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Reversed scan direction reduces electron beam damage in EBSD maps

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Summary

The deleterious effects of electron beam damage on high-resolution electron backscatter diffraction (EBSD) maps of undeformed quartz are significantly reduced by scanning in the direction opposite to that dictated by widely used EBSD acquisition software. Higher quality electron backscatter patterns are produced when the electron beam moves progressively down the sample (the apparent 'up' direction in the resulting maps) for all step sizes where beam damage affects EBSD map quality ($\leq \sim 0.4$ μm in this study). The relative improvement associated with downward scanning increases as step size is reduced. A comparison of high-resolution maps made in experimentally deformed quartz demonstrates that downward scanning reduces by a factor of ~ 2 the lower limit in step size relative to maps scanned in the conventional direction. The electron beam damages quartz at its point of entry, forming ~ 0.1-μm diameter bumps visible in Scanning electron microscope (SEM) images. Downward scanning produces better results because it minimizes the flux of electrons through these loci of damaged crystal.

Introduction

Electron backscatter diffraction (EBSD) allows for the rapid acquisition of full crystallographic orientations in many materials and has revolutionized fabric analysis in the Earth and material sciences (Schwartz *et al.*, 2009). Electron backscatter patterns (EBSP) are produced under an electron beam when electrons are refracted and 'channelled' back through an excitation volume comprising the outer few nm of a crystalline lattice (e.g. Prior *et al.*, 1999; Schwarzer *et al.*, 2009). This excitation volume is a small fraction of the interaction volume, the total volume of sample through which the electron beam penetrates. In some common minerals (e.g. quartz and feldspar, Prior *et al.*, 2009) the electron beam damages samples during analysis. This beam damage does not interfere with many EBSD applications because EBSP are generally collected rapidly, before significant damage occurs at any one point. Beam damage does however become a significant problem in high-resolution maps ($< \sim 0.25$ μm step size) due to overlap of new spots with the region damaged during previous analyses (Prior *et al.*, 2009; Bestmann, 2012). Beam damage thus limits the step size of EBSD maps in quartz to about five times the size that can be used in materials that do not damage such as olivine and pyrite (0.05 μm steps possible, Ohfuji *et al.*, 2005; Prior *et al.*, 2009) and most metals (Humphreys & Brough, 1999).

Samples undergoing EBSD analyses are steeply tilted (e.g. Fig. 1), and as a result the interaction volume is located primarily beneath the point where the electron beam enters the sample (e.g. Prior *et al.*, 1999; Schwarzer *et al.*, 2009; Fig. 1). It was thus previously assumed that when making EBSD maps, beam damage would be best avoided by positioning new analyses above previous spots (i.e. in a position at least partially out of the interaction volumes of previous analyses). The early HKL acquisition program Flamenco permitted scanning in either the up or down direction; however, more recent Oxford Instruments and EDAX acquisition software restrict mapping to only the upward direction.

We experienced significant problems due to beam damage while making EBSD maps of fine-grained, deformed quartzite and it was suggested to us by Michel Bestmann that reversing the scan direction might improve results (Bestmann, personal communication, 2010; Bestmann, 2012). We describe here a systematic investigation of the effects of beam damage on EBSP quality in the different scanning directions at a variety of step sizes. Throughout the paper, we use 'up' and 'down' to describe the true direction of the progression of analyses on

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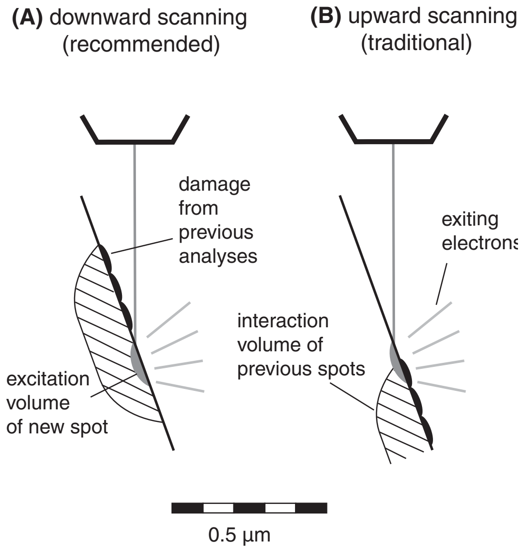

