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RECENT FEATURES IN EBSD, INCLUDING NEW TRAPEZOIDAL CORRECTION FOR MULTI-MAPPING

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Educated from University of Århus in 2001 with a MSc-degree in Microtectonics, using EBSD as a tool to investigate the role of Dauphiné twins in quartz tectonites. This was followed by a PhD in Metamorphic Petrology from the Norwegian University of Technology and Science in 2007. In recent years, Sorensen has been lucky to be part of development of improved EBSD data at the Norwegian University of Technology and Science in close collaboration with its sample preparation laboratory, developers of the NORDIF acquisition software. The developments have provided significant improvements in the acquisition, which makes application of EBSD in geology much better. Especially the distortion correction is to him a small revolution for EBSD on coarse-grained samples. EBSD has been implemented to investigate classical problems in structural geology, topo- and epitaxial mineral growths and to model optical properties of minerals. He hopes that he can continue his future research combining his interest in analytical techniques, mathematics, physics, crystallography, thermodynamics, programming and field geology to solve scientific and analytic problems both in geology and other scientific fields. So far, his research has led to 18 published journal papers and many papers are awaiting their publication.

1. ABSTRACT

EBSD has been technique in metallurgy for several decades and has proven to very useful for describing microstructures and textures in metals, ceramics and rocks.

One of the focusses in EBSD development has been speed as one of the main applications is quality control in metal production. Offline EBSD brought even faster data collection because a large part of the collection time during online EBSD acquisition is due to indexing of the patterns, which is slower than the acquisition of the patterns. This means that the indexing sets the time limit for data acquisition, something that is not ideal for product control, in-situ experiments and multiphase samples such as geological samples or ceramics. In addition, the offline data gives the possibility to check patterns of noisy areas to tell whether noise removal procedures remove noise and not correct observations, by directly comparing patterns in noisy regions. These observations are essential in judging if procedures such as grain reconstruction are correct. Patterns in unindexed regions are in some cases weak versions of patterns from the neighbouring indexed areas. Hence, it would be practical if these unindexed areas could be included in the indexed points, as normal grain determination does not have any guidance whether to include these areas in the grains or to leave them as unknown areas in the final EBSD map. One promising path towards overcoming this is to improve the pattern quality prior to indexing as traditional pattern treatment does not always handle static and dynamic background subtractions well, giving rise to artefacts, such as rings originating from different position of the diffuse scattering in the background averaged EBSP and the pattern in each pixel of the EBSD dataset. The positions of the diffuse scattering part of the EBSP varies as a function of several parameters, including the x-y of the pixel in the map, the Z-number of the phase, artefacts due to charging and topography. This is to some extent handled by dynamic background subtraction procedures, but the dynamic background subtraction cannot handle the detector noise so this has to be corrected without at the same time applying a wrong diffuse scattering noise.

Because EBSD on geological samples requires mapping of large areas, stitching of EBSD datasets is necessary. However, geometric trapezoidal distortions are common due to the tilting of the sample. This means that the maps do not fit completely together. The traditional solution has been to map very small areas to reduce the geometric distortion, but for coarse-grained samples, this is a bad approach as this gives too few different orientations, which can be used to match the maps together. Therefore, maps needs to be collected at lower magnification, giving larger amount of geometric distortion. For this purpose, an online correction procedure has been made such that the original maps have very little geometric distortion even at magnifications below 30x. This solves the problem of stitching when working with large samples; however, there are still minor issues with accuracy of orientation measurements (< 0.5° across map) and uneven illumination, which has to be considered when collecting EBSD at low magnifications.

