Orientation microscopy with fast EBSD

R. A. Schwarzer*¹ and J. Hjelen²

Automated backscatter Kikuchi diffraction in the SEM is about to become a tool for process and quality control. Mandatory requirements for these applications are a high speed and provisions to enable re-examination of the results at any time. Means and motivation for high speed electron backscattering diffraction are discussed. Separate acquisition and store of pattern sequences in an unprocessed data format, followed by offline calculation of the grain orientations, has many advantages over online orientation microscopy. With actually >1000 acquired patterns per second it is significantly faster than conventional electron backscattering diffraction. The data can be examined again using the original backscatter Kikuchi sequences, and the presence of a priori unknown phases can be checked for. Online pattern solving, as a dual task, is optional.

Keywords: Crystallographic texture, EBSD, ODF statistics, Orientation microscopy, SEM

Introduction

Electron backscattering diffraction (EBSD) has become the standard technique for orientation microscopy and texture analysis on bulk polycrystals at a grain specific level. Reasons include the wide availability of SEM instruments, the ease of operating commercial EBSD systems, the high speed of data acquisition and the awareness of texture being an import material characteristic. Conventional EBSD systems are based on pattern acquisition and simultaneous online evaluation. These systems discard the patterns once they have been indexed, making it impossible to repeat pattern solving with exactly the same raw data set if the results, for some reason, turn out to be unsatisfactory. When performing online pattern indexing, results depend on an accurate system calibration. All present phases must be known a priori. Until 2000, speed of online pattern acquisition with an analog camera as the detector was limited by the video frame rate to less than 30 orientations per second so that pattern solving could be conducted in parallel.¹ There was little motivation to further increase the speed of the indexing software.

A high speed of measurement is not only a value by itself in that the sample throughput of the SEM is improved, but is indispensable for dynamic experiments, for the study of sensitive materials that degrade under the electron beam or in vacuum, and for three-dimensional orientation microscopy using focused ion beam sputtering for serial sectioning. A short time of measurement will also alleviate some difficulties with long term stability of the SEM. The application of EBSD is not limited to investigate local texture of small areas, but can compete well with X-ray diffraction pole figure measurement to reveal global texture. Similarly large areas can be examined with digital beam scan provided that the beam is dynamically focused when scanning across the steeply tilted specimen surface.¹ However, the demand for a significant grain statistics can hardly be met with a slow EBSD system.

In X-ray diffraction pole figure inversion with discrete methods, the Euler space is usually divided in cubic cells of 5° increments in ($\phi_1$, $\Phi$, $\phi_2$). For simplicity the distortion of the Euler space is not considered. In the case of cubic crystal and triclinic sample symmetry the orientation distribution functions (ODF) is constituted by $(180 \times 180 \times 90)/(3 \times 125)=7776$ cells. A good cell statistics can easily be obtained with fast EBSD even for a flat texture to reach ~100 counts per cell in the average. Steady convergence of the texture index is an indication that the number of measured grains is sufficient. For a single crystal, one orientation is of course sufficient to measure, for a sharp texture ~1000 single orientations may be appropriate. Electron backscattering diffraction yields grain orientation with an accuracy of ~0.5° so that the number of cells could be increased by three orders of magnitude to exploit the full information. Cell statistics, in particular if subtle texture components are of interest as for instance at the onset of recrystallisation, will then demand for a huge number of measured orientations far beyond imagination. X-ray diffraction pole figure measurement, however, is hardly better off as a consequence of limited dynamics, non-linearity and drift of the detector, limited count statistics in the pole figures and numerical instabilities.

A sample area of 1 mm² will contain 10⁶ grains of an average size of 1 μm². Since the ODF only accounts for the density of orientations disregarding the location of the grains in the sample, the measured points can be set at random positions for calculating the ODF from large areas. The full potential of EBSD is, however, only exploited by orientation microscopy and in particular by orientation stereology² whereby the orientation and the location of the grains are considered. This approach will need to measure on a fine grid so that several raster...
points fall in every grain. The study of intragranular structure and grain boundary analysis demands for an ever increasing high number of measured orientations. In conclusion, a reliable statistics and orientation stereology are a strong impetus on developing high speed EBSD.

How to increase speed

There are several means to increase speed. Mesh refinement\(^1\) is an effective approach whereby in a first step an overview of the microstructure is obtained by scanning on a coarse raster grid with a step size slightly smaller than the diameter of the smallest grains. If intragranular structure is of no concern, it is in principle sufficient to measure the orientation of each grain only once. Therefore, a refined mesh is overlaid of half the step size in the second and of quarter step size in a third loop, but only patterns on those intermediate grid positions will be acquired and evaluated where orientations between neighbouring nodal points on the preceding grid differ by more than a preset value. Hence, measurements on the refined meshes in the following passes are concentrated along grain boundaries. Those grid points which could be skipped from measurement are assigned the average orientations of their neighbours. The limitations of this approach are set by small twins that might easily be overlooked in their matrix grains and by a wide distribution of grain size when mesh refinement becomes inefficient since the starting mesh grid has to be rather fine in order to observe the smallest grains.

