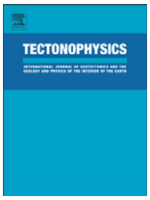
Tectonophysics 690 (2016) 190–205

Contents lists available at ScienceDirect

Tectonophysics

journal homepage: www.elsevier.com/locate/tecto



Syn- and postkinematic cement textures in fractured carbonate rocks: Insights from advanced cathodoluminescence imaging

Estibalitz Ukar *, Stephen E. Laubach

Bureau of Economic Geology, Jackson School of Geosciences, The University of Texas at Austin, University Station Box X, Austin, TX 78713-8924, USA

ARTICLE INFO

Article history:
Received 9 October 2015
Received in revised form 14 April 2016
Accepted 1 May 2016
Available online 4 May 2016

Keywords:
Calcite
Carbonate rocks
Crack-seal texture
Dolomite
Fracture
Porosity
Vein

ABSTRACT

In calcite and dolomite deposits in fractures, transmitted light and optical cathodoluminescence methods detect crack-seal texture in some fractures, but scanning electron microscope-based cathodoluminescence (SEM-CL) combined with secondary-electron images and element maps, reveals crack-seal and cement growth textures where previous SEM-CL imaging methods found massive or featureless deposits. In a range of fractured carbonate rocks, patterns and textures of calcite and dolomite cements precipitated during and after fracture growth resemble complex accumulation patterns found in quartz in sandstone fractures, suggesting that some apparent differences between carbonate mineral and quartz deposits in fractures reflect the limits of previous imaging methods. Advances in delineating textures in widespread carbonate mineral deposits in fractures provide evidence for growth and occlusion of fracture porosity.

1. Introduction

Fracture-filling cements in fractured and faulted upper crustal rocks provide information on the elemental and isotopic chemistry of formation fluids, and fluid pressure and temperature (e.g., Jensenius and Burruss, 1990; Evans and Battles, 1999; Lee and Morse, 1999; Budai et al., 2002; Blyth et al., 2004, 2009; Drake et al., 2009; Holland and Urai, 2010; Cobbold et al., 2013; Fall et al., 2015). Cement textures can record evidence of cycles of fracture opening (Hulin, 1929; Ferguson and Gannett, 1932; Durney and Ramsay, 1973; Ramsay, 1980; Cox and Etheridge, 1983; Laubach, 1988; Laubach et al., 2004a, 2004b; Nüchter and Stöckhert, 2007; Bons et al., 2012), so fracture cement deposits potentially record time sequences of fluid properties (Becker et al., 2010; Evans, 2010; Fall et al., 2015). In sedimentary rocks, deciphering sequences of geochemical and mechanical processes during and after fracture opening is essential to understand evolution of fracture patterns, preservation and destruction of fracture porosity during and after deformation, and fracture permeability (e.g., Hood et al., 2003; Laubach, 2003; Olson et al., 2009).

Textures that are poorly resolved by some imaging methods are a challenge for unraveling fluid histories from cement deposits in fractures. Cathodoluminescence (CL) reveals subtle differences in trace element composition and crystal structure that can be used to delineate textures that are not readily visible using other imaging methods (e.g., Pagel et al., 2000; Boggs and Krinsley, 2006). As an imaging method CL is well suited to reveal brittle structure in both unmetamorphosed and metamorphosed rocks (e.g., Sprunt and Nur, 1979; McLimans, 1991; Dawson et al., 1994; Trave et al., 1998; Milliken and Laubach, 2000; Hood et al., 2003; Blyth et al., 2004; Rimsa et al., 2007; Holland and Urai, 2010; Vandeginste et al., 2013; Lavenu et al., 2013; Kotta, 2014).

In quartz-rich deposits, scanning electron microscope-based cathodoluminescence (SEM-CL), which generates high resolution, high magnification images, has been effective for imaging structurally significant quartz cement textures (e.g., Sprunt and Nur, 1979; Knipe and Lloyd, 1994; Laubach, 1997; Parris et al., 2003; Laubach et al., 2004a, 2004b, 2004c; Hanks et al., 2006; Laubach and Ward, 2006; Hooker et al., 2009; Becker et al., 2010; Fall et al., 2012, 2015) including crack-seal textures, crystal growth textures, crosscutting relationships, and microfractures.

