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A FAST ACOM/EBSD SYSTEM

SZYBKI SYSTEM ACOM/EBSD

Automated backscatter Kikuchi diffraction in the scanning electron microscope allows the microstructure, crystallography and texture of polycrystalline solids to be characterized on the sub-grain size level. It is about to become a tool for process and quality control. Mandatory requirements for these applications are a high speed and measures to enable re-examination of the results at any time. Separate acquisition and store of sequences of diffraction patterns in a raw data format, followed by off-line calculation of the grain orientations, has distinct advantages over conventional on-line EBSD analysis. Aspects of hardware and software for realizing a fast ACOM/EBSD system are discussed.

Keywords: ACOM, BKD, EBSD, crystallographic texture, Kikuchi patterns, Radon transform, image processing

Automatyzowana rejestracja dyfrakcji Kikuchiego promieni zwrotnych w elektronowym mikroskopie skaninhowym pozwala na scharakteryzowanie mikrostruktury, krystalografię i teksturę polikrystalicznych ciał stałych w skali rozmiaru subziaren. Technika ta staje się narzędziem kontroli procesów i jakości. Obowiązującą normą dla tych zastosowań to duża szybkość oraz możliwość rewizji wyników w dowolnym czasie. Rozdzielenie akwizycji i przechowania sekwencji dyfraktogramów w formie danych pierwotnych w połączeniu z obliczeniami off-line orientacji ziarn posiada wyraźną przewagę nad konwencjonalną EBSD analizą on-line. Dyskutowane są aspekty hardwarowe oraz softwarowe dla realizacji szybkiego systemu ACOM/EBSD.

1. Introduction

The automated analysis of backscatter Kikuchi patterns (BKP) in the scanning electron microscope (SEM) has developed into a mature technique to characterize materials at the sub-grain size level [1, 2]. It provides quantitative crystallographic information about the microstructure in smooth, conductive and clean surfaces of bulk polycrystals. In particular crystallographic grain orientations are determined at the micro- or nano-scale. Scanning the beam across larger areas has led to Automated Crystal Orientation Microscopy (ACOM) which clearly represents the microstructure in terms of crystal orientation. This technique is also known under the commercial term Electron Backscatter Diffraction (EBSD). Morphological parameters such as grain size and grain shape distributions as well as crystal lattice related quantities such as crystal perfection and the fraction of recrystallized grains, grain boundaries, misorientations, the lattice type, and crystal texture of the sampled area as quantified by pole figures, orientation distribution, misorientation distribution and orientation correlation functions (ODF, MODF, OCF) can be deduced. Since SEM and commercial ACOM systems are readily available, it is no longer an academic technique reserved to the laboratory, but rather is on the best way to become a tool for process and quality control. It also enables a 3-D volumetric reconstruction of the microstructure from consecutive surface sections which are laid open by focused ion beam (FIB) milling. For these purposes, however, speed and ease of handling the ACOM system as well as the possibility to re-examine the results at any time are decisive requirements.

2. The actual system with off-line indexing

The actual system consists of the hardware detector NORDIF 500UF [3] and software which controls the digital beam scan of the SEM as well as pattern store. The detector contains a high-sensitive charge-coupled device (CCD) camera with GigE Vision interface to the computer. The patterns are stored on the hard disk, af-

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ter binning to 96 by 96 pixels on the sensor chip, as a sequence of raw bitmap images rather than as a reduced data set such as Hough peaks. Hence access to the original raw data is enabled at any time. At present the acquisition speed exceeds 750 patterns per second on suitable samples.

Two separate programs – SEMdif Viewer and SigmaCom – are used to perform off-line indexing of the backscatter Kikuchi patterns, calculation of grain orientations and construction of crystal orientation maps (COM), pattern quality maps (PQM) and indexing quality maps (IQM). The orientation data are interpreted in terms of Eulerian angles, axis-angle description, misorientations between abutting and distant grains, type of grain boundary and grain size as well as grain boundary statistics. Crystallographic texture can be analyzed by ODF and MODF calculation, and the construction of conventional as well as inverse pole figures.

