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1 **electron CHanneling ORientation Determination (eCHORD):**  
2 **an original approach to crystalline orientation mapping**

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8 **Keywords** : Orientation mapping, SEM, Electron Channeling, EBSD

9

10 **Highlights :**

- 11 - A proof of concept for an orientation mapping in the SEM is proposed.  
12 - This new approach is based on electron channeling.  
13 - Excellent agreement compared with EBSD maps was obtained on an aluminium alloy  
14 sample.

15 **Abstract**

16 We present a proof-of-concept attesting the feasibility to obtain orientation maps of  
17 polycrystalline materials within a conventional Scanning Electron Microscope (SEM) using a  
18 standard goniometer and Back Scattered Electron (BSE) detector. The described method is  
19 based on the analysis of the contrast variation of grains due to the channeling of incident  
20 electrons on a rotating sample. On each pixel of the map, experimental intensity profiles as a  
21 function of the rotation angle are obtained and compared with simulated ones to retrieve the  
22 orientation. From first results on aluminum polycrystals, the angular resolution is estimated to  
23 be better than one degree.

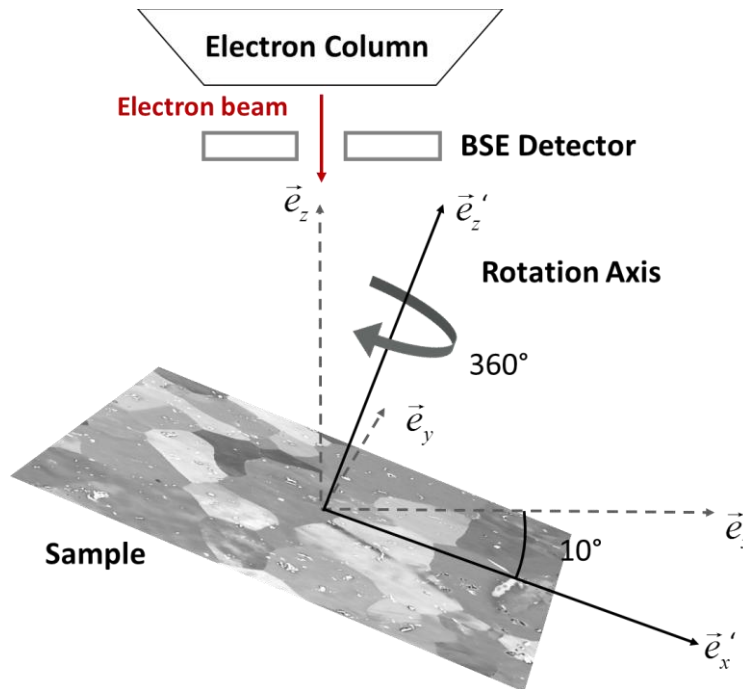
## 1. Introduction

In the past ten years, the EBSD and Transmission Kikuchi Diffraction (TKD) techniques have been modified to improve spatial and angular resolutions as well as the indexation speed. For instance, High Resolution-EBSD (HR-EBSD) [1] provides a high angular resolution at the expense of time and heavy data processing whereas TKD-on axis [2] procedure on Transmission Electron Microscopy (TEM) samples allows obtaining a better spatial resolution together with a high indexation speed. Orientation maps can also be obtained by a completely different approach using images presenting channeling contrast, as demonstrated in the article by Langlois *et al.* [3]. In this paper, an ion beam was used to produce secondary electron images, taking benefits from a strong channeling phenomenon with a high secondary electron yield. If well-defined orientation maps were obtained by ion **Channeling Orientation Determination** (iCHORD) on cubic materials, the ion beam may intrinsically affect the sample by milling, implantation or amorphisation. Such negative issues can be overcome with an electron beam. This paper demonstrates how the iCHORD method can be extended to electron channeling contrast to recover crystalline orientations in a standard SEM. This method is called **eCHORD** and opens new possibilities for fast and spatially resolved crystallographic mapping.