Fig. 1. Schematic diagrams comparing downward and upward scanning at 0.1 μm step size. (A) In downward scans, the excitation volume of the current spot occurs entirely within the interaction volume of previous spots, but avoids the intense surface damage due to earlier spots. (B) In traditional upward scans, the excitation volume partially avoids the interaction volume associated with previous spots, but involves surface material severely damaged by earlier analyses. The length of the excitation volume shown (0.15 μm) is based on our observations of the approximate step size at which major beam damage occurs (Fig. 4). To contain the figure within a reasonable area, the interaction volume shown is about 1/10th the size estimated by Prior *et al.* (1999).

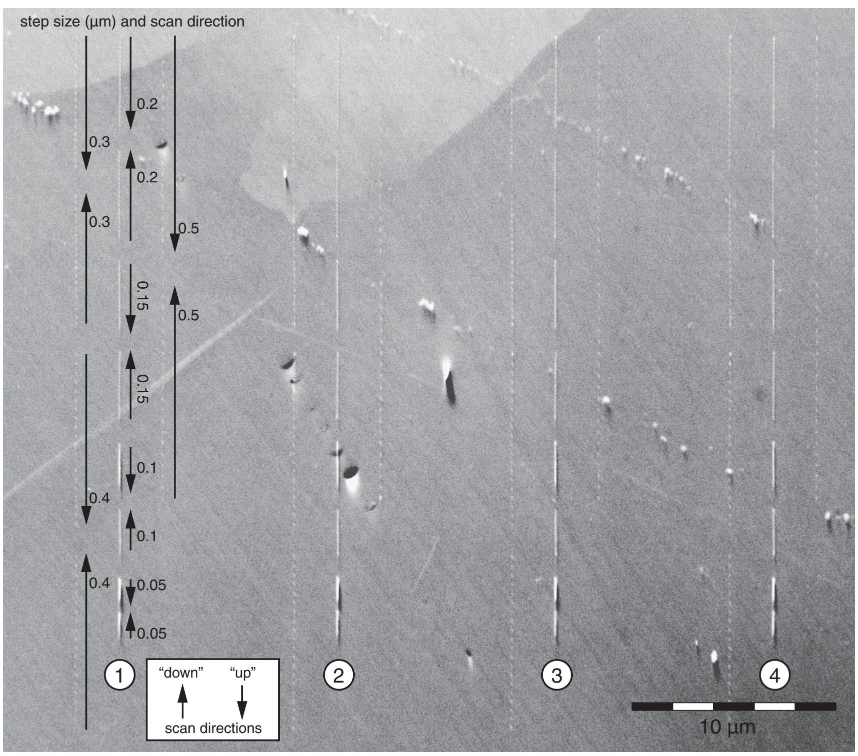

Fig. 2. Forescatter image (70° inclined surface) of a quartz grain showing beam damage induced by the analyses reported in Fig. 3. Four sets of line scans (circled numbers 1–4) were made for each direction and step size (numbered in black). The upward scanned lines at step sizes 0.05 and 0.1 μm are brighter and have greater topography (cast larger shadows) than their downward scanned counterparts indicating greater total damage. The figure is oriented as it appears on the SEM monitor with the actual bottom of the sample at the top of the image.