2. RESULTS AND DISCUSSION

2.1. Offline versus online EBSD, the main differences

During the early period of EBSD, data acquisition was mainly done in the so-called online mode, meaning that the electron backscatter pattern (EBSP) were collected, indexed and then deleted continuously during data acquisition. A successful online EBSD data acquisition experiment depends on a good setup prior to data collection. This setup starts with adjustment of the electron microscope to obtain good diffraction patterns, acquisition and calibration of geometry, definition of crystal structures for the phases in the sample and the settings for background subtraction and pattern determination scheme. If everything is set up correctly then indexing will work and correct phase and orientation determination will reach 90 % or higher. However, this is with the 'ideal' setup, something that is not always the case. If the setup is wrong and results turns out bad then the analysis in the SEM has to be redone. In contrast, the offline EBSD relies in the first instance on collecting good patterns in the SEM. Patterns are streamed to a disc and then indexed on a separate computer afterwards, this means first of all for a geological sample that data collection time is reduced [1-3], at least by one to two orders of magnitude. Secondly indexing can be redone without running data acquisition again. NTNU implemented off-line EBSD in 2007 [1]. Figure 1 summarizes the workflow in off- and online EBSD.

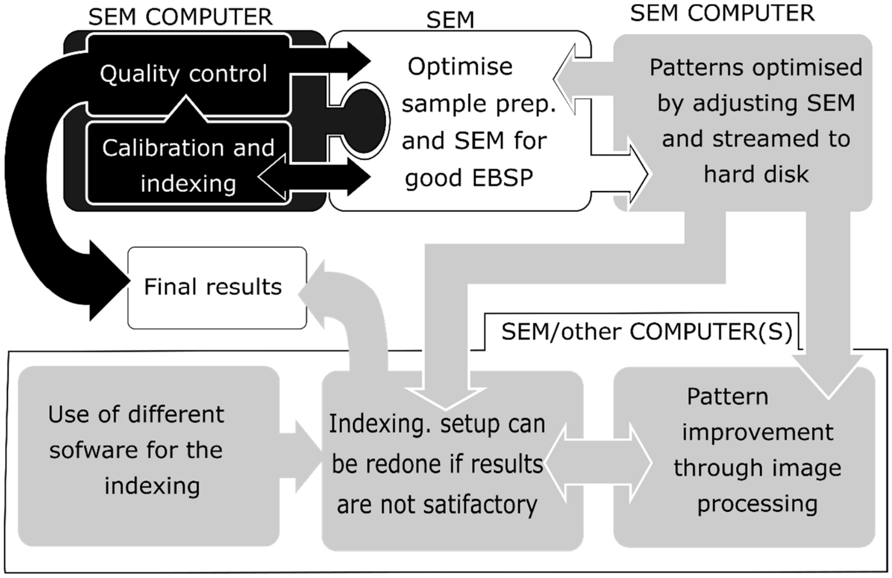

Figure 1. Comparison between off- and online EBSD procedure. Online procedure in black offers a direct solution; however, it is tied with the SEM. Offline procedure in grey opens a range of options after data acquisition on the SEM.

To obtain data fast in the SEM using online EBSD a compromise has to be made with the quality in the indexing, as the indexing is the slow part of the data collection process. This means that one has to be a little less precise and accurate when it comes for example to Hough transform setup. This can work quite well for simple materials such as metal samples, though the precision of the orientation determination is reduced from 0.1° to about 0.3°. In complex materials like geological samples the fast approach is possible, however this will be likely to introduce artefacts in the dataset that originate from pattern binning, low angle Hough resolution and the number of bands used for indexing. This relates to the complexity of geological sample in terms of both the number of phases and their complex crystal structures. Because of the low symmetry of minerals, compared to the cubic metals, more bands are needed to determine the orientation and also to deal with pseudo-symmetry. Pseudo-symmetry occurs when the symmetry of a phase is close to a higher symmetry. In the EBSP this is reflected by the majority of the bands are covered by the higher symmetry, while weaker bands reflect the lower symmetry. This can cause misindexing because orientations that are symmetrically equivalent in the higher symmetry are misindexed. A well-known example of this is Dauphiné twins in quartz, which represents a 120° rotation about the c-axis. Quartz is trigonal and pseudo-hexagonal, meaning that if the right bands are not detected then the Dauphiné twin orientations cannot be distinguished and misindexings in the form of false twins occurs (Fig. 2). By selecting the correct reflectors in the crystal structure input the false twins can commonly be avoided as long as the pattern quality is good enough.