## 2. From ion to electron channeling modelling

The channeling contrast is directly related to the orientation of the crystal relative to the incident beam [4]. Following this principle, it has been shown that recording the intensity variation of a crystal in rotation around the z axis of the goniometer (tilted with respect to the

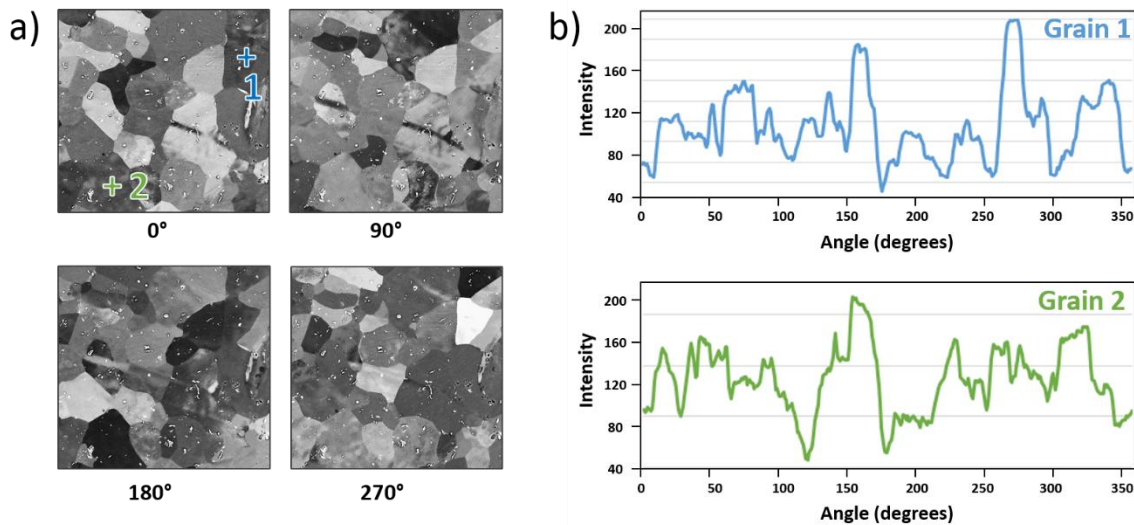
1 beam) may result in an unambiguous intensity profile for a given orientation [3]. The  
2 corresponding acquisition setup has been adapted for electrons as described in Fig.1. Serial  
3 imaging was performed using a Gemini I SEM column (Carl Zeiss Microscopy GmbH,  
4 Oberkochen, Germany) equipped with a retractable standard 4 Quadrant solid-state Detector  
5 (Namely 4Q-BSD). The electron accelerating voltage was set to 15kV and the sample tilt to 10°.  
6 The so-called High Current Mode was activated. With the use of an objective lens aperture of 60  
7 microns, a beam current of 2nA was measured. A working distance of 6 mm was chosen to  
8 collect a maximum of BSE on the detector.



9  
10 *Figure1: Experimental eCHORD setup: the sample is 10° tilted and rotated from 0° to 360°. A BSE*  
11 *detector records the backscattered electron intensity at each rotation step.*

12 For each rotation step of 1°, an image is collected from 0° to 360°. All 360 images are  
13 stacked together in FIJI software [5]. The stack is numerically treated using Xlib library

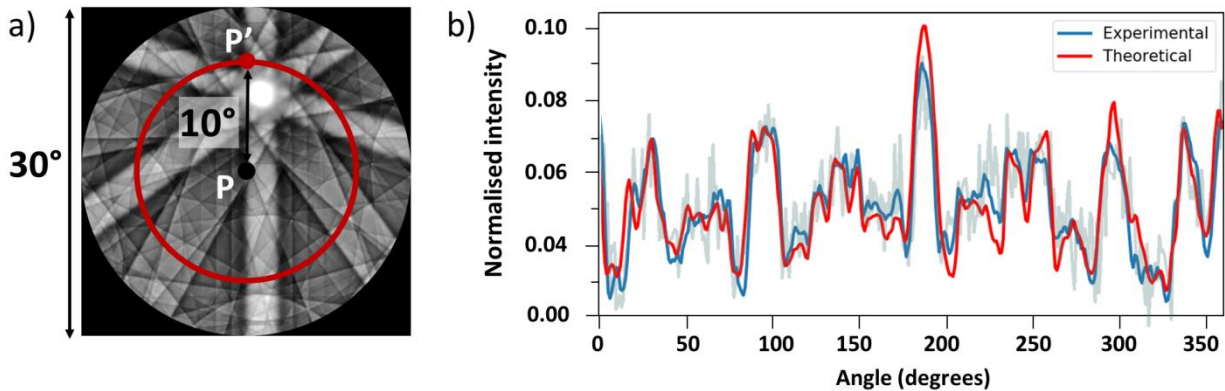
1 (developped by Beat Münch [6]) for denoising and edge detection as well as MultiStackReg  
2 (developped by Brad Busse [7]) for image alignment. Noise is removed from raw images using  
3 the Tschumperlé & Deriche anisotropic diffusion algorithm [8] implemented in Xlib with default  
4 parameters. This algorithm allows removing the noise while preserving the edges i.e. the grain  
5 boundaries. Concerning images alignment, the process is in three steps: i) edge detection using  
6 Canny algorithm [9], ii) alignment of the edge-detected stack with affine transformations [10],  
7 saving the set of alignment parameters, iii) original stack alignment by loading this parameters  
8 set. The alignment algorithm used here does not require any experimental information about  
9 rotation, tilt or stage drift; it will automatically find the best affine transformation to align all the  
10 images of the series to the initial one. In the final stack, a given pixel contrast changes  
11 throughout the stack due to the electron channeling. These intensity variations are plotted as a  
12 function of the rotation angle in order to obtain an intensity profile for each (X, Y) point of the  
13 image stack (see Fig.2.b).



