



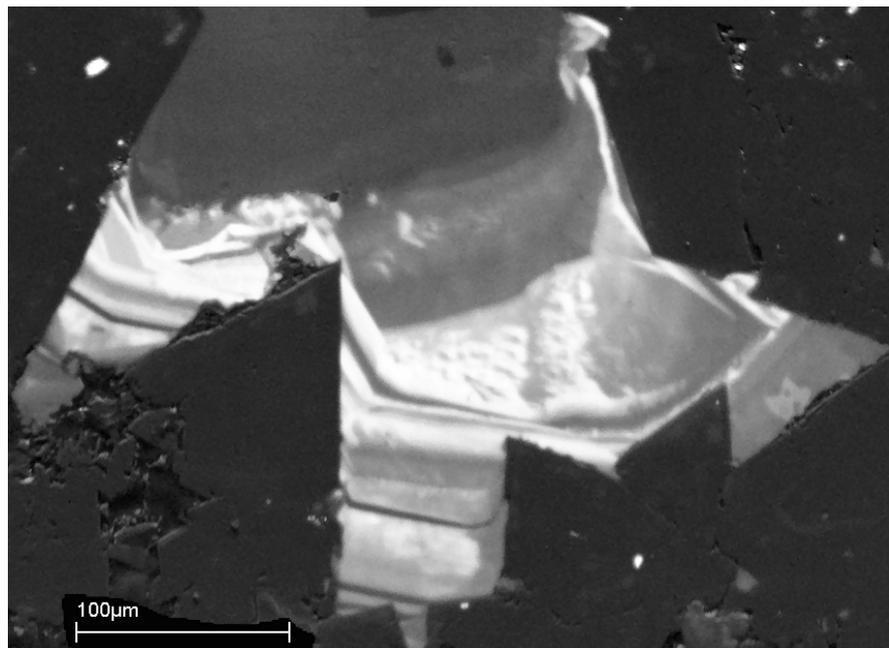
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Development of Capability in the SEM-CL of carbonates

Laboratory Operations Programme

Internal Report IR/06/111



BRITISH GEOLOGICAL SURVEY

LABORATORY OPERATIONS PROGRAMME

INTERNAL REPORT IR/06/111

Development of Capability in the SEM-CL of carbonates

J.E. Bouch

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Front cover

SEM-CL image of calcite cement in saccharoidal dolostone, showing complex internal zonation.

Bibliographical reference

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British Geological Survey offices

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Parent Body

Natural Environment Research Council, Polaris House, North Star Avenue, Swindon, Wiltshire SN2 1EU

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Foreword

This report is the published product of a study by the British Geological Survey (BGS), and was carried out using Maintenance and Development of Capability funding. It investigates methodologies to overcome problems associated with imaging carbonate minerals using scanning electron microscope-based cathodoluminescence systems.

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Summary

This report describes investigations into methodologies that can be applied to overcome imaging problems associated with carbonate minerals on scanning electron microscope-based cathodoluminescence (SEM-CL) systems. The problem arises due to the persistent nature of luminescence from carbonate minerals, which causes ghosting or streaking across SEM-CL images. Two methodologies were tested:

- The first methodology (Lee 2000) applied very long image acquisition times that, in certain situations proved capable of producing excellent images at higher resolution than is possible using optical-based CL systems. However, the image acquisition times are too slow (c. 40 minutes per image) to be useful in most day-to-day situations.
- The second methodology (Reed and Milliken 2003) uses an optical filter to remove the portion of the CL spectrum responsible for the persistent luminescence (in this case in the yellow to red portion of the visible light spectrum). This enabled capture of SEM-CL images at far faster acquisition times (c. 5 minutes per image) than was possible without the filter. However, the resulting 'filtered' images suffer from relatively poor contrast and zoning apparent in these images did not always match zoning observed in optical CL or unfiltered SEM-CL images.

Poor image contrast was observed in the filtered images because the luminescence in the studied carbonates is predominantly due to activation by substitution of Mn, which predominantly occurs in the orange to red portion of the visible spectrum. Therefore, this type of luminescence was effectively excluded by the filter. Consequently the measured signals from the detector reflect the much less intense intrinsic luminescence of the carbonate or luminescence activated by other substituted cations (e.g. rare earth elements) or thermally activated luminescence.

Although filtered SEM-CL carbonate imaging met with variable levels of success, the filtered imaging approach will prove useful in the SEM-CL analysis of quartz in mixed quartz-carbonate-bearing lithologies such as carbonate-cemented sandstones, which have previously been hindered by the persistence of the luminescence from the carbonates.

1 Introduction

CL imaging is widely used in the petrographical analysis of carbonate minerals. This is conventionally carried out on systems mounted on optical microscopes. These instruments flood the surface of the sample with a high energy (typically 10-20 kV, 800-1000 μ A) unfocussed electron beam. As well as generating visible luminescence, this also causes appreciable heating of the sample which may cause it to fracture or may ultimately quench luminescence in certain minerals.

SEM-based CL systems potentially offer greater spatial resolutions, more stable operating conditions and more sensitive detection of luminescence, as well as access to a range of other detectors such as X-ray analysis systems. However, problems arise due to the persistence of luminescence from carbonates - in simplistic terms, carbonate excited by the scanned electron beam continues to luminesce long after the beam has effectively moved on to the next pixel, and therefore continues to contribute to the signal received by the detector. This causes 'ghosting' or streaking across images as is evident in Figure 1.

Two solutions have been proposed to mitigate against this:

- 1) The first uses long pixel dwell times (e.g. Lee 2000, Lee *et al.* 2005). The beam moves sufficiently slowly for the persistently luminescent materials to stop luminescing before the beam has moved too far away from them.
- 2) The second uses an optical filter placed in front of the detector to eliminate light of the wavelengths associated with the more persistent luminescence (Reed and Milliken 2003).

This small project aims to assess the relatively suitability of each approach.

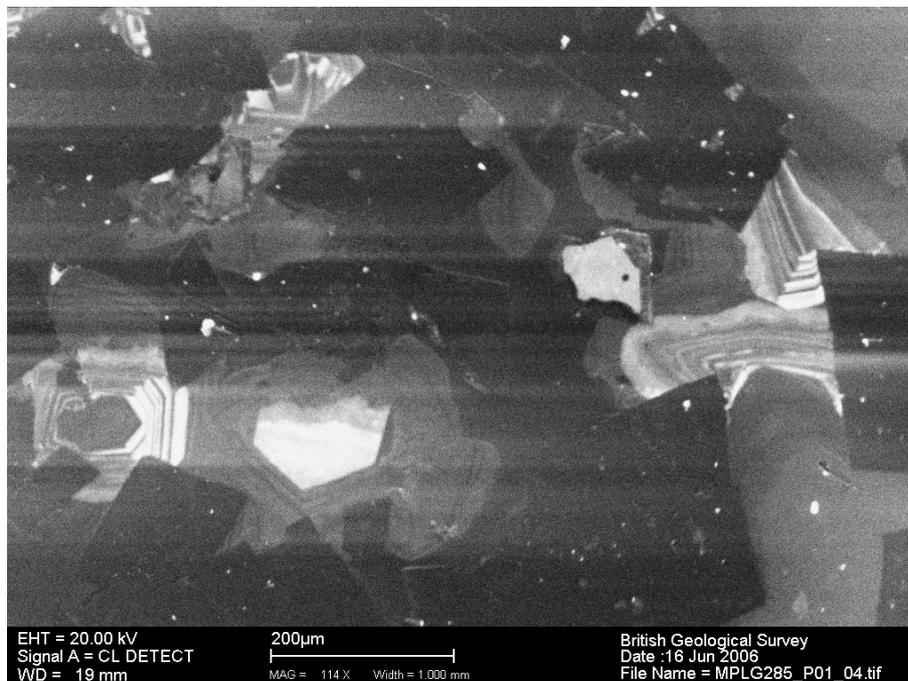


Figure 1 SEM-CL image of calcite cemented saccharoidal dolostone illustrating the ghosting caused by the persistence of luminescence in calcite (Sample MPLG285).

