

## Solving the 180° Orientation Ambiguity Related to Spot Diffraction Patterns in Transmission Electron Microscopy

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A TEM attachment using an automated crystal orientation mapping method (ACOM/TEM) has been developed the last few years [1]. The procedure is similar to the one used by the well-known electron backscatter diffraction (EBSD) attachments for scanning electron microscope. It consists in scanning the area of interest with a nano probe, collecting the diffracting signal at every location and deriving the crystal phase and orientation with dedicated software. The tool provides spatial resolutions down to 1 nm when used with a Fields Emission Gun (FEG) equipped TEM. The orientation and phase indexing strategy makes use of an image recognition algorithm known as template matching that turns out to be practically insensitive to the dislocation density and, consequently, remains efficient even for severely deformed materials. The reliability of the procedure is known to be further improved when the technique is combined to precession electron diffraction [2]

A main difference with respect to EBSD attachment, stands in the fact that spot patterns (i.e.: Bragg diffraction peaks) are preferred to Kikuchi lines to derive the local orientation and phase from the TEM diffraction patterns. This choice proved to be efficient for characterizing deformed metallic materials as well as for phase recognition. The drawback of this choice is that the diffracting signal is limited to low Bragg angles (typically few degrees). Consequently, the diffraction patterns contain frequently only the zero order Holtz zone and the orientation determination is unsafe [3]. A typical example is the [111] diffraction pattern for steels (or other body centred cubic materials) depicted in figure 1. The absence of any higher order reflexion prevent the crystal orientation to be distinguished among the two possibilities shown that are related by a 180° rotation around [111]. The disorientation (minimum orientation taking into account the symmetry of the crystal) along a line drawn over the area of interest (Fig.2) exhibits frequent jumps of nearly 60°. Note that the disorientation is calculated with respect to the first point of the line (a point to point disorientation would give practically 60°). Also, the orientation map appears spotty because of the random choice of either one solution or the other. The figure concerns a deformed sample that contains dislocation cells whose misorientations are hard to estimate due to the frequent orientation ambiguities.

A post-processing automated correction routine has been developed. It consist in (i) detecting ambiguous orientation, (ii) comparing the local orientation with the one of the neighbour pixel and (iii) in case of improvement (i.e.: decrease of disorientation) exchange the orientation with the one corresponding to a 180° rotation around the closest high symmetry axis. The orientation information is spread from one pixel to the next one starting from the safer point and with a random walking path. The degree of safety is estimated by comparing the correlation indexes (through the template matching process) between the acquired diffraction pattern and the two alternative solutions.

One of the main issues is the determination of the axis around which the rotation must be performed. Some high symmetry axes are naturally ambiguous. They may be readily recognized, in particular for cubic materials and collected in a pre-set list of axes. The important point is that other orientations may also be unreliable if they are close from one of the previous ones. This is illustrated in Fig. 1.

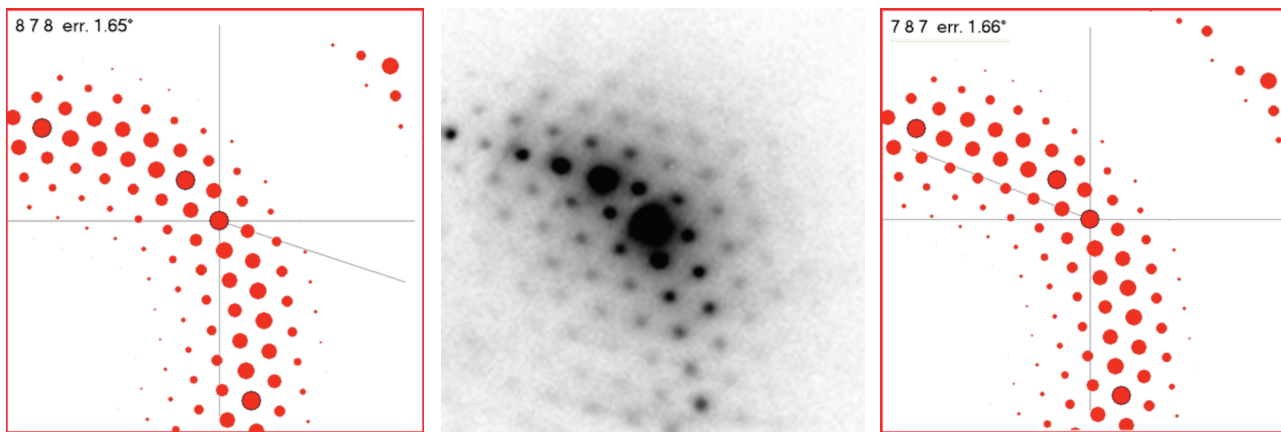
Consequently for every orientation, the procedure consists in researching the closest axis of symmetry, rotating the current orientation around this axis and checking the validity of the resulting orientation. In order to process any crystal from any space group, a systematic procedure has been established to calculate the set of symmetry axis.

