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Ion imaging in a Focused Ion Beam microscope: modeling the channelling contrast to construct EBSD-like orientation maps

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Orientation mapping

Crystallographic orientation maps are commonly obtained by Electron Back Scattering Diffraction (EBSD) in a Scanning Electron Microscope (SEM). Briefly, this technique consists in acquiring an EBSD pattern on each point of a region of interest, and then to determine the crystallographic orientation by mean of indexation algorithms relying on the lines in the EBSD pattern. Although very efficient thanks to more than twenty years of commercial developments, the EBSD technique still suffers of some drawbacks linked to the acquisition part of the technique:

- limited spatial resolution compared to imaging (due to 70° of stage tilt and acquisition time)
- restrictive acquisition geometry (camera + polar piece + tilted sample)
- delicate surface preparation
- indexation time for high resolution maps
- supplementary hardware (camera)

A real breakthrough in crystallographic orientation mapping would be to implement a new indexing solution free of the aforementioned constraints.

Channelling contrast in FIB images

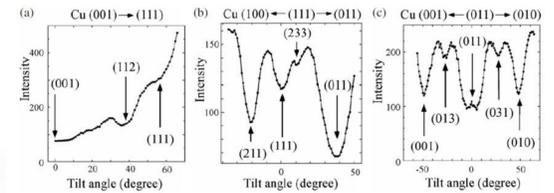
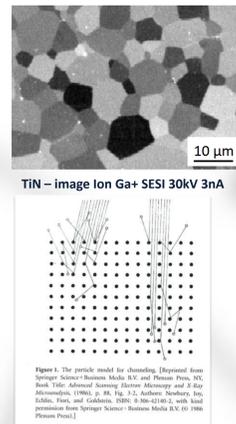


Fig. 2 Variation of the secondary electron intensity with respect to the tilt angle for Cu: (a) tilting direction from (001) to (111), (b) tilting direction from (111) to (011) and (100) and (c) tilting direction from (011) to (001) and (010).

Crystallographic orientation contrast associated with Ga⁺ ion channelling for Fe and Cu in focused ion beam method
Y. Yabuta, K. Kaneko, T. Fujita, W.-J. Moon and Z. Horita
Journal of Electron Microscopy 53(5): 571-576 (2004)

The principle of the channelling contrast is the following: when the incident beam comes parallel to a set of crystallographic planes inside a grain, the intensity of this grain decreases.

The idea of linking the contrast to the crystallographic orientation was already proposed years ago, without converging to a complete indexation solution for common polycrystalline materials like metals or ceramics.

Basis of our work

The starting point is a series of images acquired for several tilt angles, or several rotation angles. From this kind of image series, in each pixel we can extract an intensity profile due to the channelling phenomenon. This intensity profile will exhibit peaks due to the fact that, during the acquisition, the incident beam was parallel to some crystallographic planes.

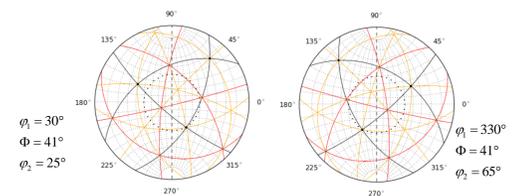
The question is: how to recover the crystallographic orientation from this kind of intensity profile ?

Among the possible solutions, we chose the « brute force » approach by calculating a theoretical database with all the possible intensity profiles using crystallographic computations. Then, for a given experimental profile associated to an (X,Y) position in the region of interest, a search is carried out in the database to find the closest theoretical profile. The corresponding Euler triplet is then assigned to the (X,Y) position. This method allows at the end obtaining a complete orientation map as for the EBSD technique.

1. Tilt vs rotation series

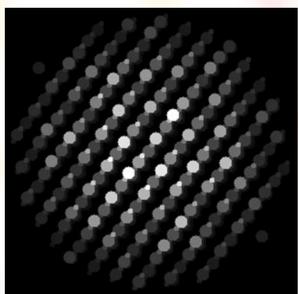
Tilt serie :

- gain and brightness corrections due to a continuous change of the sample position compared to the detector
- Indexation ambiguity: same intensity profile for twin set of Euler angles



→ rotation series @ 40° tilt
Step ; Range

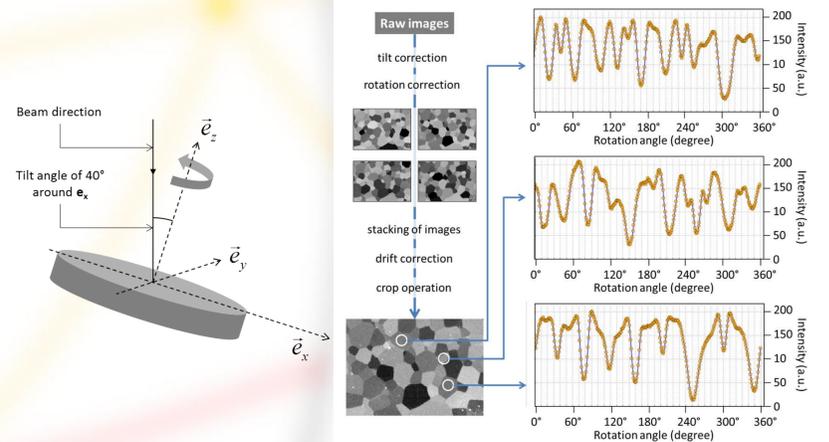
3. Theoretical database construction



For a given orientation of a crystal (i.e. one grain in one image of the series), the intensity received by the detector from this crystal can be related to the projection of the atoms on a plane corresponding to the sample surface. More precisely, it is the sum of the grey levels of the pixels composing the projection that can be related to the intensity received, after some mathematical adjustments for the relative contribution of each atom in the projection. It constitutes one data point in an intensity profile. The orientation of the crystal is then changed the same way as the experimental conditions, and the complete theoretical profile is obtained.