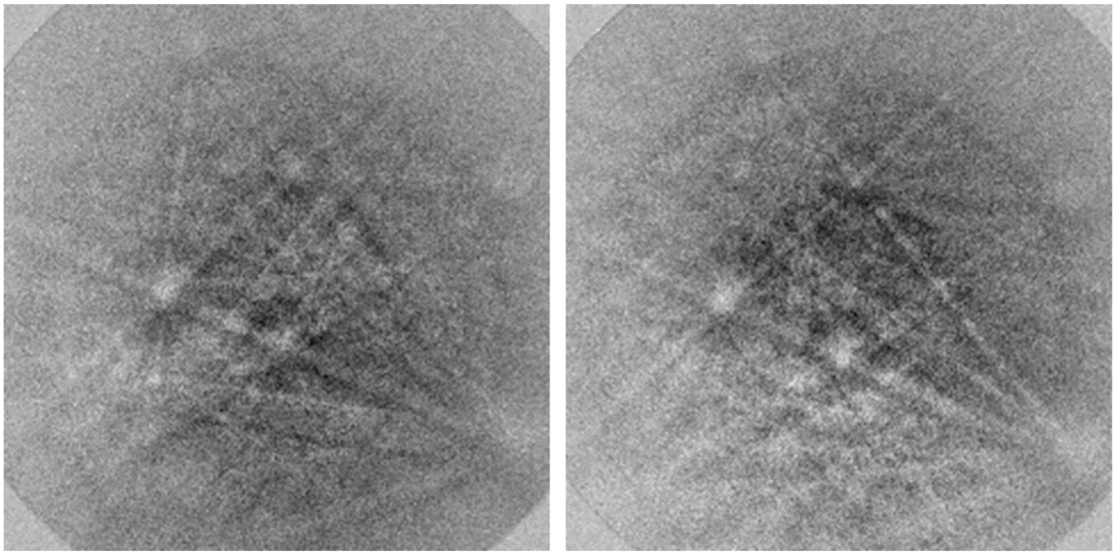

Figure 2. EBSP of two orientation of quartz across a Dauphiné twin boundary. Many bands are common, except broad bands in the background that reflect the true trigonal symmetry of the quartz Laue group.

Single/few pixel twins are commonly removed as they are assumed misindexed where patterns are noisy for example due to dirt particles or holes in the sample surface. With online EBSD there is no way of checking whether this assumption is correct. However, with offline EBSD it is possible to check if such assumptions are correct by inspecting the EBSP of the suspected misindexing (Fig. 3). Contrastingly, areas larger than 2 - 3 pixels are true twin and should not be changed during post processing. EBSD maps commonly also contain unindexed points in the map. Single pixel missing are normally not a problem as they can easily be interpolated from the surroundings, however, commonly there are also larger areas that need to be either interpolated from neighbouring grains or maintain unindexed areas. Offline EBSD offers the opportunity to investigate this (Fig. 4). Here there is a five pixel wide unindexed area between tow calcite grains. By EBSP inspection, it is revealed that the quality of the EBSPs gradually worsen toward the unindexed region, however, weak versions of the patterns of the grain are observed in the unindexed region (Fig. 4). In this case, it is therefore a reasonable solution to interpolate the grain boundary to the middle of the unindexed region (Fig. 4). Since the unindexed regions have weak versions of the indexed areas, it might be an even better solution to do pattern improvement and then re-index the dataset (Fig. 4). Another example misindexed phase along grain boundaries, in this case, false dolomite occurs along calcite-calcite grain boundaries, due to the similarity of the EBSP of calcite and dolomite (Fig. 5). By looking at the EBSP it is revealed that the dolomite is indeed misindexed as the EBSP are just mixtures of the patterns of the neighbouring calcite grains.