Figure 2 demonstrate the capability of the post processing ambiguity correction. The transformed data produce a far more regular map where most of the spotty points were converted properly. The disorientations calculated for all pixels along the line shown on the left map prove that (i) the  $60^\circ$  jumps disappear and (ii) the orientation of the converted points are close to the one of the non corrected points.

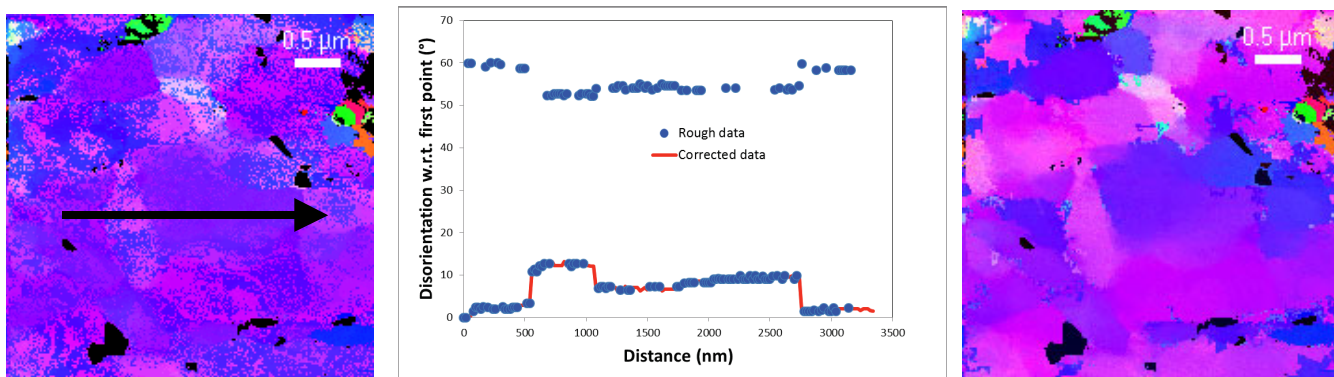
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[2] E.F. Rauch, J. Portillo, S. Nicolopoulos, D. Bultreys, S. Rouvimov and P.Moeck, Zeitschrift für Kristallographie: (2010) Vol. 225, issue 2-3 pp. 103-109

[3] Morawiec, A. and Bouzy, E. (2006) J. Appl. Cryst. 39, 101–103



**Figure 1.** An ambiguity arises when the acquired diffraction pattern (center) does not provide sufficient information to decide between two possible orientations (left and right) related by a  $180^\circ$  rotation around a highly symmetric axis (here the  $[111]$  axis for b.c.c. crystals).



**Figure 2.** Orientation maps before (left) and after (right) ambiguity corrections. The code color refers to the y axis (vertical). The frequent disorientation jumps of around  $60^\circ$  before correction (center) are related to a  $180^\circ$  rotation around the grain z axis that is close to  $[111]$ .