THE 3D PARAMETERS OF A (NANO)CRYSTAL FROM LATTICE IMAGES AT TWO TILTS

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Various techniques have been developed to infer the 3D relationship of reciprocal lattice vectors from electron diffraction patterns taken at different specimen orientations. Such relationship is subsequently applied in crystallographic analyses.[1] Due to the transmitted nature of the electron beam, TEM images present 3D information averaged throughout the thickness of the specimen on each micrograph. Techniques for acquiring 3D information from such images include stereomicroscopy for characterizing irregular dislocation networks,[2] and 3D reconstruction of non-periodic macromolecular assemblies.[3] Prospects for finding the 3D lattice parameters of nano-crystals from HREM images taken at different specimen orientations have been discussed.[4] Here we apply this stereo lattice imaging to a 7 nm crystal, and discuss its application to compact fcc and bcc lattices in general. Results on other lattice types will be published separately.

Generally 3 non-coplanar lattice vectors seen along 2 different zone axes are sufficient for inferring a subset of the 3D reciprocal lattice of a single crystal. Often these are adequate to infer the whole lattice. The goal of the experimental design is thus to look for 3 lattice spacings, within the first transfer-function pass-band, present along two zones whose angular distance is within the tilt limit of a TEM. Images with "aberration limits" r_a smaller than the analyzed spacings are specified, to lessen chances of missing other comparable (or larger) spacings in the exit-surface wavefield. Tilt protocols will vary with lattice type. For example, protocols for fcc and bcc lattices with lattice constant greater than $2r_a$ are given in Table I. Hence prior composition and microscopy data may be useful, to select the tilt protocol(s) likely to yield 3D parameter data for a given specimen.

To demonstrate, a thin film thought to contain WC_{1-x} (fcc a=4.248 Å) was tilted according to the fcc protocol in a Philips EM430ST TEM, with a crystal showing 90 cross-fringes, 45 to the effective tilt axis in the initial field of view. The 1st image was taken at an orientation of (t_1=15.0°, t_2=9.7°), and the 2nd image at (t_1=-15.0°, t_2=-9.7°), where t_1 and t_2 are goniometer readings on a Gatan double tilt holder. Later analysis confirmed the fcc indexing in Table I (Fig. 1). The direction of the (2, -2, 0) reciprocal lattice vector deviated from the effective tilt axis by 0.3°. The lattice parameters inferred are (a=2.98 Å, b=2.99 Å, c=2.96 Å, α=120.0°, β=58.7°, γ=119.8°), comparing favorably with those of WC_{1-x}, (a=3.00 Å, b=3.00 Å, c=3.00 Å, α=120.0°, β=60.0°, γ=120.0°) with spatial diagreement less than 1.5%, angular less than 1.5°. Thus the lattice parameters of an unknown fcc crystal might have been measured. Instead, the prediction of a WC_{1-x} lattice proved correct. With software support, the 3D lattice of arbitrarily small crystals may in this way be found in real-time.

References:
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2. P.Hirsch et al., *Electron Microscopy of Thin Crystals*, Krieger Publishing Co., pp.310-311 (1977)
3. J. Frank & M.Radermacher, *Adv. Tech. in Biol. Electron Microscopy*, Springer-Verlag, pp.1-72
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Table I. Tilt protocol for a single fcc or bcc crystal with lattice constant $a>2r_a$.

Lattice	fcc		bcc	
	From	To	From	To
Zone	[001]	[112]	[110]	[211]
Lattice Fringes	(200),(020)	(1, 1, -1)	(002), (1, -1,0)	(0,1,-1)
Amount of Tilt	35.3^o		30^o	
Effective Axis	(2, -2, 0)		(2, -2, -2)	

