Towards automatic alignment of a crystalline sample in an electron microscope along a zone axis.

J Jansen[1], MT Otten[2] and HW Zandbergen[1]

1. National Centre for HREM, Kavli institute of NanoScience, Delft University of Technology, The Netherlands.
2. FEI Company, Eindhoven, The Netherlands.

email.contact.of.corresponding author : j.jansen@tudelft.nl
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In the past decade, major improvements of the mechanical and electrical stabilities of high-resolution electron microscopes (HREMs) in combination with the development of aberration correctors have resulted in the capability of HREMs to produce images with a resolution of 0.5 Å. To interpret such ultrahigh-resolution images, one needs to align the crystal along a zone axis with an accuracy higher than 0.1°. The orientation of a given crystal in the desired zone axis is currently set by the operator, who corrects the alignment visually using the symmetry of the diffraction pattern of a chosen area for guidance. However, this visual method does not yield a sufficiently accurate alignment. For one thing, the eye of the microscopist is unable to detect very accurately intensity differences. In addition, in the case of acentric zones, the correct orientation does not yield a diffraction pattern in which the Friedel pairs are necessarily equal. Indeed, a mistilt of more than 0.4° can easily be identified, but for a mistilt of less than 0.4° from a zone axis, accurate alignment by sight rapidly becomes more difficult.

If the sample is oriented approximately along a zone axis (see Figure 1a) the misorientation can be determined by means of a full structural refinement using the MSLS [1] procedure from the ELSTRU package [2]. This technique provides the required accuracy, but has the disadvantages that, apart from the unit cell, all atomic positions should be (approximately) known and the calculation is time-consuming (at least several minutes).

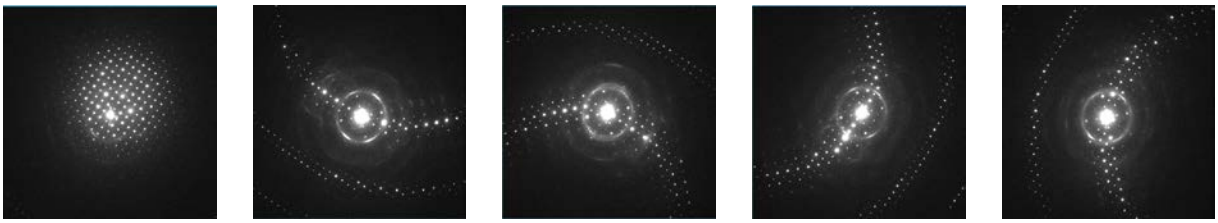
We show that even if the full structure is not known the orientation can be determined with a precision better than 0.1°. The actual deviation of the orientation of patterns like the one in Figure 1a is determined by recording several additional diffraction patterns using well-calibrated beam tilts over known angles in a few directions (Figure 1 b-e). From these additional patterns the deviation from perfect zone orientation is relatively easy to determine.

The method is based on the fact that in the tilted patterns (Figure b-e), Laue circles are visible. These circles can be determined by performing a peak-search and subsequently fitting a circle through it, using a weighting scheme based on the following facts: 1) A reflection reaches its maximum intensity if the orientation is such that the reflection is located exactly on the Laue-circle, and 2) intensities are damped (i.e. Debye-Waller-factor) if they are further away from the central beam. Laue circles determined in this way from only one picture may not be very accurate, because also diffraction spots are observed close to the Laue-circle. Analysis of many of these determinations shows that for the vector from the origin (central beam) to the centre of the Laue circle, its direction is much better determined than its magnitude. We exploit this feature by determining the centre of the Laue-circle for several beam-tilts, starting from the one to be determined, in 2 different directions. Subsequently we fit for each direction a line through the results (see figure 2). The intersection of the 2 lines will be the requested orientation. If 4 beam-tilts in each direction are used an accuracy of 0.03 degrees can be reached. This is far better that the 0.1 degrees accuracy that can be achieved with the sample tilt of modern microscopes.

To test whether the found mistilt values are correct, they were compared to values obtained from a full structure refinement using MSLS [1] (see table 1). This comparison shows that the measured misorientation is the same when either of the methods is used.

References

[1] J. Jansen et al, Acta Cryst., MSLS, A54 (1998), 91-101
[2] J. Jansen, http://nchrem.tnw.tudelft.nl/elstru/ (last accessed 15 March 2011)



a b c d e

Figure 1 *Diffraction patterns of the [100] zone of $Mg_{10}Ir_{19}B_{16}$. (a) manually set close to the zone and beam tilted over 3 degrees in +/– x-directions (b&c) and +/– y-directions respectively (d&e).*

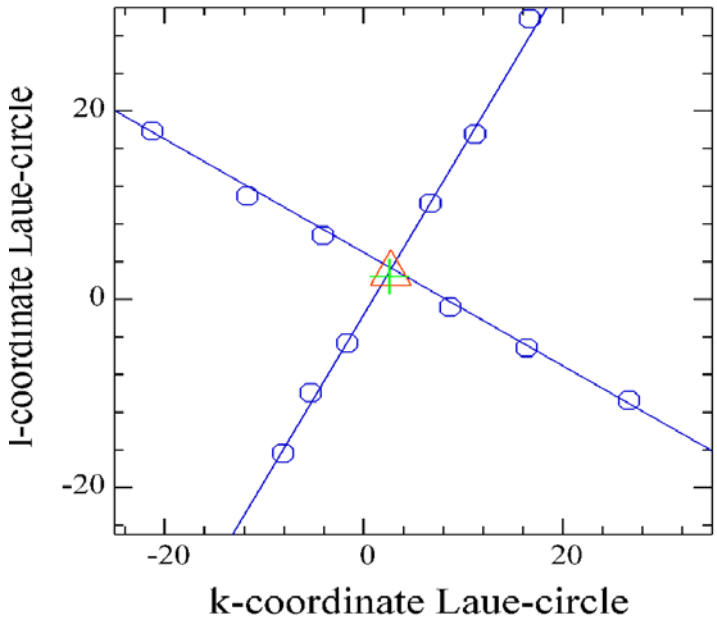


Figure 2 *Typical fitting result for the determination of the mistilt for $Mg_{10}Ir_{19}B_{16}$. ([100]) Determined misorientations for beam tilts of +/– 2, 3 and 5 degrees are marked with blue circles. The intersection point of the two lines gives the orientation. The red triangle shows the result from an MSLS refinement and the green plus is the result of a centre of gravity method.*

Compound	Zone	Tilt method mistilt (°)	MSLS-method mistilt (°)
Si	[101]	0.36(3)	0.36(4)
$SrIrO_3$	[001]	0.78(2)	0.791(7)
$SrIrO_3$	[111]	0.27(3)	0.259(4)
$YBa_2Cu_3O_7$	[010]	0.36(3)	0.41(2)
$Mg_{10}Ir_{19}B_{16}$	[100]	0.56(2)	0.548(8)

Table 3 *Comparison of some of the determined mistilt angles using different methods for different areas of various materials.*