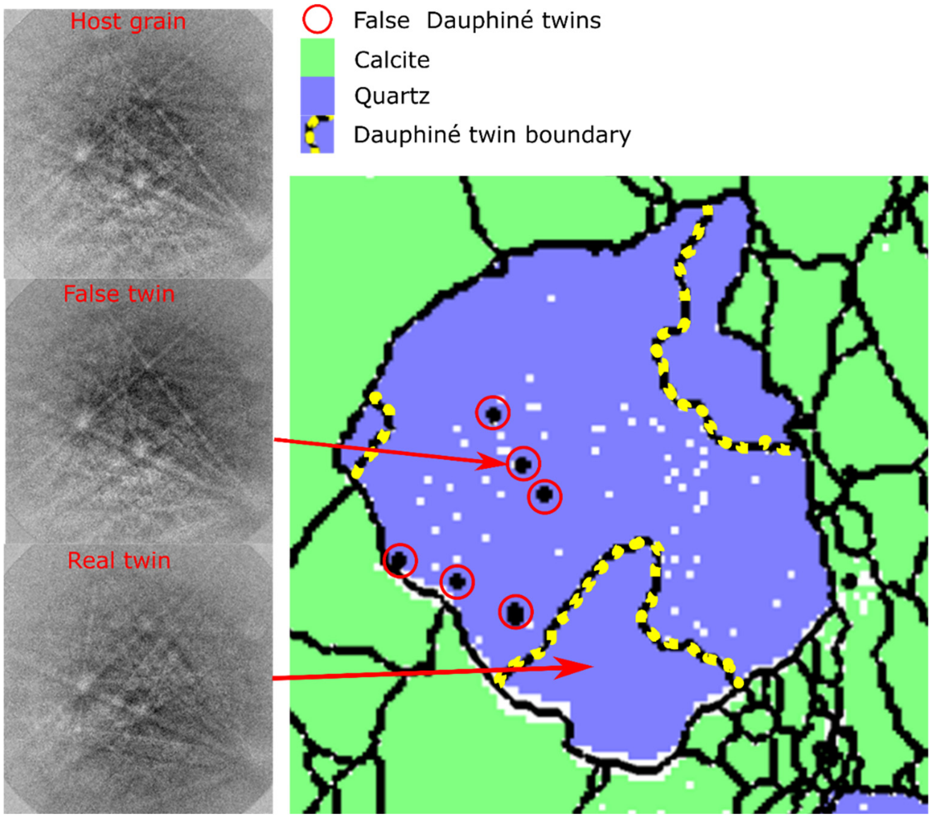

Figure 3. Distinction between false Dauphiné twin and true Dauphiné twin by pattern inspection. False twin occurs when pattern quality reduces and is sensitive to background subtraction and Hough settings.