1

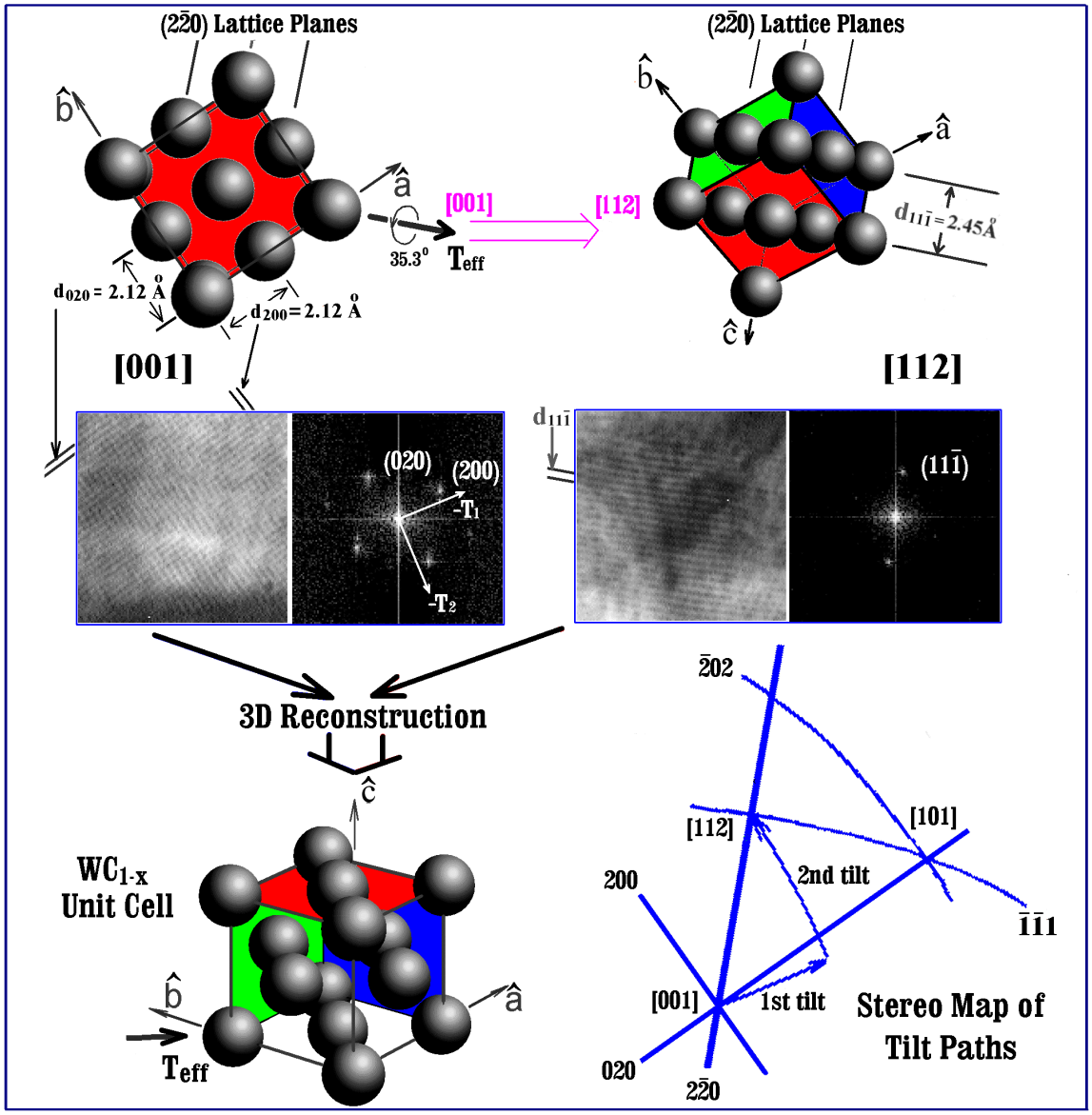

FIG. 1. Schematic illustrating application of the fcc stereo lattice tilt protocol of Table I to one crystal in tungsten-carbide thin film. Models show tungsten atoms only. The 1st tilt was around $-T_2$ by 19.4^o, the 2nd tilt, $-T_1$ by 30.0^o, where T_1 and T_2 are the side-entry goniometer tilt axis and the 2nd tilt axis, respectively.