CHARGE CONTRAST IMAGING (CCI) IN THE ENVIRONMENTAL SCANNING ELECTRON MICROSCOPE: OPTIMIZING OPERATING PARAMETERS FOR CALCITE

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Charge contrast imaging (CCI) is a useful new method for imaging sub-micron features in crystalline materials using the unique gas/ion/electron imaging system of the environmental scanning electron microscope (Griffin, 1997; Doehne, 1998). Crystal growth zoning, microfractures, solution boundaries, and areas of chemical alteration or recrystallization can be imaged in a wide range of materials (Griffin, 2000; Watt, et al. 2000). While not fully understood, charge contrast images reflect differences in the ability of materials to accept, store and discharge deposited electrons from the primary electron beam. These differences are expressed, in turn, as contrasts in secondary electron emission from flat samples (e.g. these contrasts are not related to topography, as is usually the case). Charge contrast appears be related to differences in electronic properties which are often controlled by defect density. CCI is also affected by small-scale physical defects (such as microfractures) which appear to affect the distribution and timing of charge buildup and discharge in the sample (Johansen, et al. 1997). It is not clear if charge contrast is due to differences in the number, energy distribution or depth of the secondary electrons being emitted, or some combination. The remarkable 3-D appearance of the CCI images obtained from flat samples is worth noting and suggests a potential dependence of this contrast on secondary electron escape depth. CCI has also been called charge-induced contrast (Harker 1994) while earlier authors have suggested the term impurity-sensitive contrast (Sawyer and Page 1978).

To further develop this method, we present here data on the variation of CCI contrast with typical ESEM instrument parameters. Charge contrast imaging was first noted by the authors when studying samples of Travertine stone from Tivoli, Italy, which consists mostly of calcite. The conditions in the ESEM were systematically varied and the impact on the CCI contrast noted. The average trends from a series of these tests are summarized in Figure 1. A typical sample is illustrated in Figures 2 and 3. The contrast observed in the calcite sample appears primarily as darker and lighter, often parallel bands (crystal growth banding), complex, symmetrical features (which resemble sector zoning) and irregular interfaces (apparently dissolution surfaces). Cathodoluminescence (CL) images of calcite are similar to CCI images, but much less detailed. CCI contrast in our calcite sample appears to occur under conditions of high detector gain (detector bias or electronic gain) and fast scan rate (Figure 1). Obtaining high contrast CCI images at short working distance was not possible due to arcing from the detector to the sample with a high detector bias. Somewhat longer working distances allowed the detector bias to be put to the maximum on this instrument (~535 volts). The greatest CCI contrast appears at the highest kV with decreasing contrast visible in some areas until about 5 kV. Future work on this useful method will extend to other types of carbonate samples.