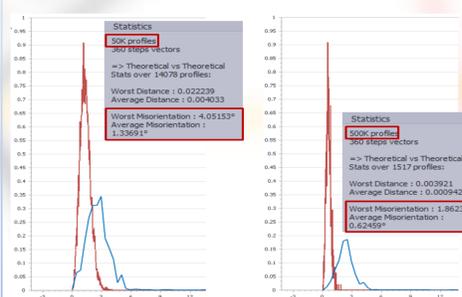
The database is then constructed by statistically sampling the orientation space, and associating an intensity profile to each Euler triplet. Euler angles are converted to quaternions for simplicity sakes in the computations.

2. Image series acquisition



The sample is first tilted to an angle of 40°. Then, it is rotated around its tilted normal. For each rotation step, an image is acquired using the secondary electron detector. A complete 360° rotation is achieved, resulting in a series of ion images. All these images are then corrected from the 40° tilt and for the rotation, in order to have the region of interest oriented the same way in all the images. After stacking the images together, the residual drift is corrected. At the end, intensity profiles can be extracted by plotting the intensity of each pixel along the stack (i.e. the rotation angle), at a given (X,Y) position in all the images.

4. Database search



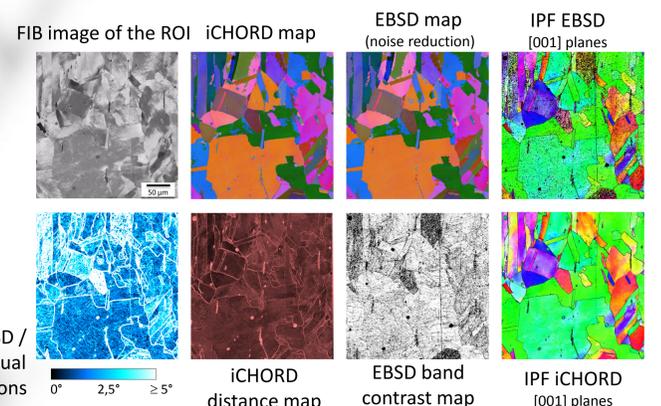
The search in the database starts with an exhaustive search for a certain number of positions in the area. Then, an algorithm relying on similarities between the profiles is used to index the pixels.

The size of the database (i.e. the fineness of the orientation sampling) increases the angular resolution but also the indexation time. However, it is possible to statistically evaluate the angular resolution obtained with a given database size by calculating the disorientation between neighbouring points in the database. A distribution of disorientation is then available (see the figure on the left) to evaluate the angular resolution that can be expected. It is then the user that choose which precision is required for a given application.

Results on Cu

30 kV – 700 pA
image size: 1324 x 1372
pixel size: 225 nm
magnification: 500X
acquisition time : 40 min
treatment: 40 min

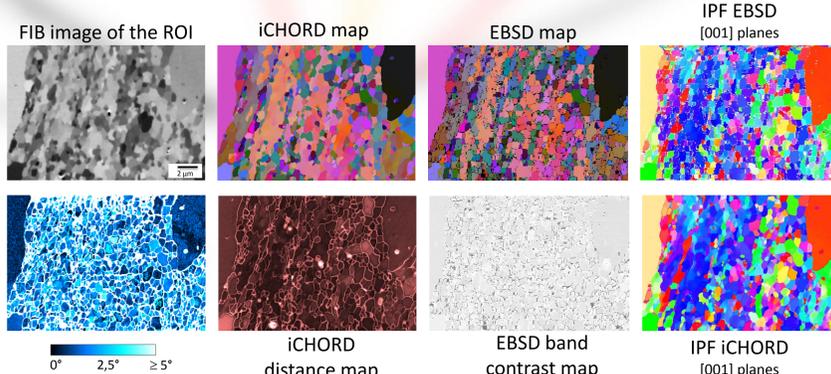
Disorientation EBSD / iCHORD after manual distortion corrections



Results on Fe

16 kV – 45 pA
image size: 749 x 512
pixel size: 22.3 nm
Magnification : 4000X
Acquisition time : 1h15min
Treatment : 40 min

Disorientation EBSD / iCHORD after manual distortion corrections



Conclusion

It is demonstrated here that the iCHORD method allows obtaining orientation maps fully comparable to the ones obtained by the EBSD method. The main advantages compared to EBSD are the better spatial resolution obtained in the maps, in less time, keeping a reasonable angular resolution estimated to 1°. The copper sample revealed itself to be not so sensitive to irradiation effect nor milling of the surface. For the Fe sample, we had to use more gentle conditions to be able to keep the initial surface. Conditions have to be adapted to each materials. The next steps will be the implementation of the other crystallographic systems, as well as the possibility to discriminate between different phases in the region of interest. Using a FIB opens also many possibilities toward 3D orientation mapping, potentially at any position of the sample, without specific sample preparation.

Acknowledgments

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