14  
15 *Figure 2: a) representative images extracted from the aligned stack; b) experimental intensity*  
16 *profiles extracted from the stack at two different positions.*

1 From such experimental profiles, the orientation can be recovered through the following  
2 procedure: 1/ sampling of the orientation space, 2/ simulation of theoretical intensity profiles  
3 for each orientation of the sampling, 3/ search into this database to find the best match  
4 between experimental and theoretical profiles using dot product as metric. Sampling of the  
5 orientation space is carried out statistically by randomly generating 1 million quaternions on  
6 unit quaternions sphere using the algorithm described in reference [11] and taking into account  
7 the symmetry operations of the crystal. The mean disorientation between nearest neighbors in  
8 the orientation space is 0.5 degrees. Simulated profiles are calculated from theoretical Electron  
9 Channeling Pattern (ECP). Actually, during an ECP acquisition, the diffracted intensity is plotted  
10 as a function of the orientation of the beam relative to the sample normal. If the sample normal  
11 is parallel to the electron beam, its diffracted intensity will be found at the center of the ECP  
12 (called P in Fig. 3). In our configuration the sample is tilted at  $10^\circ$  with respect to the electron  
13 beam, thus the diffracted intensity is described by a  $10^\circ$  shift of point P to P' on the ECP.  
14 Therefore, the intensity profile, as a function of the rotation angle, corresponds to the intensity  
15 gathered along a  $10^\circ$  radius circle centered on point P. A tilt of 10 degrees together with a  
16 tension of 15kV is a good compromise to cross enough Kikuchi lines to obtain a unique signature  
17 of the orientation. ECP patterns were calculated using EMsoft simulation suite from M. De Graef  
18 *et al.* [12]. For each of the orientations, an ECP was computed, with parameters corresponding  
19 to the high tension, the tilt angle and the materials used. This leads to 1 million ECPs with a  
20 pattern size of 261x261 pixels and an opening semi-angle of  $13^\circ$  leading to a pixel resolution of  
21  $0.1^\circ$ . The computation of the ECP database took 45 minutes on 8 cores Intel® Core™ i7-CPU @  
22 2.60GHz workstation. Because the electron beam is convergent, directions of incident electrons

1 belong to a cone of semi-angle  $\alpha$  that depends on both aperture size and working distance.  
 2 Electrons intensities as a function of the incident angle inside the cone are modelled by a  
 3 Gaussian distribution of standard deviation  $\sigma$  [13]. Therefore, instead of taking the intensity  
 4 of a unique point on the circle drawn on the ECP, one has to integrate the intensity in the  
 5 vicinity with the same Gaussian ponderation. An identical result is obtained by taking a unique  
 6 point on an ECP Gaussian-blurred with the same  $\sigma$ . In the present work, the  $\sigma$  value of  
 7 the Gaussian distribution depends on aperture diameters and working distance. Its value of  
 8  $0.08^\circ$  was determined using data available in Zaefferer *et al.* [14]. For each calculated ECP a  
 9 unique theoretical profile, corresponding to one orientation, is extracted. This process is  
 10 repeated for each of the 1 million ECP generated. Fig. 3b) shows a comparison of an  
 11 experimental eCHORD intensity profile and a simulated one for a given orientation of the  
 12 crystal.