Some advantages of off-line over conventional on-line solving of the diffraction patterns are:

- Dwell time per pattern is constant whereas time for indexing depends on the present grain orientation and phase.
- No artifacts are induced which frequently occur in on-line indexing when synchronization between the acquisition and interpretation of patterns is missing.
- The extremely high speed of acquisition is only limited by the sensitivity of the camera and speed of storing the patterns on the hard disk.
- A high acquisition speed is favorable for fast in-situ dynamic experiments.
- A high acquisition speed is economic since the turn-round of the SEM is short.
- Cold field emitters with typically low stability are accommodated.
- No compromise is made between speed of acquisition and reliability of indexing.
- The throughput of the SEM is independent of indexing and thus increased.
- Pattern indexing and interpretation can be repeated at any time by using the original diffraction patterns.
- The setting parameters of the indexing program can be optimized conveniently after acquisition.
- Reliability of indexing and the presence of a priori unknown phases can be checked.
- Off-line indexing is based on the same philosophy as EDS spectral imaging where complete X-ray spectra are acquired from 2D arrays of points and evaluated off-line.

As an alternative to off-line indexing, acquisition, store and interpretation of the patterns can be performed on-line as well, but at the disadvantage of reduced speed. A free-running camera and digital beam scan at constant speed will result in a shift of the beam position on the sample to the stored x-y position of the pattern respectively of the grain orientation in the map, since the patterns are read for indexing at varying intervals. The time required for the interpretation of a pattern is not constant, but depends on the actual grain orientation and pattern quality. This misalignment is of almost no concern as long as the beam is moving within a grain, whereas straight grain boundary lines may fray out. Furthermore, two patterns from each side of the boundary may be overlaid when the beam proceeds from one position to the next while the pattern is integrated before next read out, so that hit rate along grain boundaries is reduced or the rate of wrong indexing increases. If, on the other hand, dwell time is synchronized on the varying time required for indexing a pattern, noise and pattern quality will be affected. Asynchronous control of the camera to act as slave to the system by an external start and read signal from the indexing software can ensure constant exposure time and availability of the pattern on demand. This camera functionality, however, causes a reset at every step and thus a significant reduction of speed when a sequence of patterns has to be acquired. A better procedure is to perform pattern evaluation as a parallel task, separated from acquisition, at a slightly slower speed on an average. The patterns can thus be read with varying frequency either from the hard disk, as in off-line indexing mode, or from a first-in first-out buffer memory. Pattern acquisition and indexing will so be accomplished at almost the same time.

3. Example of application

As an example of Fast ACOM a recrystallized nickel sample is presented in Figure 1. A sequence of 10 000 backscatter Kikuchi patterns have been acquired with a NORDIF 500 UF detector an a Jeol FE SEM at 20 kV and 20 nA beam current. Speed was 770 pattern/sec so that measurement was accomplished within 13 sec. The left column (Fig. 1a) shows two typical patterns taken out of the sequence. The blue lines mark 10 automatically detected and consistently indexed bands. In Fig. 1b the crystal orientation maps for the hkl and uvw reference directions are shown. They have been constructed from the orientation data set. As usual, the colors have been encoded according to a standard triangle which was overlaid by a color triangle [4]. In Fig 1c the pattern quality map is given. Pattern quality is a measure of the clarity of the patterns. It has been determined from a FFT of the Radon transformed patterns. Pattern quality is an indicator of the perfection of the crystal lattice under the beam. From the orientation data set, further stereological and texture characteristics of the microstructure can be calculated. As an example, the high-angle grain boundaries with orientation differences >10° are depicted in Fig. 1d.