A major step ahead has been achieved by improving the pattern solving software. The speed at which a band can be localised in the pattern scales with the number of pixels in the pattern and the number of \((r, \theta)\) points in the discrete Radon transformation. An unwanted side effect of simply coarsening the backscatter diffraction patterns and, in conformity, reducing the size of the transformation is the much reduced angular resolution. Depending on pattern quality, a single band can be located in a pattern of 100 by 100 pixel at a typical deviation of \(\Delta \theta = 1.5 - 2^\circ\), whereas after coarsening to one fourth of this size the angular uncertainty will be twice as large or worse. Hence, the error limits in the indexing routine have to be widened in order to account for this inaccurate band localisation. As a consequence of this inaccuracy, fewer detected bands may be indexed unambiguously. Sometimes this leads to wrong orientations being found and the fraction of points indexed with high confidence may decrease significantly. It is worth mentioning that the grain orientation is calculated as a best fit from the locations of the \(n\) consistently indexed bands. Therefore, its mean error is by \(1/n^{1/2}\) less than the average band deviation. Gross coarsening the patterns and Radon transformations and, at the same time, allowing a lower reliability of orientation data are appropriate means for increasing speed if only for obtaining a first impression of the texture and of the quality of sample preparation.

The general Radon transform and analysis of band profiles\(^4\) is better suited to extract band positions from backscatter Kikuchi patterns than the 'modified Hough transform with butterfly mask'\(^4\) (Table 1) since the motif, that is a straight line in the pattern, can be processed before transformation. The speed of numerical calculations scales with typically two-thirds of the increase in CPU clock rate. Parallel processing of the Radon transform in several threads on a multicore CPU considerably speeds up band localisation. Threaded applications incur little additional performance penalty on single processor machines. Further progress can be made with GPU coprocessor graphic boards. The high and uneven background in backscatter Kikuchi patterns can be continuously corrected in the camera by subtracting a flat background image pixel by pixel.

A low sensitivity of a detector can be offset by a high beam current only to some extent. Hence a detector of high sensitivity is of key importance for obtaining a high speed in pattern acquisition.\(^5\) An enormous advance in the performance of image sensors has been achieved recently. Although CMOS sensors are superior in speed, charge coupled device (CCD) sensors still have a higher quantum efficiency and are more sensitive. Recent electron multiplying CCD sensors promise some advantages at very low light levels. A (proximity focus) image intensifier between the phosphor screen and the camera can be used to increase overall sensitivity of the detector.

Pixel binning on the sensor chip is a well proven means to increase sensitivity and speed (each individual photo sensor on the chip array as well as each image point is called a pixel. Binning is a way to combine charge from neighbouring pixels to a super pixel before readout). If \(n\) abutting pixels are bundled together on the chip during the readout procedure, the current to the preamplifier is increased \(n\)fold so as to be raised above the noise floor. Furthermore, the number of pixels per image to be transferred to the computer is also reduced by \(1/n\), thus speed is likewise increased. In principle, a dedicated sensor chip with a coarse array of pixels and correspondingly increased preamplification would be superior. The filling factor and capacity per pixel would be higher with the advantage of higher sensitivity and dynamic range. To further increase readout rate, CCD arrays are divided in several sectors and each sector has its own readout port.

The camera interface is an integral component of an EBSD system and warrants discussion. Frame grabbers, as common with conventional analog cameras and with

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**Table 1** Comparison of Radon and Hough transformation for band localisation

<table>
<thead>
<tr>
<th>Radon</th>
<th>Hough</th>
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<tbody>
<tr>
<td>Motif to be transformed into</td>
<td>Point ((r, \theta)) in Radon space</td>
</tr>
<tr>
<td>Implementation sequence of the loops</td>
<td>Straight line in the diffraction pattern</td>
</tr>
<tr>
<td>Advantage Application</td>
<td>(\rho, \phi, x, y)</td>
</tr>
<tr>
<td>Grey tone images, a universal method</td>
<td>Sinusoidal curve in Hough space</td>
</tr>
<tr>
<td>(e.g. computer tomography)</td>
<td>(x, y, \rho, \theta)</td>
</tr>
<tr>
<td>Transformation of binary contours, then very fast</td>
<td>Interpolation in Hough space rather than in the pattern</td>
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machine vision systems in industry using a CameraLink interface, are gradually being replaced by standardised computer interfaces such as USB-2, Firewire and Ethernet. The main advantages of GigE Vision cameras are a high bandwidth that enables a very fast transfer of the patterns from the camera to the computer, standard Ethernet boards instead of dedicated expensive frame grabbers, and thin and inexpensive cables. A cable length of up to 100 m allows the computer to be installed remotely from the SEM and the data to be transferred through an intranet. The digital signal is, in contrast to analog cameras, little affected by interference with electromagnetic stray fields. A highly welcome feature is the standardisation of controlling the main camera functions and data transfer with an easy-to-programme protocol. Cameras conforming to the GigE Vision standard protocol GenICam (Generic Interface for Cameras) can simply be exchanged without having to modify the driver nor the software. This is a particular advantage for EBSD systems, because camera performance makes rapid progress from year to year. Hence, with a GigE Vision camera as the backbone of an EBSD detector, a hardware upgrade can conveniently be conducted from time to time.