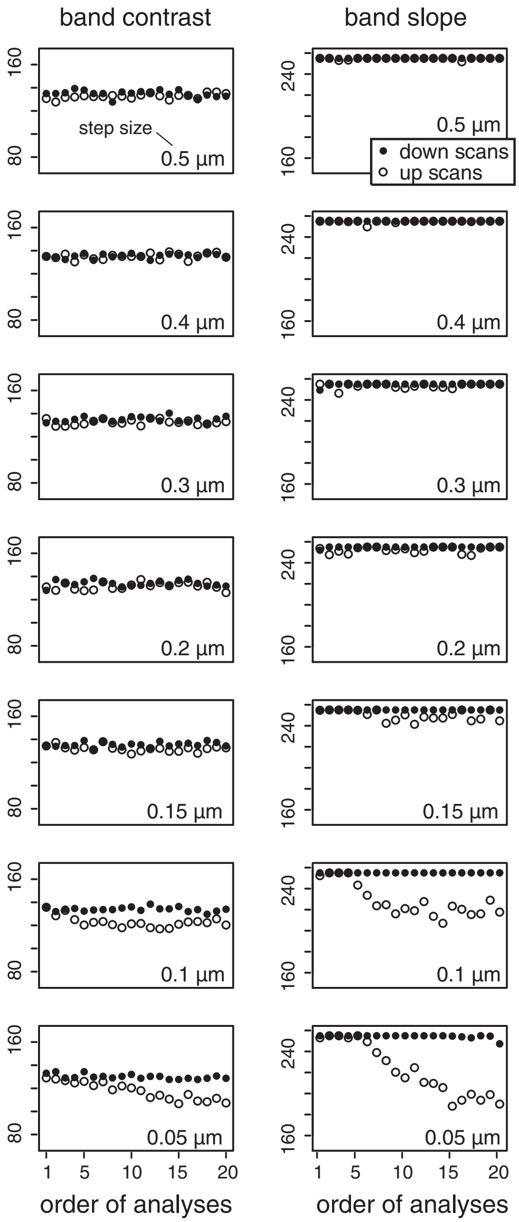

Fig. 3. Average band contrast and band slope values (y-axes) for sequences of 20 points scanned in upward (open circles) and downward (filled circles) directions. The first points analyzed are on the left of each plot. EBSP quality is relatively independent of scanning direction at larger step sizes. At smaller step sizes the first analyses are equivalent for each plot, but later analyses yield poorer pattern quality for upward scans.

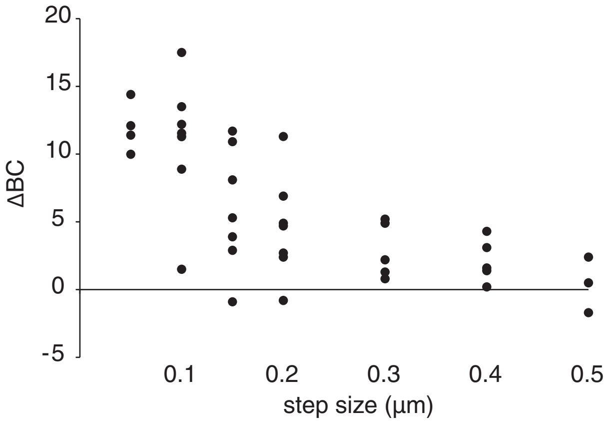

Fig. 4. Difference in average band contrast (BC) for line scans in the up and down directions ('ΔBC' = BC in down direction – BC in up direction) as a function of step size for line scans made on seven different grains. In all but three cases, line scans in the 'down' direction produced better band contrast. The advantage of scanning in the downward direction appears to coincide with the onset of detectable damage (between 0.4 and 0.5 μm step size, see Fig. 3) and increases with decreased step size. Note that the onset and degree of damage may vary significantly in other varieties of quartz.

the tilted sample (opposite the apparent direction that is typically shown on resulting screens and EBSD maps).