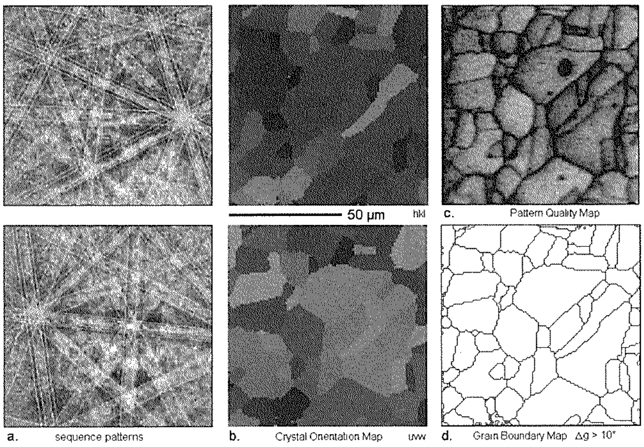

Fig. 1. Recrystallized nickel sample: a) Two examples out of the sequence of 100 by 100 backscatter Kikuchi patterns; b) Crystal Orientation Maps, top sample normal direction hkl, bottom reference direction uvw to the right. Colors correspond to directions in the standard triangle 001-011-111 RGB; c) Pattern Quality Map, gray levels in relative units; d) High-Angle Grain Boundary Map for misorientations >10°

The system has been calibrated automatically by solving 4 additional calibration patterns which had been recorded at a higher pixel number (160 by 160) in the corner points of the mapped area. Thus neither the position of the sample in the SEM nor the position of the scanned area on the sample surface need to be known for indexing the pattern sequence. The scanned points are further dynamically calibrated during pattern solving whereby a possible small misalignment of the sample due to a rotation about the microscope axis is corrected as well. Automatic and dynamic calibration [2] are mandatory for off-line indexing because the actual conditions of pattern acquisition are not known.

Figure 2 shows on the left side a calibration pattern (160 by 160 pixels) with 10 automatically located and consistently indexed bands. In Fig. 2b the profile across the accumulated Radon peak of band 9 is displayed (read line). The shaded rectangular box corresponds to the band profile which would be assumed when applying a "butterfly mask" for deconvolution [5]. It is clearly seen that the real band profile is in general distinctly different. In addition, the band profile and its width depend on the hkl indices, the lattice constants, the accelerating voltage and the position of the band in the pattern. Already for solving one pattern with high accuracy, several different "butterfly masks" would be necessary, and another set of masks is required when changing the accelerating voltage or the sampled material [5]. Therefore, band profile analysis in Radon space and applying a FFT for band localization is superior over applying a single deconvolution mask on the Hough transformed patterns [6]. Figure 2c is a representation of the Radon map of this backscatter Kikuchi pattern.

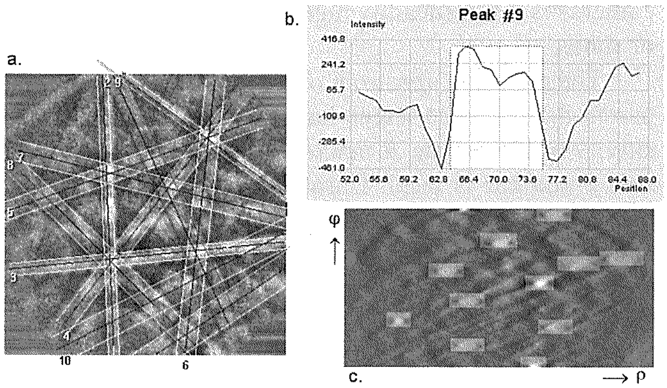

Fig. 2. Radon transform of a calibration pattern: a) The calibration pattern of 160 by 160 pixels. The automatically located and indexed bands are marked; b) Profile across band 9; c) The Radon transform. The band peaks are marked

4. Technical considerations how to achieve a high speed

4a. Detector hardware

A high sensitivity of the detector is of key importance for high speed. An enormous advance in image sensor performance has been achieved recently mainly due to the demands of small consumer cameras. Although CMOS sensors are superior in speed, CCD sensors still have a higher quantum efficiency and are more sensitive. Recent electron-multiplying CCD (EMCCD) sensors promise some advantages at very low light levels. They have an additional gain register inserted between the usual shift register of the CCD and the pre-amplifier where, in a high electric field zone, further electrons are generated by impact ionization. Since this amplification is done before the usual pre-amplifier, signals below the read-out noise of the pre-amplifier can be detected.