**New fast EBSD system**

At present, acquisition speed exceeds 1000 patterns per second on suitable samples and is thus by far faster than solving the patterns even with a high performance multicore PC. This has been motivation enough to split EBSD in two tasks that are first the high speed acquisition and storing of backscatter Kikuchi patterns as a sequence of raw, un compressed and unprocessed bitmap images, and second the repeatable offline evaluation of the original pattern sequence on any available PC apart from the SEM. 4, 5 Exactly the same area can be repeatedly indexed while experimenting with different Radon transform settings, phases, calibrations and pattern processing. Offline indexing is based on the same philosophy as energy dispersive spectroscopy spectral imaging where complete X-ray spectra are acquired from two-dimensional arrays of points and evaluated offline. Technical details of this ‘fast EBSD’ approach can be found in Ref. 7.

Offline pattern solution has many advantages over online orientation microscopy:

(i) the extremely high speed of acquisition is limited only by the CCD sensor of the camera and the speed of storing the patterns on the hard disk
(ii) cold field emitters with typically low stability are accommodated
(iii) less hydrocarbon contamination is induced at high speed
(iv) dwell time per pattern is constant whereas time for indexing depends on the actual grain orientation and phase
(v) no artefacts are induced that frequently occur in online indexing when synchronisation between the acquisition and interpretation of patterns is lacking.

A high acquisition speed is

(i) economical because the occupation time of the SEM is short. It is favourable for:
   a. fast in situ dynamic experiments
   b. study of sensitive materials
   c. serial sectioning by focused ion beam sputtering and three dimensional orientation microscopy in a dual beam SEM
   d. mapping of local lattice strain (elastic and plastic). 9, 10 Pattern analysis is time consuming and lends itself to offline pattern solving
   (ii) pattern indexing and interpretation can be repeated offline at any time using the original diffraction patterns. The documentation of unprocessed patterns is of particular value for process and quality control in industry
   (iii) the setting parameters of the indexing program can be optimised conveniently after acquisition
   (iv) reliability of system calibration, Radon transformation, indexing and the presence of a priori unknown phases can be checked
   (v) no compromise is made between speed of acquisition and reliability of indexing.

An alternative option of fast EBSD is acquisition, storage and online interpretation of the patterns, but at the disadvantage of reduced speed and reliability. Online evaluation will need the synchronisation of acquisition and indexing at a constant speed that should be slightly slower than the slowest task (actually pattern transformation and indexing). A constant speed is essential to guarantee a constant exposure (or dwell time) and so a constant brightness of the patterns. Speed of indexing, however, is not constant, but depends for instance on the actual grain orientation. If the camera (slave) is controlled by the indexing software (master), a lot of time is lost because the camera needs to be initialised for each pattern from raster point to raster point. If the camera is the master to trigger the indexing routine, acquisition speed must be slower than indexing takes for the most difficult patterns of a sequence. If the camera is running at free ‘continuous video’ speed, acquisition is fastest, but the position of the spot and the indexed pattern are not strictly correlated. This is of no concern within a grain, but vertical grain boundaries are then no longer straight lines. Therefore, the best compromise seems to have the camera running at a constant fast speed, storing the patterns on the hard disk and, if the user wants to see indexed results during measurement, solving the patterns in a second parallel task by reading them from the hard disk. The hard disk will thus function like a buffer memory. Parallel execution is quite fast with multicore processors and a fast hard disk. This procedure will allow for highest acquisition speed, give almost simultaneously (with a short delay) orientation and pattern quality maps, and does not need synchronisation.

**Example of application**

The new offline system is able to collect >1000 patterns per second on suitable specimens, considerably higher than any other available EBSD system. It is now achievable to perform real time EBSD characterisation combined with dynamic in situ thermomechanical experiments. Since the maximum speed of pattern acquisition is proportional to the beam current, it is essential to have an SEM with high probe current capability to reach very high scan speeds. The next generation of solid state hard disks will remove any practical speed limit in storing Kikuchi patterns.
Figure 1 shows crystal orientation maps from a sample of recrystallised nickel. The microscope, a Jeol JSM-840 with tungsten filament, was operated at a tilt angle of $70^\circ$, a working distance of 23 mm, an accelerating voltage of 20 kV, and a beam current of 50 nA. Beam currents in this range are quite common in energy dispersive spectroscopy analysis on metal samples. The acquired diffraction patterns had a resolution of $68 \times 68$ pixels, acquisition speed was 1050 patterns per second. Errorneous points in the map are concentrated at grain boundaries, giving a high hit rate of approximately 99%. Additionally, individual patterns used for system calibration were recorded separately with higher resolution and longer exposure times in the corners of the scanned field.

## Conclusion

In summary, the new fast EBSD acquisition system is capable of collecting crystallographic information in a fast, flexible and reliable way. Because of many advantages, high speed EBSD with offline solving the acquired pattern sequences will soon become the standard EBSD technique.

## References