13  
 14 *Figure 3: Modelling of the intensity profile for orientation (300.7°, 40.6°, 6.7°). a) ECP simulated*  
 15 *from EMsoft at 15 kV for aluminum: in red, circle corresponding to the beam path when a*  
 16 *sample is tilted at 10° and rotated from 0° to 360°; b) comparison between the theoretical*

1 *profile extracted from the ECP in (a) (red), the experimental profile before denoising (light blue)*  
2 *and after denoising (blue).*

### 3 **3. Results and Discussion**

4 Fig. 4 presents a comparison between EBSD and eCHORD data on the same area of a  
5 polycrystalline aluminum alloy sample, with a database of 1 000 000 theoretical profiles. The  
6 EBSD map has been acquired with a Nordlys F camera and data were post-processed with HKL  
7 Channel5 software (Oxford Instruments, Abingdon, UK). EBSD mapping was performed with an  
8 accelerating voltage of 20kV, with a step of 400nm and a size of 498x509 pixels. The sampling of  
9 the pattern was 106x80 pixels whereas the resolution of the Hough transform was of 65. The  
10 acquisition speed was 283 Hz (leading to an acquisition of 15 minutes) for a hit rate of 92.8%.  
11 The eCHORD map has been obtained from an image series of 360 images corresponding to a  
12 360° rotation of the stage. Each image has been acquired at 15kV, 10° tilt, with a beam scan  
13 speed of 10 seconds per image (leading to an acquisition of 1 hour) and a step size of 400nm.  
14 Rotation and acquisition of images processes were automated. Size of the region of interest  
15 after processing is 405x387 pixels. By nature, the hit rate is 100%. To have an idea about the  
16 confidence of the indexation, one can refer to the 'distance map' (Fig 4.d) which corresponds to  
17 one minus the dot product between the experimental profile and the assigned theoretical  
18 profile. Considering that acquisition and database generation were carried out simultaneously,  
19 the total time to obtain this eCHORD map was approximately 100 minutes: 1 hour for  
20 acquisition and database generation + 30 minutes for image processing and 5 minutes of  
21 indexation (~500 experimental profiles indexed per second). The disorientations angles between