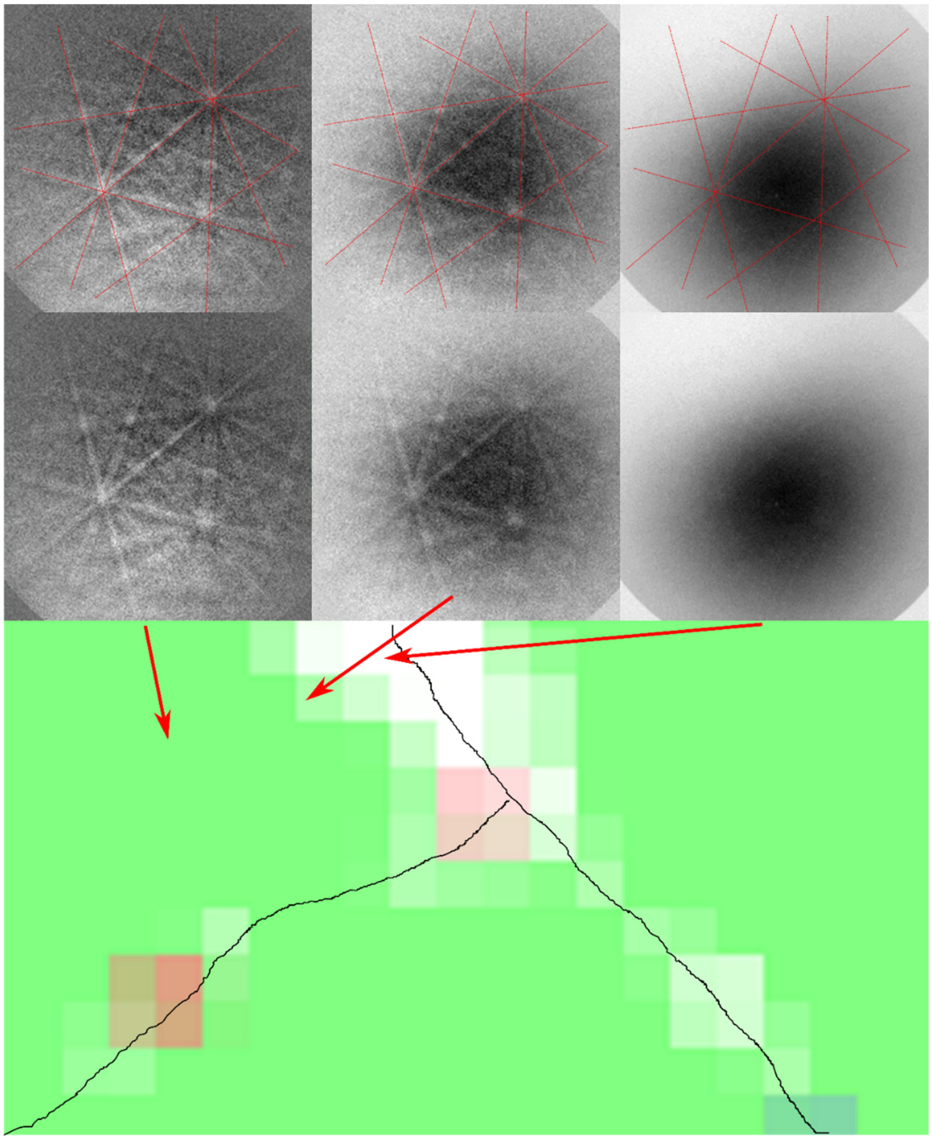

Figure 4. EBSP inspection in unindexed areas. Green is calcite, the lighter the colour the lower confidence index. Reds are false dolomite (see Fig. 5 for discussion). EBSP pattern quality worsen as we enter the unindexed area, but the same diffraction pattern is visible behind all the noise.

Coarse-grained materials are hard to handle in EBSD because running at high magnifications only show a few grains or even only parts of one grain, something that makes automatic stich near impossible. Instead samples needs to be mapped at lower magnification. This brings along some artefacts into the raw EBSD dataset. One significant effect is uneven illumination, which is also visible in TSL-OIM image quality (IQ) maps. IQ is very commonly used to differentiate regions that should be removed from the indexed parts, and hence this is very unfortunate. Figure 6 displays maps indexed using different setups on the same offline EBSD dataset. In the first instance, only a standard static background subtraction was applied. The IQ-map reveals