2 Materials and Methods

2.1 SAMPLES

The samples selected for testing (Table 1) have all been characterised by earlier BGS projects and are known to contain calcite and dolomite with a range of luminescence characteristics.

2.2 INSTRUMENTATION

Optical CL images were acquired using a Technosyn 8200 MKII luminoscope attached to a Nikon Labophot microscope. The system vacuum was regulated to give an accelerating voltage of between 10-15 kV, and a beam current of 800-1200 μ A. Digital images were captured using a Nikon Coolpix 4500 camera.

The SEM-CL tests were carried out using a LEO 435VP variable pressure digital SEM, fitted with an Oxford Instruments ISIS energy dispersive x-ray analysis (EDXA) system, KE-developments 4-quadrant backscattered electron (BSE) detector and Oxford Instruments miniCL CL detector. This instrument also has an Oxford Instruments monoCL CL spectrometer, which is not normally mounted on the microscope and was not used for this study.

The SEM was run at 20 kV, with a working distance of 19 mm - the optimum distance on this system for CL imaging and EDXA. A range of probe currents were applied (1 to 10 nA). The microscope was run at high-vacuum for carbon-coated polished thin-sections, and at variable pressure for non-coated sections.

Reed and Milliken (2003) conducted their imaging using an Oxford Instruments monoCL system of comparable specification to the one owned by BGS, however as our monoCL detector is normally left un-mounted we decided to attempt to replicate their results using our miniCL detector. The miniCL is sensitive in the range 185-850 nm (ultraviolet to red).

A 'short pass' optical filter of comparable specification to that used by Reed and Milliken (2003) was obtained from Glen Spectra (<http://www.glenspectra.co.uk>, product code 500CFSP). The transmission curve of this filter is shown in Figure 2 alongside with published emission curves for dolomite, calcite and aragonite luminescence (Richter *et al.* 2003; Reed and Milliken 2003).

The filter was positioned over the tip of the miniCL detector using a holder manufactured by BGS workshops. This filter holder is a replica of a mirror attachment that is fitted as standard on the miniCL detector, but with the addition of a thin lip to hold the filter in place. This attachment is also c. 1 mm longer than the standard mirror attachment on which it is based. Therefore, clearance between this attachment and the SEM BSE detector during BSE detector insertion/retraction is very close, and care needs to be taken to avoid collisions between the detectors. The filter and filter holder could equally be fitted to our older S360 instrument, which is also equipped with a miniCL detector, although again there are issues related to clearance between the miniCL and BSE detectors in this instrument.

Samples were imaged using a variety of SEM scan speeds (Table 2), a range of probe currents with the filter in place on the end of the detector, and with the filter removed.

Table 1 Summary details of the sample used for testing purposes.

Sample ID	Grid Reference (BNG)	Location	Formation	Carbon-Coated	Comments
MPLG278	441120, 321520	Cloud Hill Quarry, Leicestershire	Milldale Limestone	No	Dolostone with fracture lined with ankerite and later calcite. Reference: Bouch <i>et al.</i> 2004
MPLG285	441320, 321200	Cloud Hill Quarry, Leicestershire	Milldale Limestone	Yes	Saccharoidal dolostone with calcite cement. The calcite shows considerable variations in luminescence. Reference: Bouch <i>et al.</i> 2004
MPLH217	404780, 283397	Birmingham University Borehole BH-1, 18.73-18.78m bGL	Wildmoor Sandstone	Yes/No	Medium grained sandstone containing abundant grains of pedogenic dolomite. The sample is also cut by a cm-scale calcite-cemented fracture. Reference: Bouch <i>et al.</i> 2006

Table 2 LEO scan speed, pixel dwell time and image acquisition times (1024*768 pixel images).

SEM Scan Speed	Pixel Dwell Time (μ s)	Scan Time (Seconds)	Scan Time (Minutes: Seconds)
0	0.10	0.077	-
1	0.19	0.153	-
2	0.39	0.306	-
3	0.78	0.613	-
4	1.6	1.225	-
5	3.1	2.450	-
6	6.2	4.90	-
7	12.5	9.80	-

SEM Scan Speed	Pixel Dwell Time (μ s)	Scan Time (Seconds)	Scan Time (Minutes: Seconds)
8	25	19.6	-
9	50	39.2	-
10	100	78.4	01:18
11	200	157	02:37
12	400	314	05:14
13	800	627	10:27
14	1600	1254	20:54
15	3200	2509	41:49