Because of the high speed of pattern acquisition and read-out noise being the main contribution to the noise floor of the sensor, cooling of the sensor chip for shot noise reduction is not necessary. If present, the IR filter in front of the sensor chip should be removed. In the factory, machine vision cameras are usually set for low gains. This is fine in situations where sufficient light is available, whilst backscatter Kikuchi patterns are dim. Therefore, camera gain must be increased considerably, although the patterns appear to become noisy. This is not that serious. In contrast to former analog cameras, the amplifiers in modern digital cameras do not add significant noise to the signal, rather than amplify the signal at the same rate as the noise within it, so the signal-to-noise ratio will not change. Furthermore, the Radon transform of the pattern is amazingly robust against noise in the detection of straight bands. A further advantage of digital cameras is a significantly higher dynamic range of 12 bit or more.

The emission spectrum of the phosphor on the transparent fluorescence screen has to match the spectral sensitivity of the sensor. A macro lens with the lowest available f-number is used. Chromatic lens aberration is of no concern, important are low distortion, low vignetting and a flat image field. Only a marginal increase in sensitivity is gained with a (tapered) fiber optic between the fluorescence screen and the sensor for image transfer, but at much higher costs. The future might be a lensless direct exposure of the sensor chip to the pattern forming electrons.

Pixel binning on the sensor chip is a well proved means to increase sensitivity and speed. (Each individual photo-sensor on the chip array as well as each image point is called a pixel.) The pre-amplifier on the sensor chip is optimized for high dynamics and low noise at full pixel resolution and medium to high illumination levels. This is adequate for most situations in machine vision applications. At faint illumination however, as is the case in backscatter Kikuchi diffraction in the SEM, the working point of the pre-amplifier drops to the flat foot of the current-voltage characteristic curve, hence the signal of one single photo-sensor will be submerged by noise. If n abutting pixels are bundled together on the chip already during the read-out procedure, the current to the pre-amplifier 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. A further advantage is the possibility to adjust the pixel resolution of the pattern by a software call. Pixel binning ranges from 2 by 2 up to 8 by 8. In principle, a dedicated sensor chip with a coarse array of pixels and correspondingly increased pre-amplification would be superior. Filling factor and capacity per pixel would be improved with the advantage of higher sensitivity and dynamic range. However, the commercial demand for this type of sensors is too small, whereas consumer and machine vision cameras boast of ever increasing pixel numbers.

A multiple tap sensor is a tool to significantly increase the camera speed. It allows for the image to be split in sections so that the charge transfer and paralleled read out can be accomplished much faster. One side effect of using a multiple tap camera is a channel mismatch which is visible as a difference in sensitivity, contrast and brightness of the image sections. Another image defect may be a very small spatial shift between the image sections. These artifacts with a dual tap camera, for instance, can result in a small vertical seam between the image halves which might erroneously be interpreted for a vertical Kikuchi band in the pattern which then has to be filtered out in the Radon transform.

A low detector sensitivity can be offset by a high beam current only to some extent. SEM with a field emission source enable high currents of several ten nA in beam spots of a few nm diameter, whereas current in small beam spots drops down dramatically when produced with a thermal emitter gun. Therefore, FE SEM are the first choice for high speed ACOM. It is worth to keep in mind, however, that contamination rate increases rapidly with current density. Therefore, it is wise to focus the beam only down to a useful diameter according to the actual grain size and the physical resolution limit of BKD which is in the range of some ten nm, depending on the material and accelerating voltage. Furthermore, a low beam current is in general desirable for the gentle production of patterns to reduce sample damage and charging of low-conductive materials.

The camera interface is worth of some consideration. Frame grabbers, as common with conventional analog cameras and with machine vision systems in industry using a CameraLink interface, are gradually replaced by standardized computer interfaces that are USB-2, Firewire and Ethernet (see Table 1). They are usually supplied with the PC for consumer appliances or available as a low-cost PCI board and can conveniently be used for the EBSD detector on the SEM as well.

TABLE

Parameters of Ethernet, USB-2 and Firewire computer interfaces

	Ethernet (GigE Vision)	USB-2	Firewire
topology	peer-to-peer	master-slave	peer-to-peer
bandwidth	1 Gbit/sec	480 Mbit/sec	400 Mbit/sec IEEE 1394a 800 Mbit/sec IEEE1394b
cable length	100 m	5 m	5-15 m
current	—	0.5 A at 5 V	1.5 A at 8-30 V

CameraLink has been the interface of choice for professional machine vision applications until lately. It is a fast and rugged solution which complies with several cameras at the same time. The disadvantages are high costs and clumsy cables. A dedicated frame grabber and proprietary software drivers are required. USB-2 and Firewire interfaces are commonly used in consumer equipments.