References

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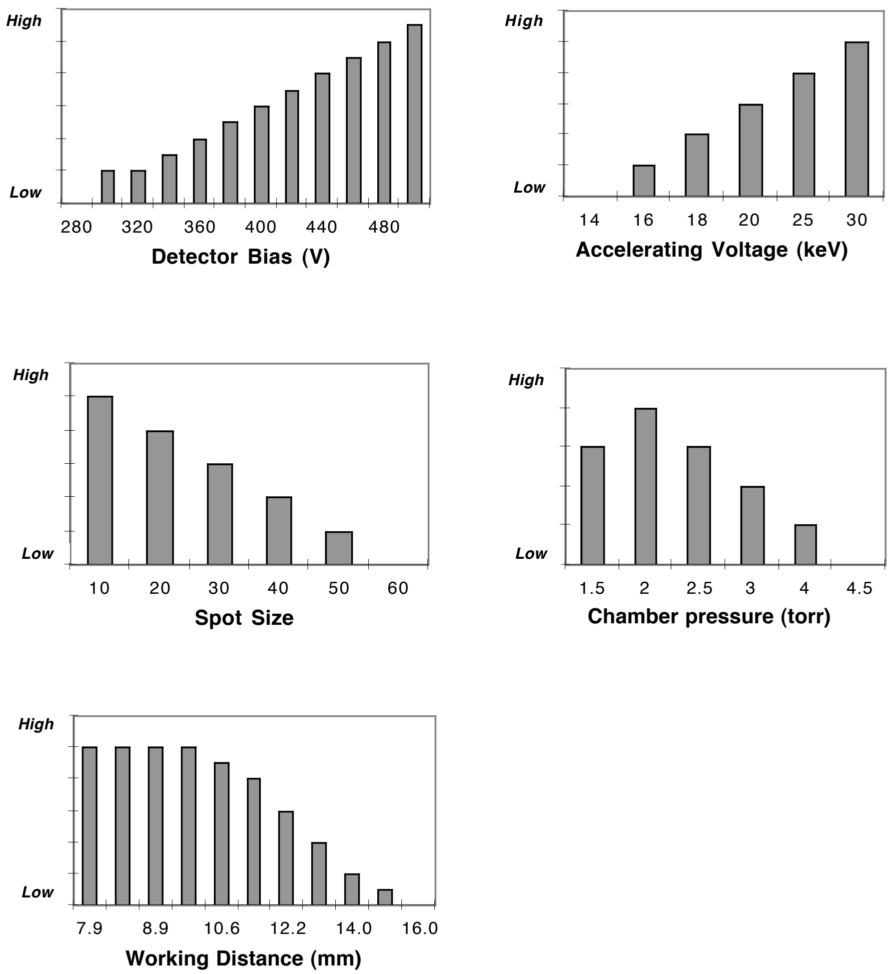
Figure 1. Variation of CCI contrast in calcite with ESEM operating parameters. Scale of contrast is in arbitrary units representing qualitative observations. Standard conditions for all experiments were: condenser setting 40 (on a scale of 0-100, with 100 = minimum spot size), 4.3 sec/fr scan rate, 2.4 torr chamber pressure, 20 keV accelerating voltage, working distance 9 mm. One parameter at a time was varied to gain an understanding of how they alter the CCI contrast.



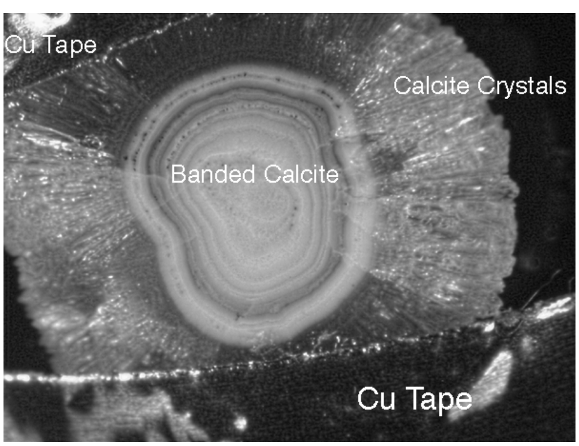


Figure 2. Reflected optical microscope images of travertine sample (calcite). Note field of view is 1 cm.

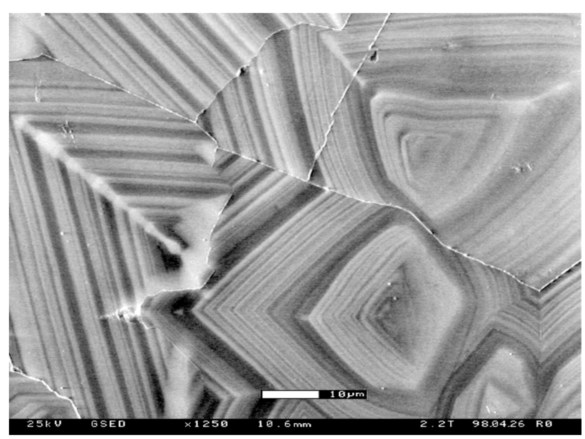


Figure 3. High-resolution charge contrast image (CCI) of crystal growth banding in calcite. Also note sample shows multiple contrasts: i.e. dust on sample surface. Scale is 10 µm.