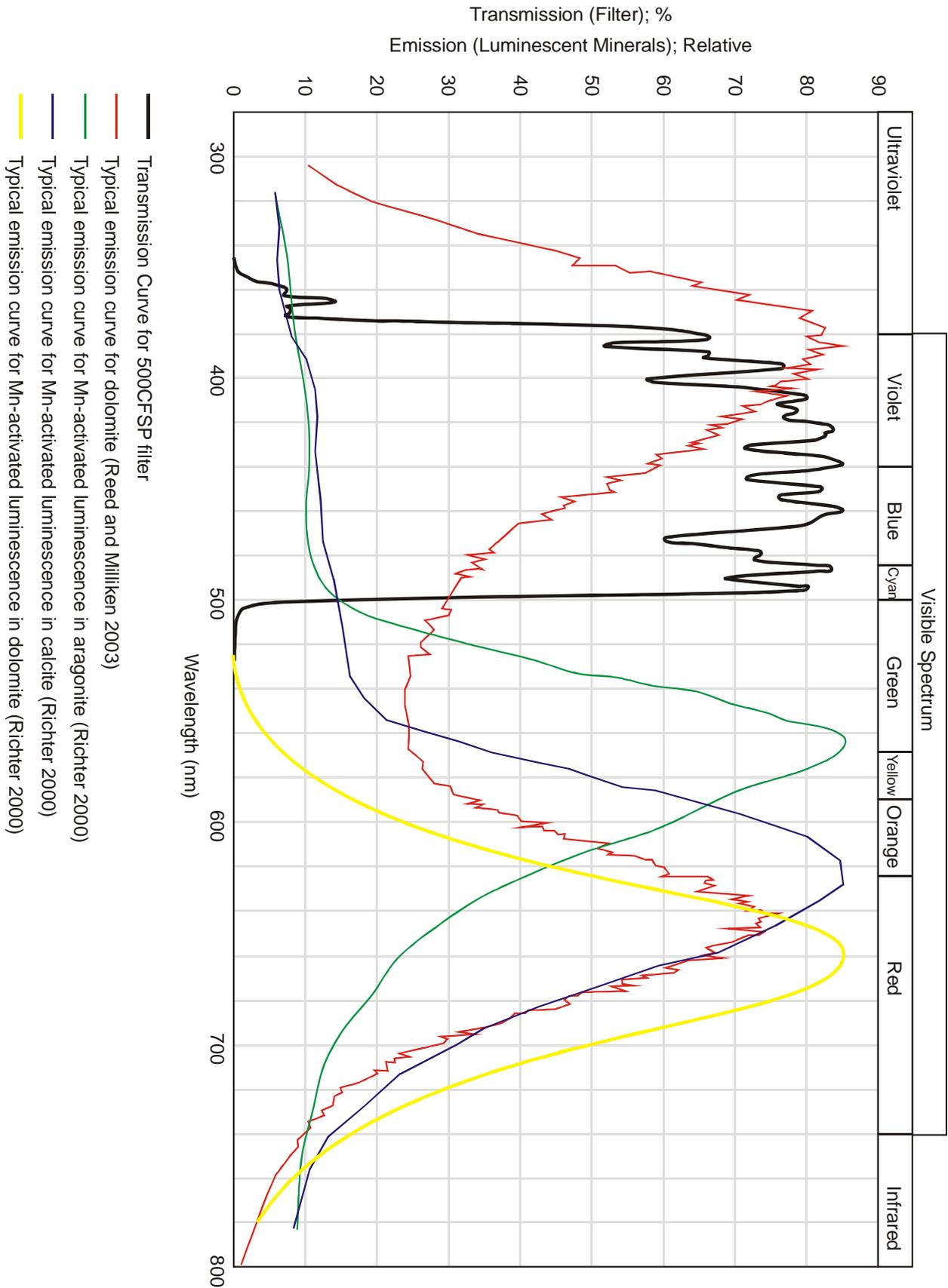


Figure 2 Transmission curve for filter 500CFSP, and curves for light emission from dolomite, calcite and aragonite. The filter transmits light in the wavelength range c. 380-500 nm and blocks wavelengths outside this range.

3 Results

3.1 LONG PIXEL DWELL TIMES

The simplest solution to the problem of persistent luminescence is to use longer SEM scan times. This gives persistently luminescent materials time to stop luminescing before the beam moves on to the next point. For example, Lee (2000) generated detailed images of zoning within calcites using a pixel dwell time 3200 μs . Table 2 documents the ‘scan speeds’, the equivalent pixel dwell time of each scan speed, and the total image acquisition time (for a 1024*768 pixel image) available on our LEO435VP instrument. 3200 μs equates to a 40 minute acquisition time (scan speed 15) on this instrument. Experiments indicated that images acquired using pixel dwell times of 1600 μs or less invariably suffered from ghosting (Figure 3B and Figure 4B). Pixel dwell times of 3200 μs were usually (Figure 4C), but not invariably (Figure 3C) successful in eliminating these effects.

In Figure 3, progressively longer pixel dwell times is seen to reduce the degree of ghosting associated with the calcite. However the grains of orange luminescent dolomite proved to have particularly persistent luminescence which remained problematic even at pixel dwell times of 3200 μs . Furthermore, the fine scale variations in calcite luminescence seen in the optical CL image (Figure 3A) were not detected using SEM-CL.

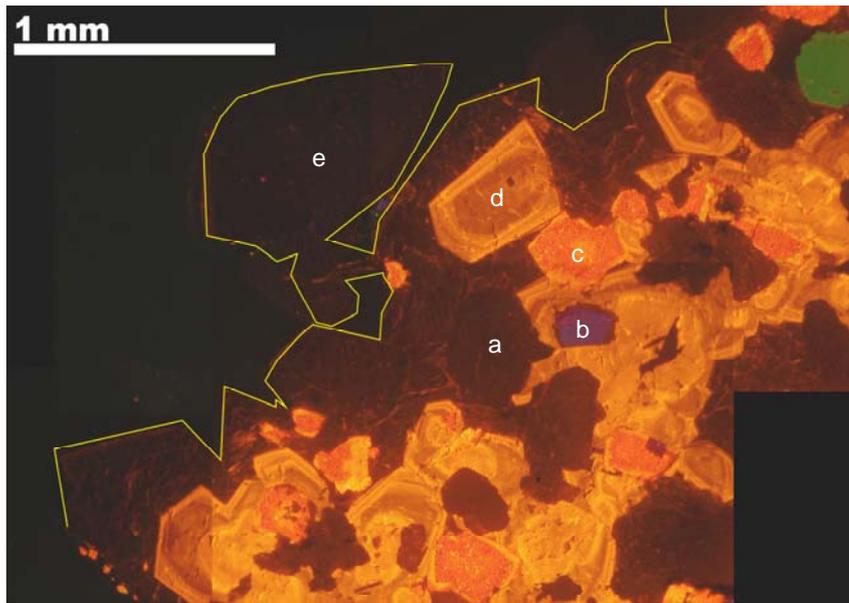
In Figure 4, the SEM-CL images bear closer resemblance to the optical CL image and retain a high level of detail and the fine-scale zoning is clearly detected. In this sample, in which the dolomite is non-luminescent, it proved possible to use long pixel dwell times to acquire images of higher resolution than is possible using our existing optical CL system (Figure 4B and C). The greater sensitivity of the miniCL detector also enables detection of luminescence from calcite, which appears non-luminescent using the optical CL system (compare Figure 4A and B). However, whilst the images captured using long pixel dwell times in this case are of excellent quality, the acquisition time of 40 minutes makes this an impractical solution for day-to-day imaging work.

Figure 3 (opposite) Comparison of optical and unfiltered SEM-CL images in calcite-cemented sandstone (sample MPLG217).

