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Image-based 3D Nanocrystallography by Means of Tilt Protocol/Lattice-Fringe Fingerprinting with Contemporary Side-entry Specimen Goniometers

Peter Moeck*, Bjoern Seipel*, Wentao Qin**, Eric Mandell***, and Philip Fraundorf***

* Department of Physics, Portland State University, P.O. Box 751, Portland, OR 97207-0751
** Technology Solutions, Freescale Semiconductor Inc., MD CH305, Chandler, AZ 85224
*** Department of Physics and Astronomy and Center for Molecular Electronics, University of Missouri at St. Louis, MO 53121

The feasibility of discrete atomic resolution electron tomography by means of exit-wave reconstruction in future aberration-corrected transmission electron microscopes (TEM) has recently been demonstrated for *individual* crystalline nanoparticles [1]. While fully eucentric highly precise specimen goniometers will be required for that novel technique to reach its full potential, image-based 3D nanocrystallography by means of tilt protocol/lattice-fringe fingerprinting can already be practiced with the current generations of TEMs and side-entry specimen goniometers. A Philips EM430 ST microscope equipped with a double-tilt goniometer that allows for ± 15° eucentric tilt and ± 10° non-eucentric tilt was, for example, used to determine the lattice parameters of sub-stoichiometric WC_{1-x} nanocrystals with the rock salt structure [2,3]. Due to this relatively small tilt range, only the so called "cubic minimalistic" tilt protocol, which requires a combined tilt of 35.3° about an effective <110> tilt axis that coincides with the eucentric axis of the specimen goniometer, could be employed. The angular tilt range (i.e. accessible region of orientation space) of a ± 20° tilt 360° rotation goniometer is nine times larger that that of a ± 20° double-tilt goniometer. A ± 20° double-tilt 360° rotation goniometer, on the other hand, possesses in addition to the approximately 1.4 times larger tilt range of the above mentioned tilt rotation goniometer the advantage of an extra degree of freedom to tilt an assembly of crystalline nanoparticles. This allows for concepts from classical crystallometry [4] to be applied to image-based 3D nanocrystallography [5]. This extra degree of freedom also allows for the alignment of the effective tilt axis of any tilt protocol parallel to the eucentric axis of the specimen goniometer. This alignment can be done in small angular increments so that a novel type of discrete atomic resolution electron tomography for an *ensemble* of crystalline nanoparticles can be practiced. The instrumental parameters for the applicability of this novel technique are directly interpretable resolution and available tilt range. Table 1 gives these parameters for a variety of existing and future aberration-corrected TEMs together with a measure for the viability of our new technique. We take as a measure for viability simply the number of different tilt protocols that are possible for the above mentioned WC_{1-x} nanocrystals. It is clear from this table that our method is practicable with currently existing equipment and will become much more viable in future aberration-corrected TEMs.

References

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TABLE 1. Parameters of current and future aberration-corrected TEMs for image-based 3D nanocrystallography by means of tilt protocol/lattice-fringe fingerprinting. The visible lattice fringe types and zone axes of WC_{1-x} nanocrystals for these parameters are also given. The number of possible tilt protocols - as given in the last column - may be considered as a measure of the viability of our novel discrete atomic resolution electron tomography technique for an *ensemble* of nanocrystals.

Spherical aberration coefficient, C_s, of objective lens [cm], (prototypes, kV)	**Directly interpretable (electron phase contrast, Scherzer) point to point resolution (x) [nm]**	**Relative resolution improvement RRI (x) = (1 – $^{x}/_{0.24\,nm}$) 100 %** (i.e. with respect to Tecnai G^2 F20 SuperTwin)	**Visible lattice fringe types* & zone axes,** i.e. lattice fringe crossings, **within one stereographic triangle [001]-[011]-[111]**	**Average angle between visible zone axes**	**Minimum double-tilt range requirement to achieve average angle between visible zone axes**	**Tilt range of a Gatan Model 925 double-tilt rotation goniometer****	***Number* and type of tilt protocols***** (i.e. a simple measure for the feasibility of our novel electron tomography method)
1.2 (Tecnai G^2 F20 SuperTwin, 200 kV)	0.24	0 %	{111} & [011]	60°	± 22.5°	± 30° eucentric tilt, ± 18° non-eucentric tilt	***one*** [011]-[110] tilt protocol
1.2 (Philips EM430 SuperTwin, (300 kV)	0.19	≈ 21 %	{111}, {200} & [001], [011]	50° (out of the 3 pairs in two stereographic triangles, [001]-[011]-[111]-[101])	± 18.4°	± 25° eucentric tilt, ± 15° non-eucentric tilt	***two*** [001]-<110> ***and the*** mc**** tilt protocol
0.5 (Tecnai G^2 F20 UltraTwin, 200 kV)	0.19	≈ 21 %	{111}, {200} & [001], [011]	50° (out of the 3 pairs in two stereographic triangles, [001]-[011]-[111]-[101])	± 18.4°	± 15° for both eucentric and non-eucentric tilts	***the*** mc**** protocol
≈ 0 (C_s -corrected Tecnai G^2 F20 SuperTwin, 200 kV)	0.12 (approaching the information limit)	50 %	{111}, {200}, {220}, {311} & [001], [011], [111], [112], [013], [114], [233], [125]	18.2° (out of the 14 pairs in one stereographic triangle)	± 6.5°	± 30° eucentric tilt, ± 18° non-eucentric tilt	***28*** different tilt protocols
≈ 0 (C_s and possibly also chromatic aberration-corrected TEAM***** project microscopes, 200-300 kV)	≤ 0.06	≥ 75 %	≥ 12 lattice fringe types, e.g. {111}, {200}, {220}, {311}, {331}, {420}, {422}, {511}, {531}, {442}, {620}, {622}, … resulting in ≥ 2^4 zone axes with [u + v + w] ≤ 8	≤ 9.3° (out of the 21 pairs of zone axes with [u + v + w] ≤ 8 in one stereographic triangle that are along {111}, {200}, and {220} bands	≤ ± 3.3° (when aiming only for those zone axes with [u + v + w] ≤ 8 that are along {111}, {200}, and {220} bands	no specification, but there could be space in the cm range in all 3 dimensions to construct fully (compucentric) goniometers with 3 degrees of freedom to tilt and rotate	***21*** different tilt protocols (when only aiming for those zone-axis pairs mentioned in rows 5 and 6), ***more than 50*** different tilt protocols without this restriction

***** Different types of lattice fringes have different crystallographic multiplicities; ** as communicated to us by Gatan Inc. in January 2005; *** note also that the wider the difference between the maximal combined tilt range and the required tilt for a certain tilt protocol is, the more orientation space can be assessed; **** mc stands for "minimalistic cubic", i.e. from <001> as revealed by crossed {020} fringes to <112> as revealed by a single set of {111} fringes, see refs. [2-3] and [5]; ***** TEAM stands for Transmission Electron Aberration-corrected Microscope, http://ncem.lbl.gov/team3.htm.

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