Materials and methods

We analyzed a thin section of Black Hills quartzite polished with colloidal Si. Black Hills quartzite is undeformed with a grain size of ~70 μm (Stipp & Kunze, 2008). We used the Zeiss field emission scanning electron microscope equipped with an HKL EBSD system at the University of Otago, New Zealand. Patterns were acquired at a working distance of ~18 mm, an accelerating voltage of 20 kV, 70° sample tilt, ~7 nA 'high' beam current at a Nitrogen gas pressure of 20 Pa. We used frame averaging of 1–4. Analysis times per spot were ~0.1 seconds. These settings are typical for EBSD analyses of quartz (Prior *et al.*, 1999; Trepmann *et al.*, 2007; Halfpenny, 2010; Menegon *et al.*, 2011; Billia *et al.*, 2013). We experimented with all of the above settings (with the exception of sample tilt) verifying that these conditions optimize pattern quality in our samples. Longer dwell times, for example, increase pattern quality for the first few analyses in a map or line scan; however, beam damage is increased in later analyses resulting in an overall decrease in pattern quality for a given map or line scan.

For each step size analyzed (0.05, 0.1, 0.15, 0.2, 0.3, 0.4 and 0.5 μm), four line scans were made in both upward and downward scanning directions (Fig. 2).Data from the first lines were discarded since it was found that the beam dwells on the first point of the first line for each set of lines. Band contrast and band slope values for the remaining lines (2–4) were averaged for each step size and direction to compare the results of upward and downward scans (Figs. 3 and 4). Band contrast and band slope are EBSP pattern quality factors that are derived from the Hough transformation and are thus independent of indexing routines. Band contrast and slope describe, respectively, the average intensity of Kikuchi bands relative to overall intensity of an EBSP, and the maximum intensity gradient at the margins of Kikuchi bands (Maitland &

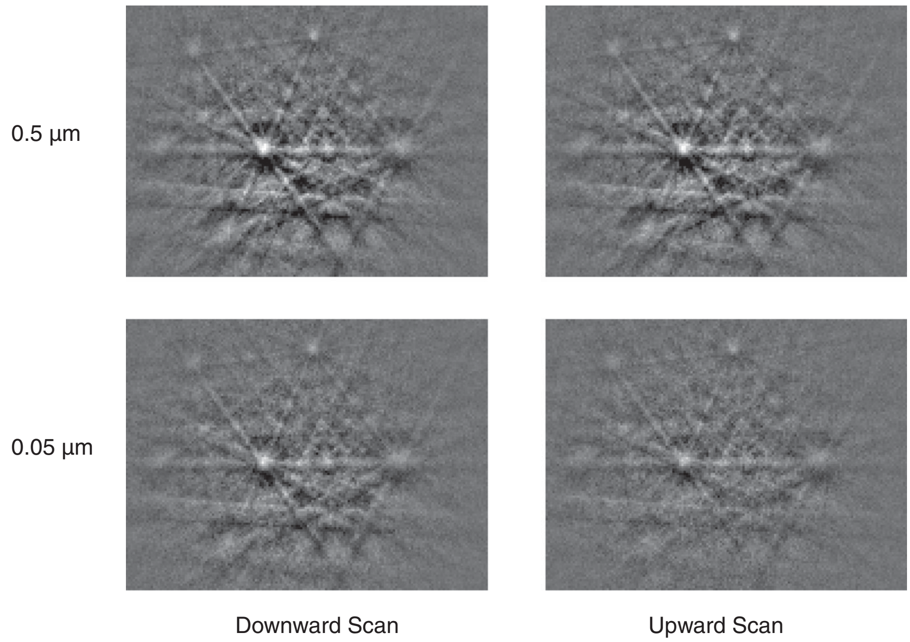

Fig. 5. Representative EBSP from the last point analyzed in four line scans. The first row (0.5 μm step size) shows no visible difference associated with the different scan directions. At 0.05-μm step size, significant pattern degradation is evident in both EBSP patterns but is less severe in the downward scan.