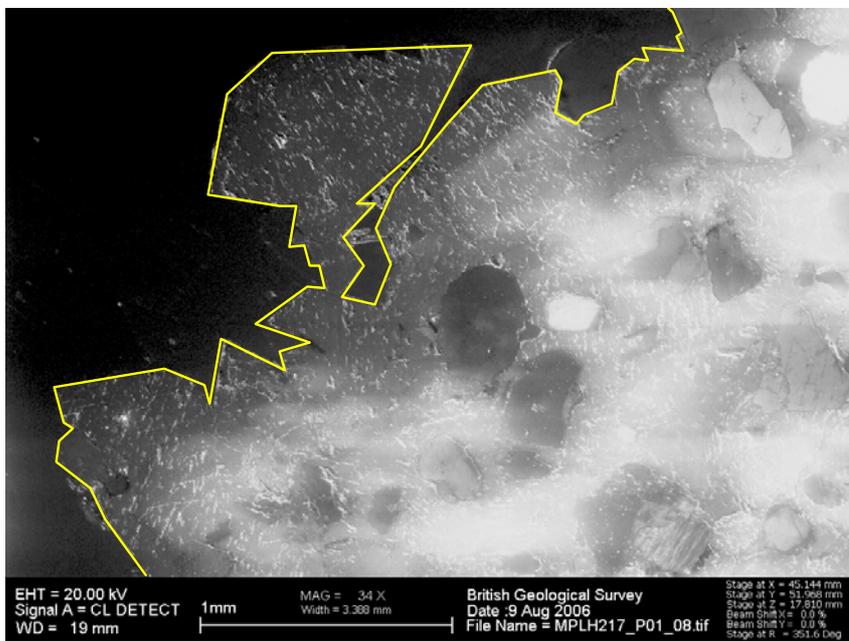
A) Optical CL image. Quartz grains are non-luminescent (a), feldspar is purple (b), pedogenic dolomite is orange luminescent with yellow speckling (c), and calcite is variably brown-orange luminescent with well-developed concentric zonation (d) and non-luminescent (e). The outline of the non-luminescent calcite is marked in yellow.

B) Unfiltered SEM-CL image of the same field of view as that shown in (A). This was acquired at a pixel dwell time of 1600 μ s. Gross variations in carbonate luminescence are evident, with the clearly resolved differences between dolomite (bright white luminescence) and the variably luminescent calcite. However, the fine scale detail is not resolved, and the dolomite is causing ghosting across the image. (20 kV, 10 nA, variable pressure 0.375 Torr.)

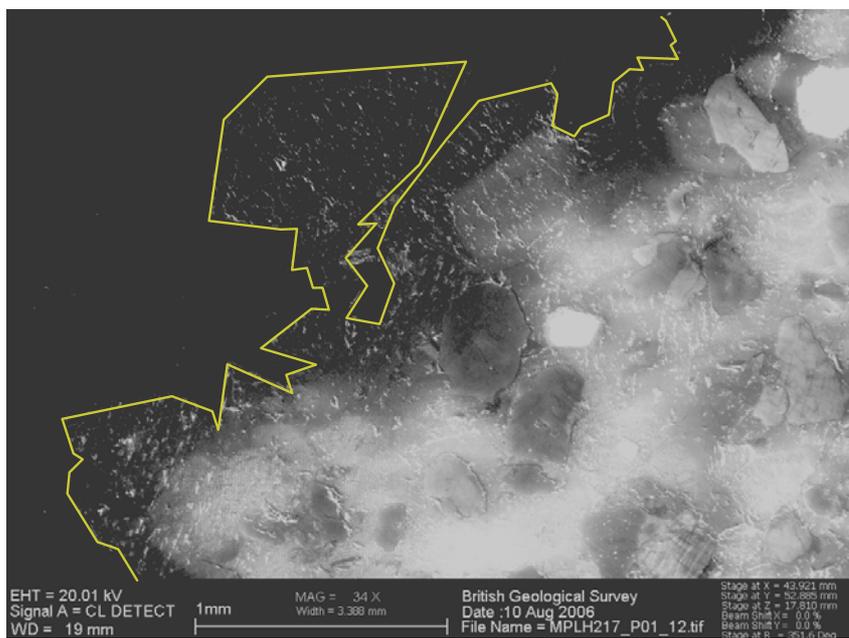
C) Same field of view as (B), but using pixel dwell time of 3200 μ s. Even at this long pixel dwell time, some ghosting across the image is evident, particularly from the relatively brightly luminescent dolomite. (20 kV, 10 nA, variable pressure 0.375 Torr.)



A



B



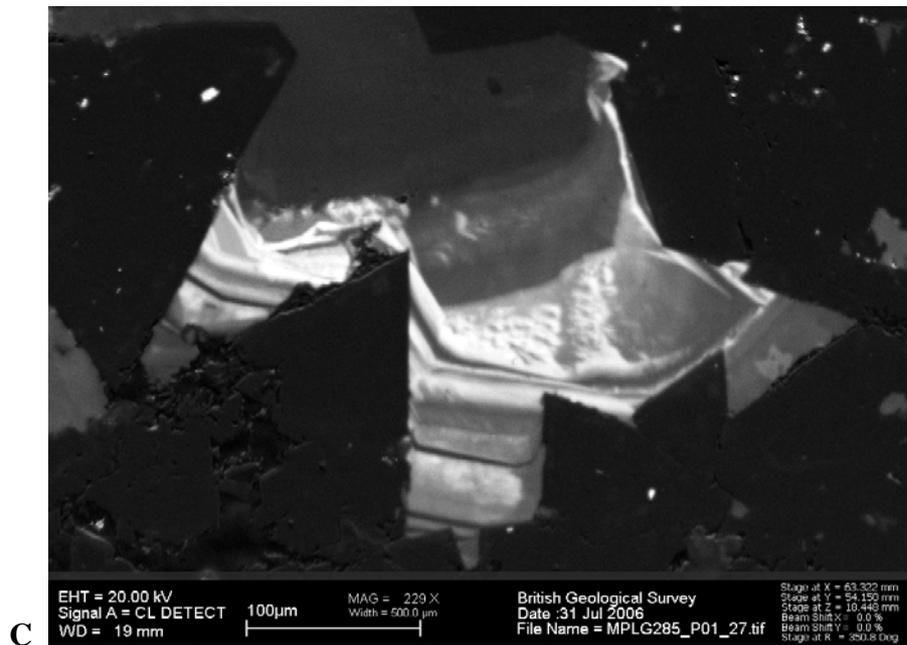
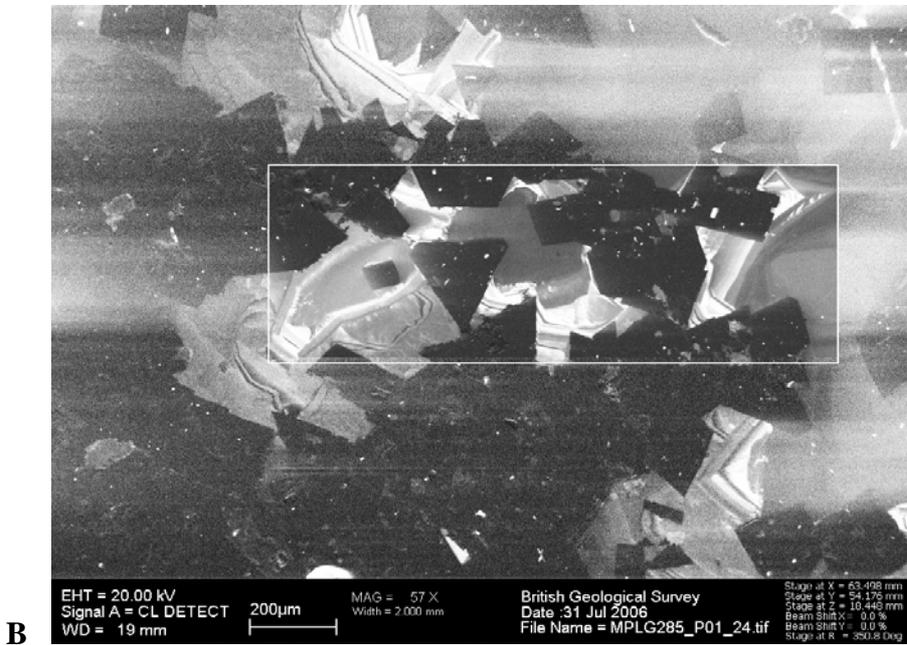
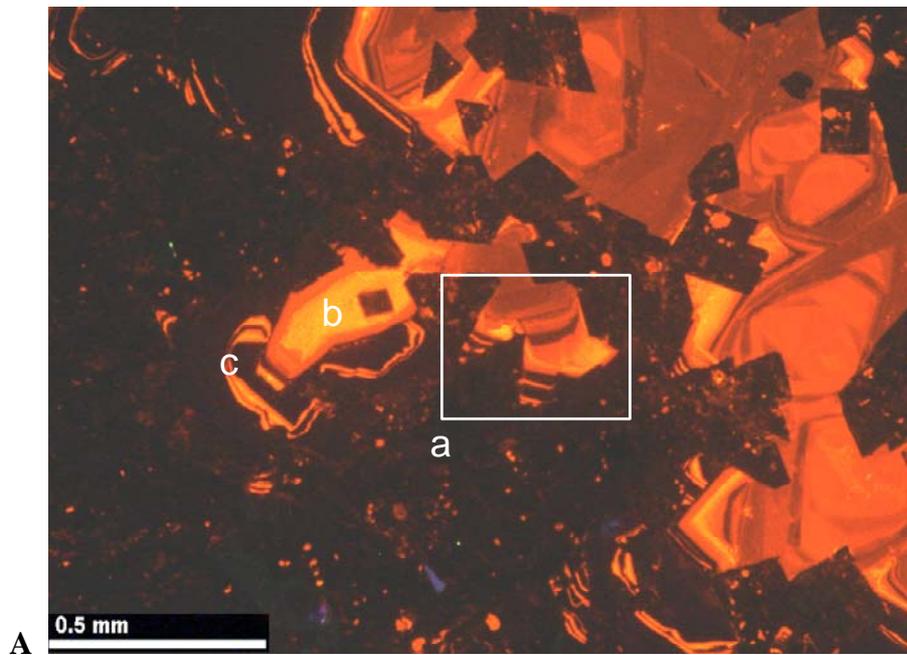
C