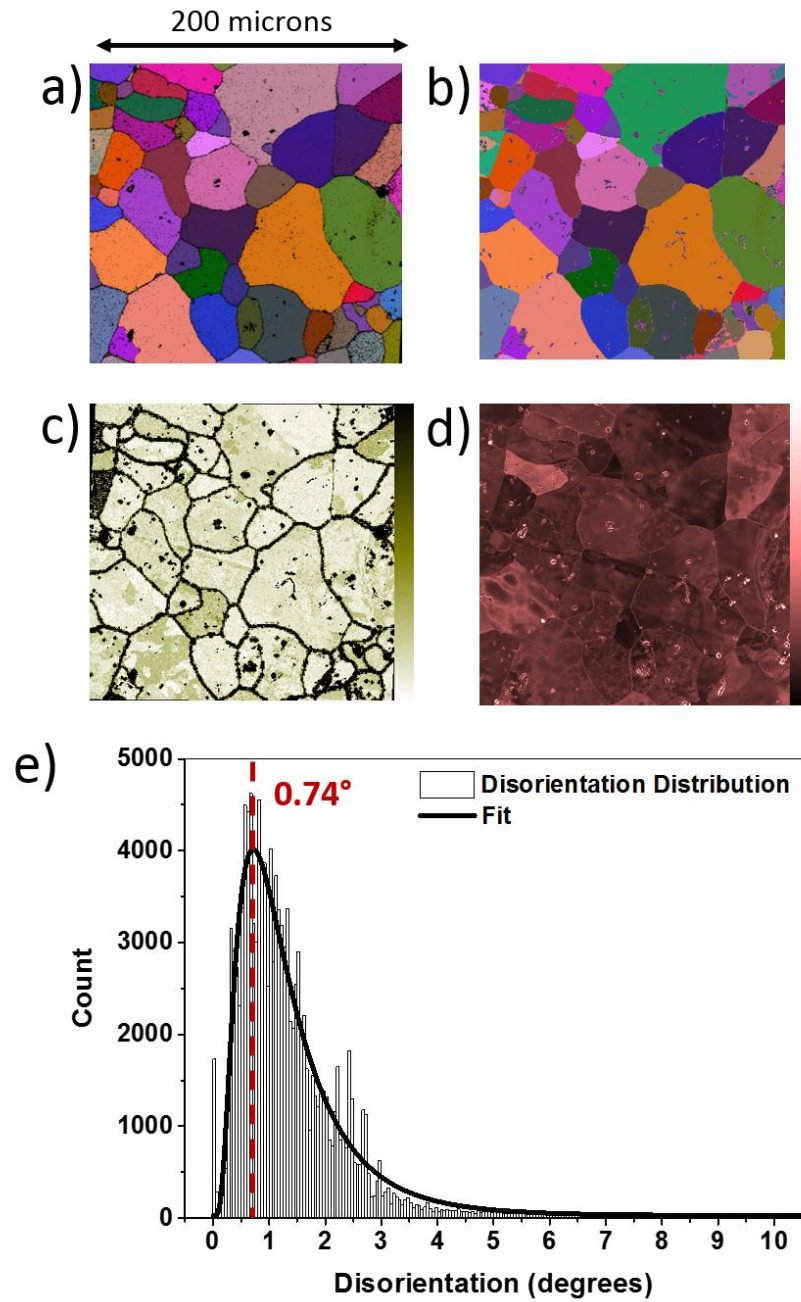


1 EBSD reference map (after denoising) and eCHORD map follow a log-normal distribution peaked  
2 at 0.74 degree. Almost all grains are correctly indexed with an error of less than one degree  
3 compared to EBSD data. One has to keep in mind that such a point-to-point comparison is  
4 subject to angular and spatial imprecisions that occur when moving from EBSD acquisition  
5 geometry to the eCHORD one.

6         Concerning the angular resolution, the sampling of the orientation space, and therefore  
7 the size of the database, needs to be at least 1 million, otherwise some experimental profiles  
8 are not found in the database. This observation could witness an angular resolution potentially  
9 better than 0.5°, which is the mean disorientation between two neighbour orientations in the  
10 database. However, a limitation arises at low magnification due to the scanning of the region of  
11 interest which provokes a beam deflection. Depending on beam position during scanning, the  
12 angle between the rotation axis and the electron beam slightly differs from 10°. Many other  
13 parameters also influence the spatial resolution such as pixel size, accelerating voltage, sample  
14 tilt and quality of image alignment. Their respecting roles will be discussed in another paper.

15         Compared to all existing orientation mapping methods, eCHORD opens new  
16 perspectives. Firstly, the acquisition geometry is very simple (tilt of ~10° and 6mm for WD). This  
17 allows movements of the sample on a large scale without touching the electron column, which  
18 is definitely an advantage when mapping and stitching large areas. Another advantage linked to  
19 this geometry is the possibility to acquire 3D orientation volume (potentially on any given  
20 location on the surface of the sample) by coupling eCHORD with an ion column. Last but not  
21 least, the eCHORD method only requires standard BSE detector and goniometer, and can be  
22 implemented on any standard SEM.

1



2

3 *Figure 4: a) processed EBSD reference map (Euler map); b) eCHORD map (Euler angles) of the*

4 *same area; c) Disorientation map from 0° (white) to 10° (black) between EBSD and eCHORD*

5 *map; d) Distance map corresponding to 1 - dot product(experimental profiles; assigned*

1           *theoretical profile) from 0 (black – confident indexation) to 0.05 (white – less confident*  
2                           *indexation); and e) corresponding disorientation distribution.*

### 3   **4. Conclusion**

4           This proof-of-concept study shows that orientation maps can be obtained using BSE  
5 channeling contrast. Similar results within an error of 1° were obtained using eCHORD and EBSD  
6 techniques on aluminum alloy sample. Concerning acquisition and indexation time, it is worth  
7 noting that, in this proof-of-concept, it was not our goal to optimize them. Many improvements  
8 directions exist to reduce the time while keeping the performances: reducing numbers of  
9 images, increasing scan speed coupled with efficient denoising, selecting only pertinent beam  
10 directions in the ECP, improving database search algorithm... Further experiments will also be  
11 carried out to evaluate how eCHORD can be used for non-cubic, multi-phased or deformed  
12 materials. One main advantage of eCHORD is that no additional hardware is necessary for this  
13 method: standard BSE detector and goniometer are sufficient. Our set-up will potentially  
14 improve spatial resolution and acquisition time and may even open new perspectives towards  
15 3D orientation imaging.

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