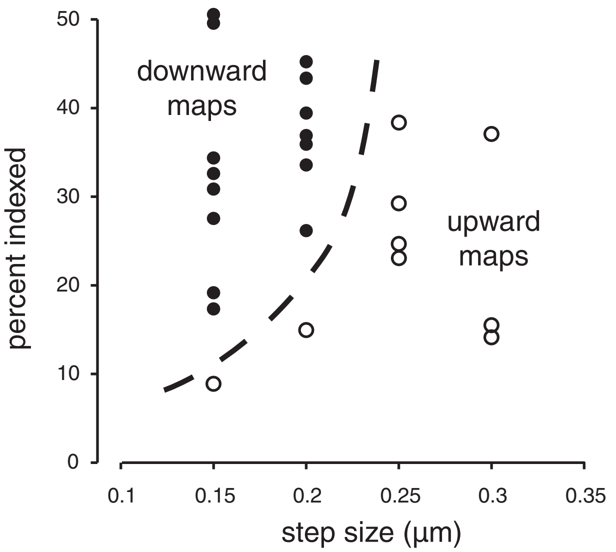

Fig. 6. Comparison of EBSD map indexing results from four experimentally deformed quartzite samples. The dashed line separates maps scanned in the upward and downward directions. SEM set up and indexing choices were similar in the various maps (e.g. all involved recognition of 6–7 band edges). Each map contains 10 000–250 000 analyses. Only relative differences in per cent indexed (y-axis) are notable since differences in indexing routines can dramatically affect these numbers (these maps used the indexing engine in the Flamenco software; indexing rates would be much higher using the AZTEC software). Better indexing and smaller step sizes are possible when scanning in the downward direction.

Sitzman, 2007). Band contrast and band slope values are scaled to the byte range (0–255) with higher values indicating higher quality.

We focused our study on line scans in order to minimize the potential effects of beam drift, which can occasionally occur at significant rates relative to the line-by-line upward or downward velocity of high-resolution EBSD maps. To test our results in a mapping application, we produced several EBSD maps on samples of experimentally deformed Black Hills Quartzite. These samples were deformed at 900°C at high differential stress (200–900 MPa; i.e. in regimes 1 and 2 of Hirth & Tullis, 1992).

Results

Pattern quality did not significantly deteriorate in downward-scanned lines at any step size (Fig. 3). Upward-scanned lines however showed major deterioration in pattern quality at small step size (Fig. 3). The advantage of downward scanning appeared at ~0.4 μm step size and increased as step size decreased (Figs. 3 and 4). Representative EBSP are shown in Figure 5.

The higher quality EBSP resulting from downward scanning result in significant improvements in the indexing of EBSD maps. Figure 6 summarizes the results of maps made in both directions at a variety of step sizes in experimentally deformed Black Hills quartzite (grain size 1.3–1.5 μm). The mapped samples are highly strained resulting in low overall indexing rates, but downward scanning still proved to be superior to upward scanning. At step sizes of 0.15 and 0.2 scanning in the downward direction improved indexing by a factor of 2 to 4 and was equivalent to or better than indexing of upward maps made at step sizes of 0.25 and 0.3 μm (Fig. 6).

A physical change occurs at each point where the electron beam intersects the sample's surface (Fig. 2). The damaged points cast shadows in tilted samples (Fig. 2) indicating a transformation of quartz to some less-dense molecular arrangement. The damage points induced by our analyses have diameter ~0.1 μm and at small step sizes they merge into continuous lines. The magnitude of damage involved in these lines is smaller in downward scans than upward scans (Fig. 2).

Discussion

We attribute the majority of the deterioration of EBSP in our analyses to physical changes in quartz occurring at the point where the electron beam intersects the sample surface. We propose that upward scanning is inferior to downward scanning at small step sizes because previously damaged spots occur within the excitation volume during upward scanning (Fig. 1). This results in re-damaging of earlier damaged points and causes increased physical damage in the upward-scanned lines. The excitation volume involved in downward scanning better avoids earlier beam damage and thus provides better quality EBSD maps (Fig. 1).