Figure 4 (opposite) Comparison of Optical and unfiltered SEM-CL images of calcite-cemented saccharoidal dolostone (Sample MPLG285).

A) Optical CL image showing non-luminescent rhombic dolomite (a), and variably luminescent calcite (b). The outermost generations of calcite are non-luminescent with thin brightly luminescent bands (c).

B) Unfiltered SEM-CL image of the same field of view as that shown in (A). This was acquired at a pixel dwell time of 400 μ s and is affected by ghosting from persistently luminescent calcite. The boxed area in the centre of the image was acquired at a pixel dwell time of 3200 μ s, which in this sample was sufficient to remove the ghosting. (20 kV, 5 nA, high vacuum.)

C) Detailed, unfiltered SEM-CL image of the area highlighted by the white box in (A). This image was acquired using a long pixel dwell time (3200 μ s), and illustrates the higher resolutions it is possible to achieve using SEM-CL relative to optical CL. (20 kV, 5 nA, high vacuum.)



3.2 FILTERED IMAGING

The filtered approach of Reed and Milliken (2003) met with variable levels of success for the imaging of carbonates (Figures 5 and 6), and a number of differences are apparent between the filtered SEM-CL images, the unfiltered SEM-CL images and the optical CL images. Whilst the persistent luminescence was eliminated very effectively, this was accompanied by a serious loss of signal in the filtered SEM-CL images. This meant that the detector had to be run at maximum contrast in order to generate a meaningful signal, and that offline processing in a image manipulation package (Paint Shop Pro in this case) was required in order to adjust brightness/contrast/gamma levels to produce useful images.

In some cases, even though image contrast was significantly reduced, useful images could still be acquired using much shorter pixel dwell times (400 μ s) than were possible without the filter (Figure 5). In other cases (Figure 6), the degradation of image contrast was so severe that zoning apparent in optical CL and unfiltered SEM-CL images could no longer be discerned. Furthermore, in the case of the dolomite shown in Figure 6 luminescence was ‘inverted’, with previously brightly luminescent zones appearing non-luminescent.

The variations in the appearance of the zoning between unfiltered and filtered SEM-CL images are explicable by the fact that there are a number of different mechanisms by which luminescence in carbonates may be activated or quenched. The most significant activator of luminescence is substitution of Mn for Ca and/or Mg, which typically produces yellow, orange or red luminescence depending upon the host mineral (i.e. emission in the wavelength range c. 560-760 nm; Figure 2; Richter 2003). These wavelengths are eliminated using the filter used here, so the loss of signal and contrast in the filtered images is not entirely surprising. ‘Intrinsic’ luminescence in carbonates tends to occur in the ultraviolet to violet range (<c. 400 nm; Walker 2000) and is therefore, at least partially, transmitted by our filter and detected by the miniCL detector. These short wavelengths may be relatively poorly detected by the digital camera attached to our optical CL system (due to its low intensity and/or its position within the invisible part of the spectrum). This means that luminescence of this type would be detected using SEM-CL from zones that would appear non-luminescent using optical CL.

It therefore seems appropriate to suggest that, in general terms, filtered SEM-CL imaging of carbonates tends to capture variations in intrinsic luminescence, luminescence activated by substitution of other cations (e.g. rare earth elements) or thermal luminescence caused by heating of the sample by the electron beam. In contrast, unfiltered SEM-CL (and optical CL) imaging of carbonates captures variations in Mn-activated luminescence. In some cases these images show close degrees of similarity, in others, significant differences are observed.

Figure 5 (Opposite) Filtered SEM-CL images of calcite-cemented saccharoidal dolostone (Sample MPLG285).

A) Filtered SEM-CL image of the same field of view shown in Figure 4A and B acquired using a pixel dwell time of 400 μ s. Whilst the image suffers from poorer contrast relative to the images in Figure 4, the zoning is still apparent. (20 kV, 5 nA, high vacuum.)

B) Filtered SEM-CL detail of the boxed area in (A) also shown in Figure 4C, acquired using the relatively short pixel dwell time of 400 μ s. Again, this image suffers from a relative lack of contrast, however the zoning is still evident. (20 kV, 5 nA, high vacuum.)

C) BSEM detail of the boxed area in (A) showing a similar set of zoning to that evident in the CL image. (20 kV, 5 nA, high vacuum.)