The main advantages of GigE Vision cameras are low costs, a high bandwidth which enables a very fast transfer of the images from the camera to the computer, thin and cheap CAT5e or CAT6 cables. The cable length of up to 100 m allows the computer to be installed remotely from the SEM, or the data are 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 standardization to control the main camera functions and data transfer with an easy-to-program protocol. Cameras conforming to the GigE Vision standard protocol GenICam (Generic Interface for Cameras) can simply be exchanged without having to modify the driver or software. This is a particular advantage for EBSD systems, because camera performance makes rapid progress from year to year, indeed faster than personal computers. Hence, with a GigE Vision camera as the backbone of an EBSD detector, a hardware upgrade can conveniently be done from time to time. The user can choose from a broad collection of economic cameras from several suppliers.

4b. Software

Until 2000, speed of on-line ACOM with an analog camera as detector was limited by the video frame rate to less than 25 respectively 30 orientations per second. There was little motivation to further increase the speed of the indexing software. After the introduction of digital cameras the situation has changed. At the time of writing this article, speed of our ACOM system is about 100 orientations per second on a fast single-core CPU PC and approaches 200 orientations per second on a high performance dual-core CPU PC (pattern size 96 by 96 pixels, 7 or more detected bands). A commercial system [7] is said to make about 400 orientations per second (pattern size 80 by 60 pixels, quad-core CPU 3 GHz Xeon PC) on suitable samples. This boost in performance is partially due to improved performance of the computer hardware. The speed of numerical calculations scales with typically 2/3 of the increase in CPU clock rate. Further progress was made with fast 16 bit DA converters for digital beam control, graphic boards with co-processors and hard disks. The next generation of solid state hard disks will remove any practical speed limit in storing Kikuchi patterns. The high und uneven background in backscatter Kikuchi patterns can be corrected for already in the camera by subtracting a flat background image pixel by pixel on the fly.

A major step ahead has been achieved by improving the software. The speed at which a band can be localized in the pattern scales linearly with the number of pixels in the pattern and the number of $\varphi-\rho$ points in the discrete Radon transform. The calculation and evaluation of a Radon transform of 100 by 100 points takes on the average about 10 times longer than indexing the detected bands. So speed can be quadrupleted by simply coarsening the backscatter Kikuchi patterns and – in conformity – reducing the size of the Radon transform to one fourth. An unwanted side effect, however, is then 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 $\Delta\varphi = 1.5 - 2°$, 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 localization. Even though and as a consequence to this inaccuracy, less detected bands are then indexed unambiguously. What is even worse, wrong orientations are found and hit rate 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 fortunately by 1/n less than the mean band deviation. Coarsening the patterns and Radon transforms and, at the same time, putting up with a lower reliability of orientation data is an appropriate means of increasing speed in on-line ACOM only for getting a first impression of the texture and the quality of sample preparation.

The Radon transformation is a linear operation [6]. It is, therefore, well suited for multi-threading and parallel computing. The pattern as well as the Radon transform can be cut in several parts, in particular with a multi-core processor, to execute the transformations in parallel tasks. So speed is increased almost proportionally with the number of processors without having to concede a compromise in accuracy. In addition, multi-core processors surpass conventional processors in a higher CPU clock rate. Threaded applications, on the other hand, incur little additional performance penalty on single-processor machines.

5. Conclusion

With a bundle of high-performance hardware and optimized software, more than 500 orientations per second can be achieved in on-line ACOM measurement at present. Separate acquisition and store of pattern sequences is still significantly faster. Its limit is mainly set by the sensitivity of the presently available cameras. Off-line pattern solution has many advantages over on-line ACOM, in particular since pattern interpretation can be repeated at any time by using the original backscatter Kikuchi sequences. The data can be examined again and the presence of a priori unknown phases can be checked for.

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