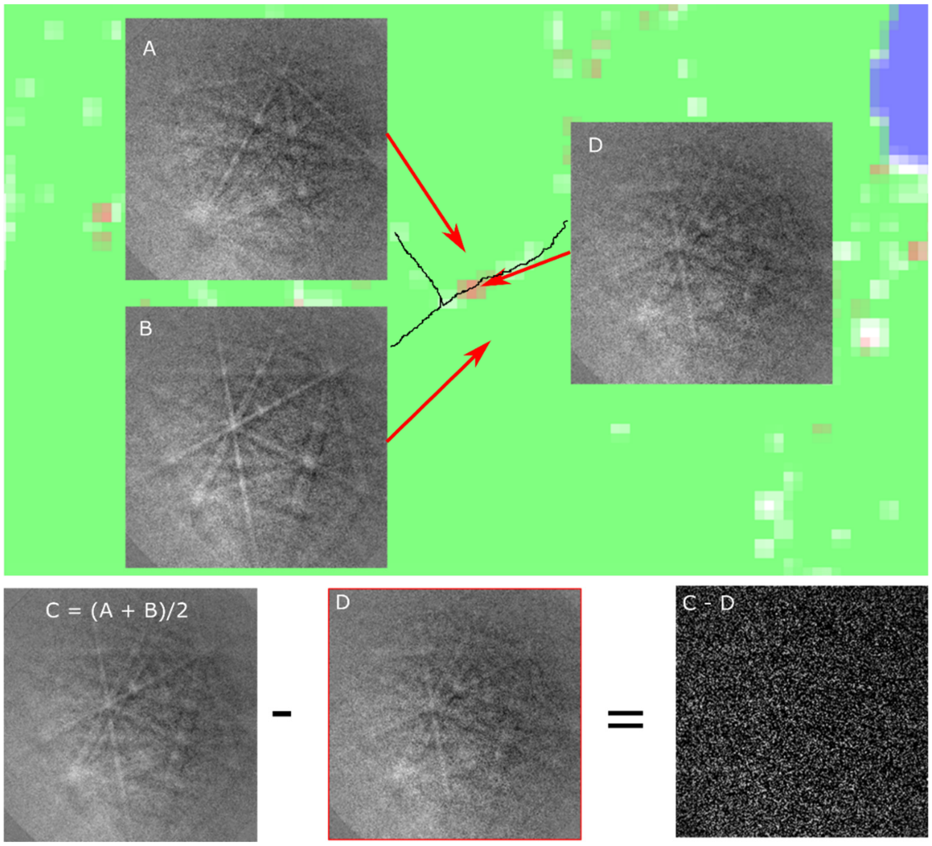

Figure 5. False dolomite recognised as the sum of two neighbouring calcite grain EBSP (A and B) as the average of A and B is the same as the “dolomite pattern” in D.

that the uneven exposure does not allow for use of EBSP-IQ as a discriminator between good and bad patterns as the shades from the uneven exposure creates a shading artefact in the IQ values with a bright centre in the middle of the map (Fig. 6). This is significantly improved by adding a dynamic background subtraction in the correct way, however, the effects are still visible though the better background procedure allows for a more strict procedure of indexing and areas with no patterns are disregarded by the indexing (Fig. 6). By applying advanced image analysis (KikuchiPy; H.W. Ånes, unpublished code) to the patterns prior to indexing the shading effect is completely vanished and the IQ seems to reflect the crystallinity of the phases quite well with higher IQ for quartz than albite and k-feldspar (Fig. 6). This procedure is promising but still needs some improvements as it introduces artefacts in the form of washing out of grain boundaries and perhaps over-improvements of areas with no patterns (Fig. 6). Phase maps agree in the main part, however there are subtle differences and in some areas it seems that the map is as good as the ones with corrected patterns (Fig. 6). More work will be done to look further into the details of this. Another thing that is possible with the offline-data is to use different approaches, both commercial and freeware such as AstroEBSD [4] and EMsoft [5] which may be more suitable in some cases.