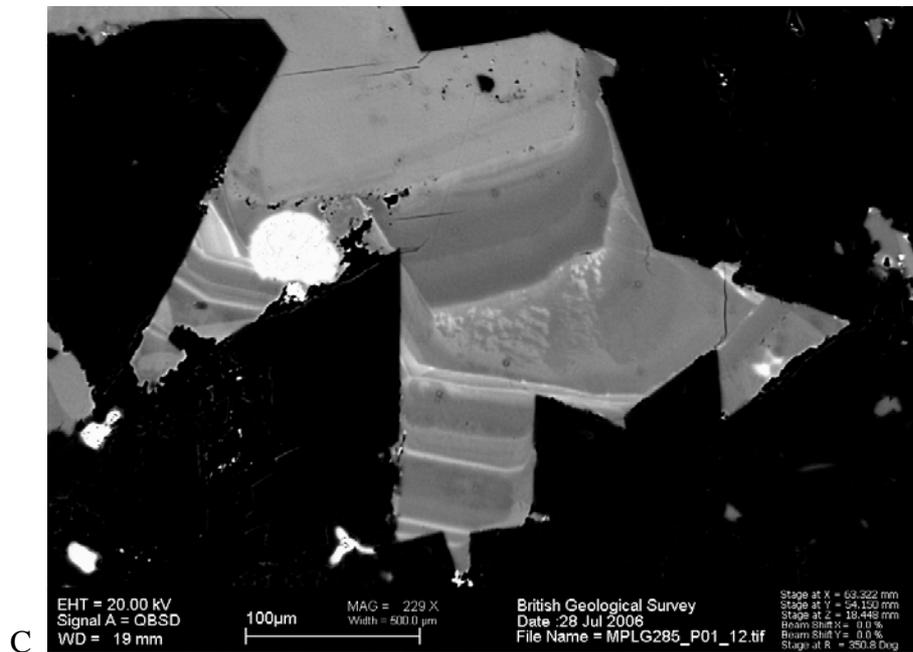
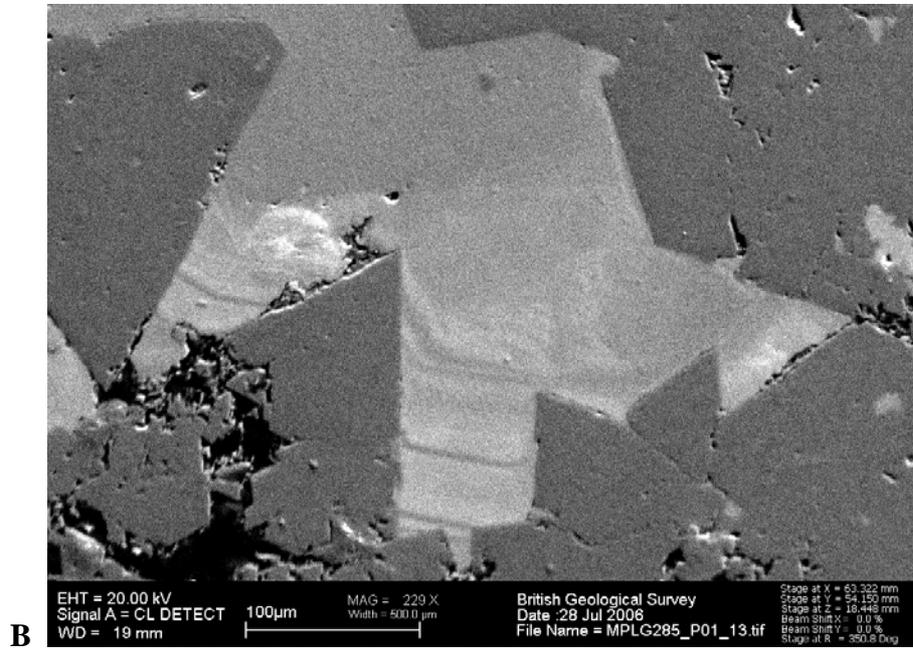
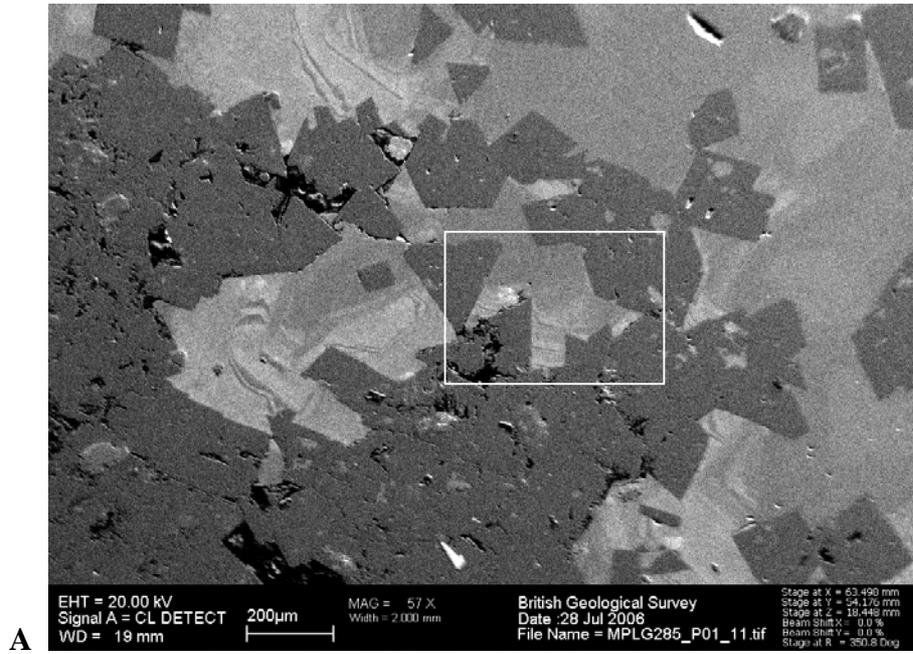
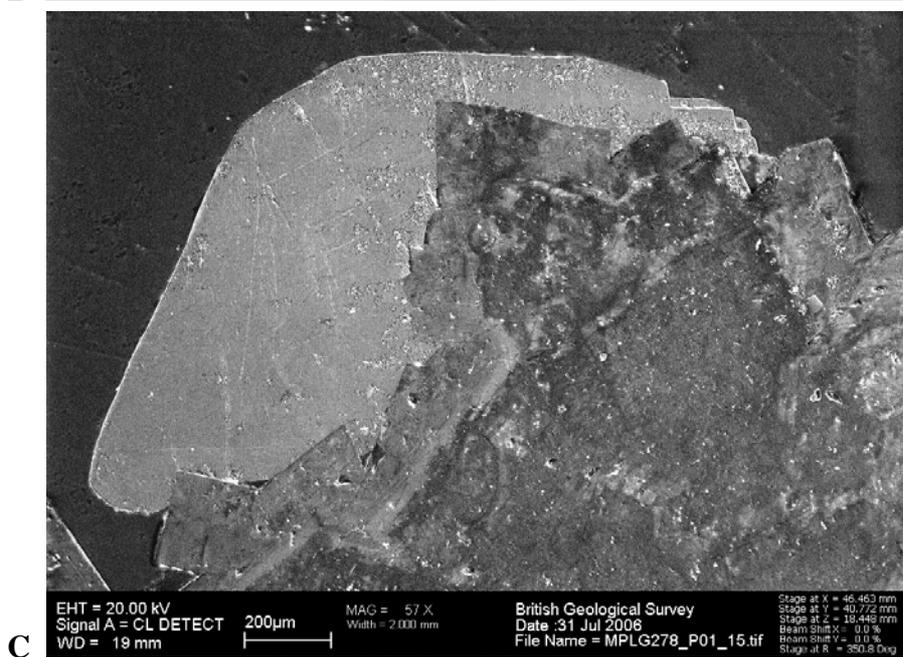
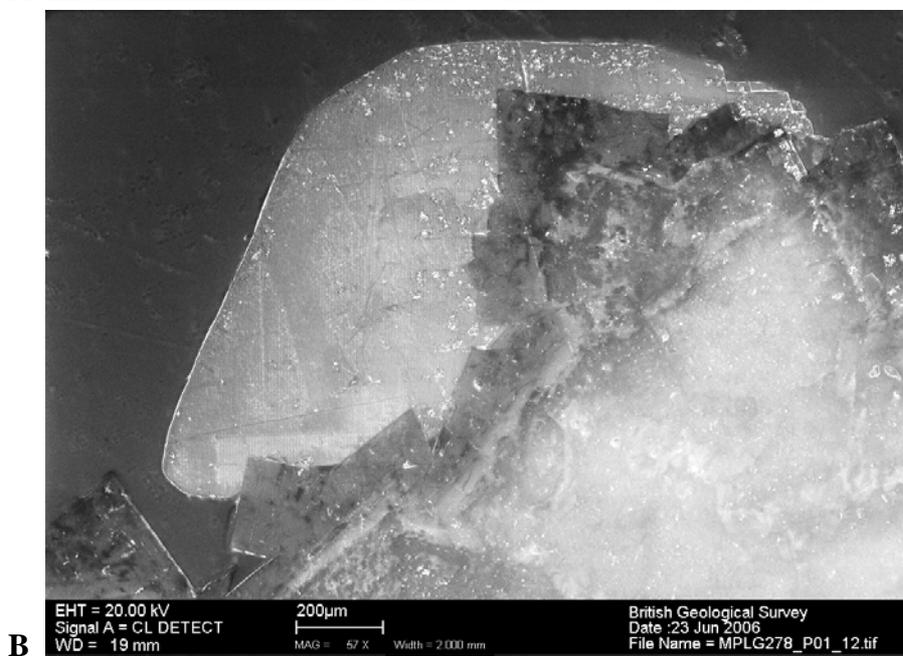
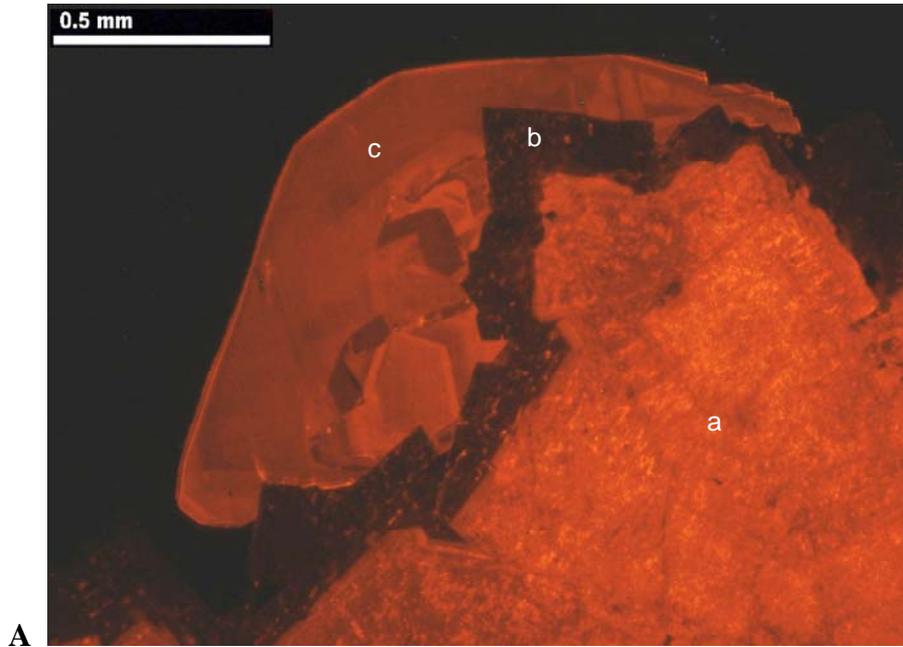