In contrast, in calcite, dolomite and other carbonate mineral deposits, SEM-CL has generally not revealed the same level of textural detail evident in quartz SEM-CL images. Many calcite and dolomite deposits appear—and may be—massive and featureless. But even where optical evidence shows that crack-seal and other structural significant textures *are* present in calcite deposits, SEM-CL images are rarely sufficiently resolved to unravel fracture histories to the same extent

---

* Corresponding author.
E-mail address: esti.ukar@beg.utexas.edu (E. Ukar).

http://dx.doi.org/10.1016/j.tecto.2016.05.001
0040-1951/

as can now be done for quartz deposits. This apparent imaging limitation impedes structural interpretation of fractures containing calcite and dolomite/ankerite, which are the most common phases in fractured limestones and dolostones as well as being widespread in fractures in sandstones and shales.

CL has long been used in carbonate rock diagenesis studies (Marshall, 1988; Richter et al., 2003) including delineation of fractures (e.g., Montanez, 1994; Marquez and Mountjoy, 1996; Gale et al., 2004, 2010). The intrinsic blue CL of calcite is transient, but luminescence in longer-wavelength emissions in calcite and dolomite takes a relatively long time to decay (up to 1000+ ms) following cessation of excitation by the electron beam. This phenomenon, termed phosphorescence, produces an orange-to-red smearing on the image as the electron beam is rastered over the area of interest (Reed and Milliken, 2003; Lee et al., 2005) because these longer wavelengths are persistent. For this reason, and owing to greater convenience and accessibility, most imaging of carbonate cements in fractures has been done using optical-CL (e.g., McLimans, 1991; Trave et al., 1998; Hood et al., 2003; Rimsa et al., 2007; Holland and Urai, 2010; Vandeginste et al., 2013; Lavenu et al., 2013; Kotta, 2014). The spatial resolution, magnification, and sensitivity—for direct observation—of optical-CL are, however, much lower than that of SEM-CL.

In this paper we document fracture cement textures in mostly fibrous/tabular cement deposits in a suite of fractured carbonate rocks using a state-of-the-art SEM and MonoCL 4 detector system and a combination of SEM-CL, secondary-electron images, and element maps. Samples were selected based on coverage of a range of rock types and sample depths for sedimentary rocks. Fractures also have similar macroscopic patterns of cement deposits to those found in other sedimentary rock fractures (Laubach, 2003; Gale et al., 2014). Sampling is therefore representative rather than comprehensive. We show that with minimal phosphorescence, some apparently massive and featureless fracture calcite deposits contain crack-seal and cement growth textures. A key result is the discovery of calcite cement deposits that have bridge-like shapes and crack-seal texture that resembles textures in quartz in more thoroughly studied sandstone fractures, suggesting that some of the apparent differences between carbonate mineral and quartz deposits in fractures is an artifact of limitations in imaging.

2. Sample suite

Petrographic, SEM-CL and elemental analysis (EDS) are used to document cement textures from ten carbonate rock units (Fig. 1, Table 1). Rock types include wackestone, packstone, silty limestone, dolomitic mudstone, dolomitic packstone, dolostone, and calcareous shale. Samples are from core and outcrop. The opening-mode fractures are oriented at a high angle to bedding, in beds having dips that range from nearly horizontal to as much as 30°. Possible loading paths that created these fractures include lowered effective stress caused by some combination of burial loading, regional stretching, thermoelastic contraction, and elevated fluid pressure including gas and/or liquid generation and migration.