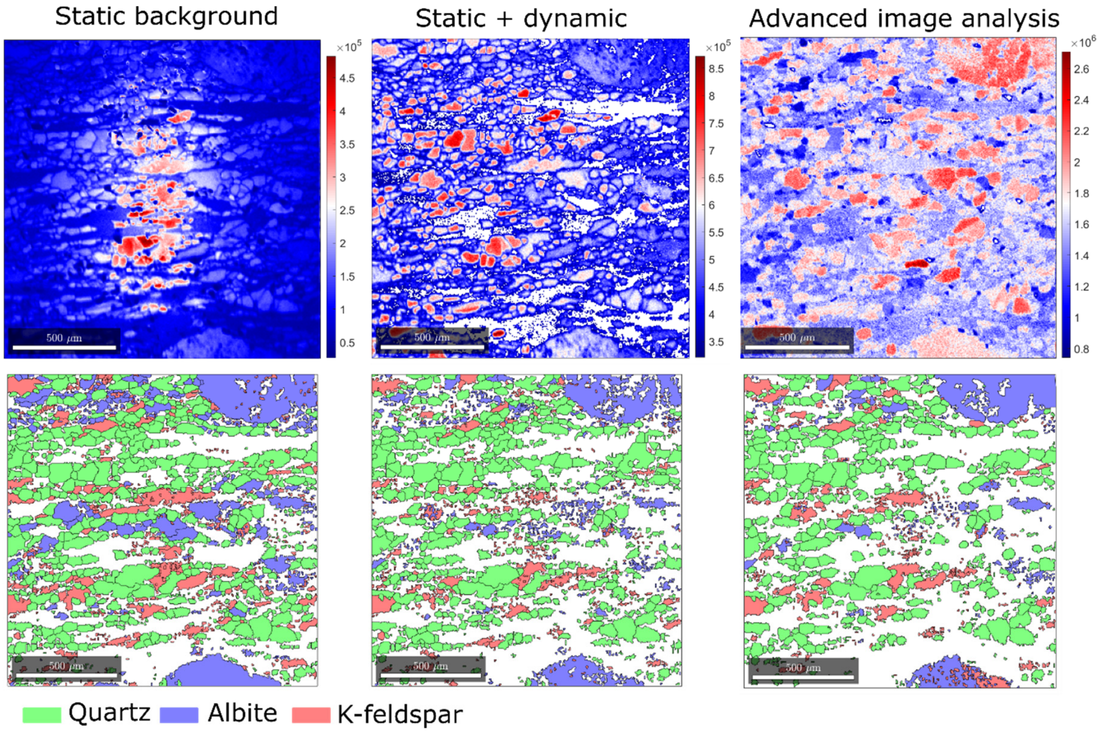

Figure 6. Comparison of different indexing on the same dataset. Upper row is EBSP IQ from the TSL-OIM software. Lower row is the phase map. Left column is static background only, middle with TSL-OIM dynamic background and right is with advanced image analysis EBSP improvement in KikuchiPy (H.W. Ånes, unpublished code). See text for discussion.

2.2. EBSD acquisition improvements

Although a lot can be done in offline mode there is still new and unexploited ways of improving the raw data beforehand. The low magnification EBSD maps has big geometric trapezoidal distortions due to the tilt of the sample, that if not corrected will make it impossible to do good stitching. At low magnification, these effects are smaller, but still present. If maps are corrected for this after acquisition then the x- and y-coordinates are no longer equally spaced, giving problems with many EBSD processing algorithms. It is therefore more suitable to have a correction before acquisition. This was implemented in the NORDIF v3 data acquisition software, by correction of the electron beam and making it move in a quadratic grid. The calibration procedure uses a square grid on a Si-single crystal standard at different working distance, acceleration voltages and magnifications (Fig. 7). If performed correctly this gives undistorted maps even at low magnifications (Fig. 7) and these can then be stitched. Other possibilities that lie within the acquisition part is to make uses of the options that lie in the increasingly advanced EBSD cameras. As an example, the auto exposure functionality could reduce some of the uneven illumination problems discussed in the previous section and in addition this is also interesting for samples with high variation in phase average Z-number, where the different phases therefore would give totally different amount of signal intensity for the same exposure time. In addition, background subtraction schemes could be improved to reduce the background related artefacts in EBSPs.