Figure 6 (opposite) Comparison of optical, unfiltered SEM-CL and filtered SEM-CL images of fracture in dolostone, coated by ankerite and calcite (sample MPLG278).

A) Optical CL image of calcite developed on the wall of a fracture within dolostone. The dolostone is relatively brightly orange-yellow luminescent (a), with a thin outer band of non-luminescent Fe-rich dolomite (ankerite; b). The calcite (c) shows well-developed internal zoning.

B) Unfiltered SEM-CL image of the same field of view shown in (A) acquired with a pixel dwell time of 3200 μ s. Minor ghosting from the luminescent dolomite in the wallrock is evident on the image even at this slow scan speed. The finer-scale zoning within the dolomite is clearly resolved, whereas that in the calcite is poorly defined. (20 kV, 2 nA, variable pressure 0.235 Torr.)

C) Filtered SEM-CL image acquired using a pixel dwell time of 800 μ s. The zoning in the calcite is not resolved, and the luminescence within the dolomite has been 'inverted' – the area that was brightly luminescent in the unfiltered image is only very dully luminescent, whereas the previously dully-luminescent band now appears to be more strongly luminescent. (20 kV, 5 nA, variable pressure 0.218 Torr.)



3.3 SEM-CL OF CARBONATE CEMENTED SANDSTONES

One application where the suppression of luminescence from carbonate is desirable is in the SEM-CL imaging of mixed carbonate- and quartz-bearing lithologies such as the study of detrital and/or authigenic quartz in carbonate cemented sandstones (Figure 7A). Quartz is typically only very dully luminescent using optical CL, and unfiltered SEM-CL images suffer from the same problems of ghosting produced by the persistently luminescent calcite (Figure 7B), necessitating impractically long image acquisition times as discussed earlier.