3. Advanced CL imaging methods

3.1. Sample preparation

CL image quality varies with sample coating and polish. Several sample preparation techniques have been tested for optimizing CL imaging of carbonate minerals. We tried three types of conductive coating: a 20–25 mm-thick carbon coat, a 5 nm-thick iridium coat, and a 10 nm-thick iridium coat. Due to relatively large sample currents generated or contamination from samples containing hydrocarbons, carbon-coated samples may bubble during acquisition and artifacts are introduced into CL images. In contrast, iridium-coated samples are more stable. If no bubbling occurs, quality of CL images is similar with both types of conductive coatings. Thinner iridium coats correspond with sharper images.

A tradeoff of working at low kV as described below is that because the generation volume of photons is reduced, surface features such as scratches on standard polished thin sections may become visible. Such surface artifacts can be avoided by ion-milling. Iridium coatings tend to bring out surface scratches, so unless the sample is well polished or ion-milled we found that carbon-coated samples give better images. Samples having problematic surface artifacts were ion milled with a Leica TIC 020 triple cross-sectional ion beam for 10 h. The images presented here are mostly from carbon-coated standard polished thin sections, unless otherwise stated in the figure caption.

3.2. Optical and CL methods

Excitation of samples by high-energy electrons produces photon emission in the visible range, or cathodoluminescence (CL). Intrinsic or primary luminescence has been ascribed to natural defects in mineral crystal lattices (vacancies, dislocations) whereas brighter luminescence at higher wavelengths results from the presence and concentration of trace element and rare-earth element activators (Götze et al., 2001, 2004; Boggs and Krinsley, 2006; Götze, 2012; Edwards and Lee, 2014).

Optical-CL microscopes are microscopes with a CL detector that allows a specimen to be bombarded with high-energy electrons from a cathode gun (also called cold-cathode CL) to produce a color CL emission at visible wavelengths (Lee et al., 2005; Boggs and Krinsley, 2006; Götze and Kempe, 2008). The integration of light over time of exposure or digital acquisition required by optical-CL works well for phosphorescent minerals. However, SEM-CL has a number of advantages over optical-CL, including higher spatial resolution and magnification, higher sensitivity (lower-intensity emissions are detectable), and ability to image wavelengths outside of the visible range, such as ultraviolet and infrared emissions. In principle, with sensitive infrared and ultraviolet cameras, some of these emissions could be detected with microscope-based systems. In addition, secondary electron (SE), backscattered electron (BSE) images, and X-ray maps (EDS) can be simultaneously or sequentially obtained for the same area as SEM-CL images, and properties revealed by the different emissions can be correlated.

Lee (2000) showed that increasing the dwell-time of the electron beam at each point in the raster—so that the luminescence contribution from each point to net signal is minimal—reduces phosphorescence and improves panchromatic images formed using all light emitted. However, required dwell times of 1200 ms increases acquisition time for a high-resolution image to several tens of minutes. Another method is to extend the inter-pixel delay, allowing decay of CL from one pixel before the next pixel is collected, but this also increases acquisition time. Reed and Milliken (2003) reduced phosphorescence by using a filter to excise the long wavelength orange-to-red CL emissions characteristic of carbonate minerals. They produced sharp SEM-CL images solely using ultraviolet (UV) to blue wavelengths, termed limited-wavelength images. Because short-wavelength luminescence decays more rapidly than longer wavelengths, limited-wavelength images can be acquired using shorter dwell times than those suggested by Lee (2000). However, using hyperspectral mapping, Lee et al. (2005) demonstrated that there is a nonlinear relationship between luminescence intensity variations at UV-blue wavelengths and intensity variations at orange wavelengths, indicating that short-wavelength emission is an imperfect proxy for longer wavelengths. Thus, images of features such as CL zoning formed from UV-blue emissions only may not capture the signal found in panchromatic images (Lee et al., 2005; Boggs and Krinsley, 2006).

Besides phosphorescence, another problem in SEM-CL imaging of carbonate minerals is lack of luminescence. Almost all CL in carbonates is caused by trace elements, including rare earth elements (REE), which can act as activators, sensitizers, and quenchers (Fairchild, 1983; Richter et al., 2003). The UV-blue luminescence of calcite is believed to be