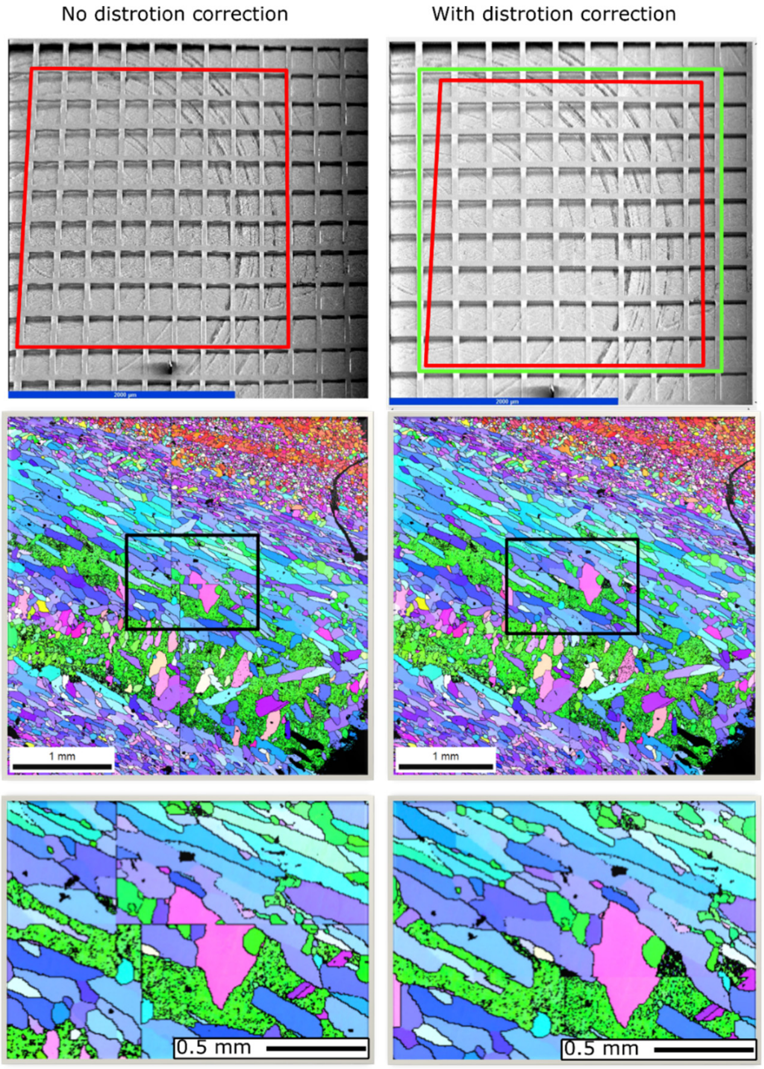

Figure 7. Correction procedure for removing the trapezoidal distortion. Column to the left is without the correction, clear visualised by the square grid not being square in the image. This also gives distorted images of the samples, such that the EBSD maps cannot be stitched properly. In the column to the right distortion correction has been applied and the calibration grid is square and EBSD maps are undistorted and hence stich easily together.

2.3. *Processing of indexed EBSD data in MTEX toolbox*

Commercial software that commonly follow the EBSD acquisition software can do much of the data interpretation, which is commonly used, however it is limited in some areas and if users want to do specialised solutions then a more script-based method is more appropriate. The MTEX toolbox is a strong toolbox for EBSD and texture analysis that has a rigorous statistical treatment of orientation and misorientation data and also implements an unbiased orientation colour scheme [6-11]. MTEX is freeware; however, it works from MATLAB so one needs a MATLAB license. Further, the user threshold is higher than the proprietary ESBD software. However, MTEX is much more flexible than any GUI software could ever be and the ODF and MDF functionality is comprehensive. In additionally crystallographic phenomena can be treated both with respect to grain boundaries and physical property calculations within the full realm of point groups. The flexibility also makes things like investigating twins, topo- and epitaxial relations easier.

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