In filtered SEM-CL images, however, the problematic persistently luminescent wavelengths from carbonates are suppressed, enabling the acquisition of SEM-CL images of quartz using relatively short pixel dwell times (400 μ s; Figure 7C). However, as in the case of SEM-CL of carbonate, differences may be observed between the filtered and unfiltered SEM-CL images, with some quartz grains appearing brightly luminescent in unfiltered images and non- or dully-luminescent in the filtered images. This reflects variations between the wavelength spectra of the light emitted by different quartz types:

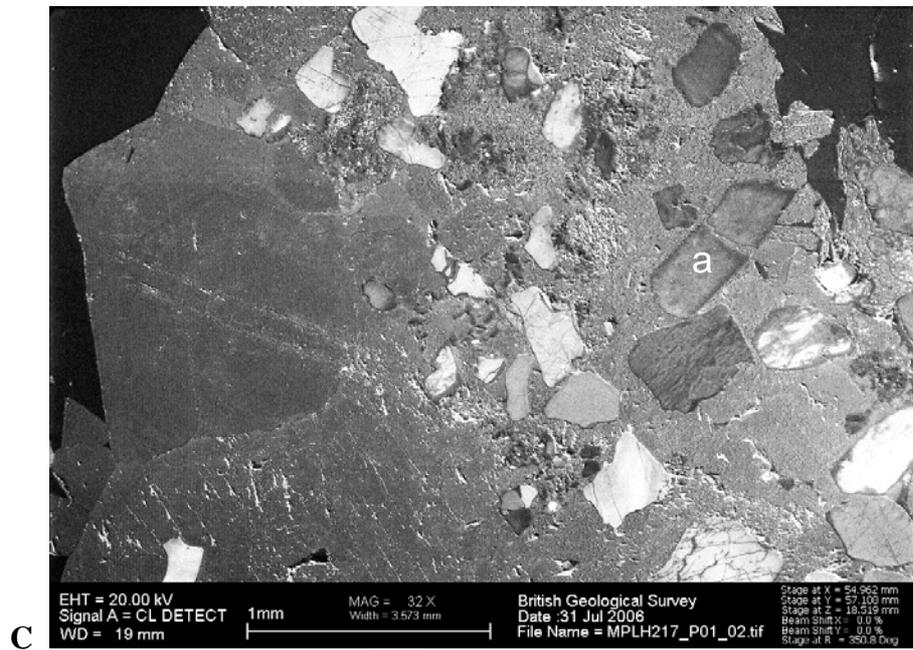
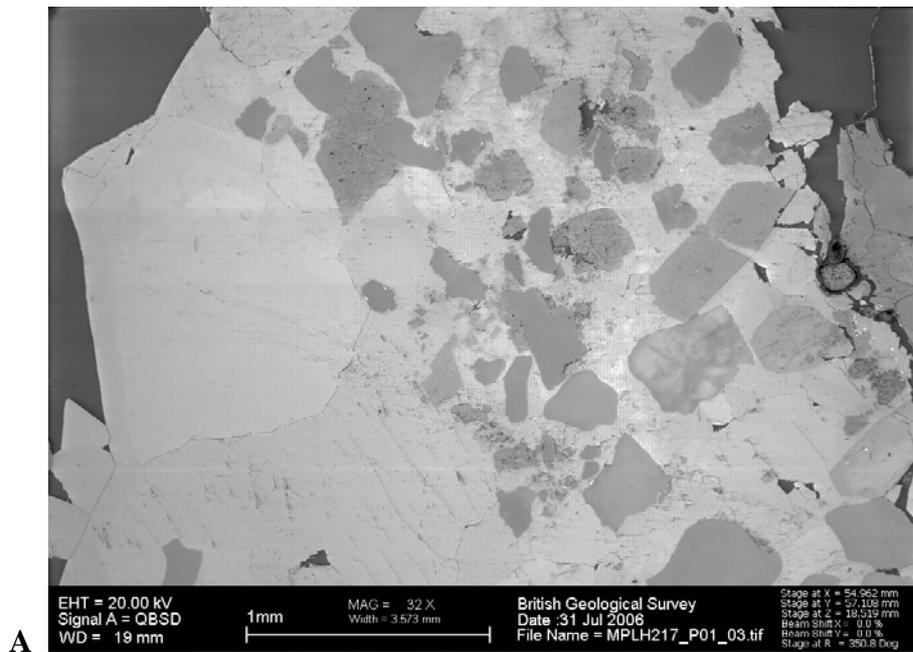
- Grains that show no change in luminescence between the unfiltered and filtered SEM-CL images are presumably emitting light predominantly in the violet to yellow portion of the spectrum,
- Grains that show a change in luminescence intensity are presumably emitting light predominantly in the green to red portion of the spectrum that is blocked by the filter.

Figure 7 (opposite) Comparison of BSEM, unfiltered SEM-CL and filtered SEM-CL images in calcite cemented sandstone (sample MPLH217).

A) BSEM image showing calcite cement (light-grey) and detrital quartz grains (darker grey). (20 kV, 5 nA, variable pressure 0.158 Torr.)

B) Unfiltered CL image acquired using a pixel dwell time of 400 μ s. In this image the dolomite and calcite produce significant ghosting across the image, which obscures luminescence variations in the quartz. (20 kV, 5 nA, variable pressure 0.158 Torr.)

C) Filtered SEM-CL image acquired using the same 400 μ s pixel dwell time as (B). The ghosting is removed and a number of quartz grain types of different luminescence characteristics are evident. Furthermore, some quartz grains that are brightly luminescent in the unfiltered image (B) are only dully luminescent in this image (compare grain 'a'). This indicates that these particular grains luminesce relatively strongly in the wavelength range 380-500 nm which is being blocked by the filter. (20 kV, 10 nA, variable pressure 0.158 Torr.)



4 Conclusions

When capturing SEM-CL images of carbonate bearing materials, persistent luminescence of the carbonate minerals causes ghosting across images. This can be mitigated using slow scan times, but the scan durations required to generate images of sufficient quality for even informal publications are too long (c. 40 minutes) to be of use in all but the most rare occasions.

A short-pass optical filter, of comparable specification to one used by Reed and Milliken (2003) to suppress the persistent luminescence, was fitted to our miniCL detector. This methodology severely restricted detection of the luminescence caused by Mn substitution – which tends to produce yellow, orange or red luminescence – but permitted detection of intrinsic luminescence, luminescence activated by other cations (e.g. rare earth elements) or thermally activated luminescence.

Intrinsic luminescence in carbonates occurs at relatively low intensity compared with the luminescence resulting from Mn-substitution. Consequently, filtered SEM-CL images are of notably lower contrast and require application of larger offline brightness/contrast/gamma corrections than their unfiltered equivalents. Furthermore, the zoning evident in optical and unfiltered SEM-CL images arising from Mn-substitution was not always observable in the filtered SEM-CL images. However, for some carbonates, it was possible to acquire useful images of intrinsic luminescence using reasonably short image acquisition times (typically c. 5 minutes).

Suppression of luminescence from carbonates is potentially very useful in the SEM-CL analysis of mixed carbonate- and quartz-bearing lithologies such as carbonate-cemented sandstones, which has previously proved problematic. Use of the filter enables capture of SEM-CL images of quartz in calcite cemented sandstones using relatively short acquisition times (c. 5 minutes).

Comparison of unfiltered and filtered SEM-CL images provides an additional method to discriminate between different types of both carbonate and quartz, which may luminesce with different colours. This approach is similar to that applied in 'colour' SEM-CL imaging, which combines images acquired using a range of filters into colour channels to generate a single colour image (e.g. Reed's Scanned CL webpage, or Gatan's commercially available ParaCL system).

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Most of the references listed below are held in the Library of the British Geological Survey at Keyworth, Nottingham. Copies of the references may be purchased from the Library subject to the current copyright legislation.

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