Given the clear benefits of scanning in the downward direction, commercially available EBSD acquisition packages should be modified to allow the flexibility of scanning in the recommended downward direction. A scan rotation on the SEM of 180° is not a satisfactory substitution since the resulting orientation data are incorrect by a 180° rotation and, when combined with tilt and dynamic focus corrections employed during EBSD analyses introduce significant distortion in most SEMs we have tried. This distortion is only avoided when scan rotation is effected by a physical rotation of the scan coils – a facility not available in most modern SEMs.

Acknowledgements

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References

Bestmann, M. (2012) High Resolution EBSD mapping of quartz deformation microstructures. In *10th EMAS Regional Workshop on Electron Probe Microanalysis of Materials Today*. Padua, Italy.

Billia, M., Timms, N.E., Toy, V.G., Hart, R.D. & Prior, D. (2013) Grain boundary dissolution porosity in quartzofeldspathic ultramylonites: implications for permeability enhancement and weakening of mid-crustal shear zones. *J. Struct. Geol.* **53**, 2–14.

Halfpenny, A. (2010) Some important practical issues for the collection and manipulation of Electron Backscatter Diffraction (EBSD) data from geological samples. *Journal of the Virtual Explorer* **35**, doi:10.3809/jvirtex.2011.00272.

Hirth, G. & Tullis, J. (1992) Dislocation creep regimes in quartz aggregates. *J. Struct. Geol.* **14**, 145–159.

Humphreys, F.J. & Brough, I. (1999) High resolution electron backscatter diffraction with a field emission gun scanning electron microscope. *J. Microsc.* **195**, 6–9.

Maitland, T. & Sitzman, S. (2007) Electron backscatter diffraction (EBSD) technique and materials characterization examples. *Scanning Microscopy for Nanotechnology: Techniques and Applications* (ed. by W. Zhou & Z.L. Wang), pp. 41–75. Springer, New York.

Menegon, L., Nasipuri, P., Stünitz, H., Behrens, H. & Ravna, E. (2011) Dry and strong quartz during deformation of the lower crust in the presence of melt. *J. Geophys. Res.* **116**, B10410.

Ohfuji, H., Boyle, A., Prior, D. & Rickard, D. (2005) Structure of framboidal pyrite: an electron backscatter diffraction study. *Am. Mineral.* **90**, 1693–1704.

Prior, D.J., Boyle, A., Brenker, F., *et al.* (1999) The application of electron backscatter diffraction and orientation contrast imaging in the SEM to textural problems in rocks. *Am. Mineral.* **84**, 1741–1759.

Prior, D.J., Mariani, E. & Wheeler, J. (2009) EBSD in the earth sciences: applications, common practice, and challenges. *Electron Backscatter Diffraction in Materials Science* (ed. by A.J. Schwartz), pp. 345–360. Springer Science, New York.

Schwartz, A.J., Kumar, M., Adams, B.L. & Field, D. (2009) *Electron Backscatter Diffraction in Materials Science*. 2nd edn. Springer, New York.

Schwarzer, R.A., Field, D.P., Adams, B.L., Kumar, M. & Schwarz, A.J. (2009) Present state of electron backscatter diffraction and prospective developments. *Electron Backscatter Diffraction in Materials Science.* 2nd edn., pp. 1–19. Springer, New York.

Stipp, M. & Kunze, K. (2008) Dynamic recrystallization near the brittle-plastic transition in naturally and experimentally deformed quartz aggregates. *Tectonophysics* **448**, 77–97.

Trepmann, C., Stöckhert, B., Dorner, D., Moghadam, R., Kuster, M. & Roller, K. (2007) Simulating coseismic deformation of quartz in the middle crust and fabric evolution during postseismic stress relaxation — an experimental study. *Tectonophysics* **442**, 83–104.