems (Marshall, 1988; Matter and Ramseyer, 1985; Seyedolali et al., 1997; Zinkernagle, 1978). The major disadvantages of this instrumentation include low magnifications and resolution of images partly due to the detectors inability to detect CL emissions outside the visible range of the electromagnetic spectrum. Provenance studies were limited by the efficacy of optical-CL until the advent of attaching a CL detector to an SEM. This SEM-CL technique made more detailed CL imaging possible due to higher resolution and magnification capabilities which greatly improved observations of distinct CL textures. The intensity of SEM-CL emissions is dependent on the geochemical and structural variations within a crystal which makes this technique ideal for examining textural, compositional, and structural information for any given sample.

The application of quartz SEM-CL has been a robust research technique used in hydrothermal ore systems for the past two decades (Götte et al., 2011; Lehmann et al., 2009; Marshall, 1988; Rusk, 2012; Rusk and Reed, 2002; Rusk et al., 2008a,b; Vasyukova et al., 2013a,b). When various quartz generations and their textural variations within a single, potentially ore-bearing vein are recognized by CL, a preliminary interpretation of quartz growth histories related to ore deposition can be constructed. CL patterns can then be used in conjunction with fluid inclusion microthermometry, trace element compositional variation, and isotopic compositional analyses to relate to specific mineralization events (Donovan et al., 2011; Flem et al., 2002; Götte et al., 2011; Landtwing and Pettke, 2005; Leeman et al., 2012; Müller et al., 2003a, b; Rusk, 2012; Rusk et al., 2008a,b). Therefore, it is imperative to capture high quality CL images during the initial research phase of vein quartz for proper follow-up analyses.

The goal of this review is to provide a general outline of the SEM-CL principles and optimal experimental conditions to capture high quality SEM-CL images and to apply the SEM-CL technique to quartz derived from epithermal and porphyry environments. The authors recognize that obtaining quality SEM-CL images is dependent largely on samples, sample preparation and experimental conditions specific to the instrument used. Case studies of the Red Hills porphyry Mo–Cu, Texas, and the Ertsberg–Grasberg porphyry–skarn Cu–Au, Indonesia deposits utilizing these SEM-CL techniques are used to illustrate the application of SEM-CL for ore geology research.

2. Principles and instrumentation of SEM-CL

SEM-CL is a near surface effect, that can be used to reveal complex crystal growth histories and cryptic microtextures of luminescing minerals which are otherwise invisible with optical, SE, and BSE imaging (Marshall, 1988). When an electron beam is scanned across

the polished surface of a sample, the CL signal is captured by a parabolic or elliptical mirror and a digital CL image is produced as visible light is incident upon the surface of the photomultiplier.

According to Marshall (1988), Boggs et al. (2002), Götze (2009), Götze and Freiberg (2012), and Henry (2012), this technique can be explained by the solid state band theory in which a high energy electron beam bombards a material causing the excitation of electrons to move from the low energy valence band toward the higher energy conduction band. Once the electron reaches the conduction band and the transient energy begins to diminish, the electron will travel back to the valence band and return to ground state energy. When returned to ground state energy, a small amount of radiation (light) is released and translated into wavelengths in the visible light (400–700 nm) or ultra violet light (>400 nm) spectrum. This emission of light is captured via a photomultiplier tube and used to construct an image of the samples luminescence (Fig. 1).

The space between the valence and conduction bands that the electron must pass through is known as the band gap. The band gap may contain holes or traps caused by intrinsic or extrinsic defects in the material which may capture the electrons momentarily. Once the electron is able to escape the trap and recombine in the valence band, a photon is emitted and luminescence will also occur (Fig. 1c).

3. Causes and textures of quartz SEM-CL

Quartz derived from plutonic, volcanic, metamorphic, and hydrothermal systems emit unique CL signatures that can aid in identifying different generations of quartz formed in a specific geologic environment. The reader is referred to Rusk (2012) for illustrations on the following quartz SEM-CL texture descriptions.

3.1. Causes of quartz SEM-CL

Luminescence intensity is dependent on the density of intrinsic and extrinsic defects within the band gap. Intrinsic defects are structural imperfections in the quartz crystal due to vacancies within the crystal lattice. The most common types are point defects, translations, radiation damage, shock damage, melt inclusions, and fluid inclusions which reflect the actual structure of the crystal and may ultimately provide information of the physiochemical conditions during mineralization and subsequent post-mineralization events such as deformation or metamorphism. Fig. 2 displays a quartz SEM-CL image taken by Baline (2007) of hydrothermal quartz from the early Kali stockwork of the Ertsberg–Grasberg district where a bright-CL zone is caused by the abundance of fluid inclusions trapped during a specific period of crystal growth. Therefore, the intrinsic defects can reflect the genetic processes of crystal growth and the specific physiochemical conditions following afterwards (Hamers and Drury, 2011; Müller et al., 2003b; van den Kerkhof et al., 2004).

Extrinsic defects include impurities of cations and anions that have been substituted into the crystal structure. These impurities can be cathodoluminescence activators, sensitizers, and quenchers which are mineral specific. Trace element concentrations and their relative proportion to one another in quartz are considered the major proponents that influence the CL response in most luminescent minerals (Götze et al., 2001; Müller et al., 2003b; Pagel et al., 2000). Quartz's high purity only allows a limited number of trace elements such as Ti^{4+} , P^{5+} and Ge^{4+} to substitute for Si^{4+} within the crystal lattice. Trivalent cations such as Al^{3+} , Ga^{3+} , and Fe^{3+} coupled with H^+ , Li^+ , Na^+ and K^+ monovalent cations for charge balance may also substitute for Si^{4+} (Bahadur, 1993; Dennen, 1965; Lehmann, 1975; Müller et al., 2003a; Weil, 1984). Baline (2007) correlated bright-CL growth zones with elevated concentrations of Ti^{4+} and Al^{3+} from Kali stockwork hydrothermal quartz from the Ertsberg–Grasberg district (Fig. 3). As the CL bright and dark zones are largely deposit specific and the relations between CL zonations and abundances of trace elements in quartz are uncertain,

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