LATTICE FRINGE VISIBILITY AFTER TILT

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Factors that affect the visibility of lattice fringes include crystal orientation and thickness, as well as instrument response. As a crystal gets smaller, lattice fringes stay visible for larger deviations from the Bragg condition. Hence the persistence of fringes under tilt affects the abundance and range of lattice spacings (and angles) that one sees in an image of randomly-oriented crystals. A subset of the fringes in an image are "still-visible" after large (e.g. 35°) single or double axis tilts.[1] If one is looking for "new-fringes" from the same crystal (e.g. to analyze it's 3D lattice parameters[2]), rules for recognizing redundant fringes might also help out.

Here we examine a semi-empirical model for predicting the visibility of lattice fringes after tilt, by connecting the visibility to intersection of the corresponding crystal reciprocal-lattice spot with the illuminating Ewald sphere. In particular, we assume that the fringe disappears if the angle between beam direction and lattice plane normal deviates from 90° by more than the half-angle $\alpha \approx \theta_B + \tan^{-1}[fd/t]$, where θ_B is the Bragg angle, d is lattice spacing, t is crystal thickness in the direction of the beam, and f is a specimen-dependent signal/noise factor of order 1. Based on the fraction of randomly-oriented crystals showing <001> fringes, for example, f for one Au/Pd specimen was shown[3] to be $\cong 0.95$. These assumptions allow one to predict, among other things, the range of azimuthal deviations of the tilt axis from the lattice plane normal (φ) for which fringes will remain visible, for any given goniometer tilt from the "plane edge-on" position (θ). We examine this question in the experiment that follows.

A tungsten carbide thin film specimen was deposited by PECVD on glass at a substrate temperature of 330^{o}C and a distance of 4cm from the vapor inlet. It was argon ion-milled for about 5 hours to perforation prior to the HREM study, at an incidence angle of 3^o. Both electron diffraction and X-ray diffraction studies indicated that the non-stoichiometric WC_{1-x} (f.c.c, a = 4.248 Å) is the dominant diffracting phase in the films, and Auger electron spectral analysis suggested a value of x between 0.4 and 0.8.[1, 4] Figure 1(a) is an image of the specimen, which was to be tilted by 14.5°. The tilt axis direction is marked with "T_1" which denotes the side-entry goniometer tilt axis. 13 sets of {200} and {111} lattice fringes of WC_{1-x} are seen and labeled. The angle between the normal of a set of lattice planes and the tilt axis was measured from Figure 1(a) and compared with φ_0, after which the fringe visibility in the second specimen orientation is predicted. A set of lattice fringes predicted to be visible in the second view is labeled with a solid arrow. Hollow arrows are used for those fringe sets that are predicted to disappear. Should the model prediction be consistent with experimental observation, only fringe sets labeled with solid arrows will remain visible after tilt. This is confirmed with the image taken after the specimen was tilted over 14.5° in Figure 1(b). Figure 2 is a plot of φ_o versus crystal diameter, together with the experimental data points. Similarly, solid symbols are used for fringe sets predicted to remain visible, and hollow symbols otherwise, after tilt. A consistency exists since all the solid symbols are below the corresponding curves, and hollow symbols, above. Both kinds of lattice fringes have been similarly observed in a double tilt where the effective tilt is 35.3° and the effective tilt axis is parallel to the images plane.

References:

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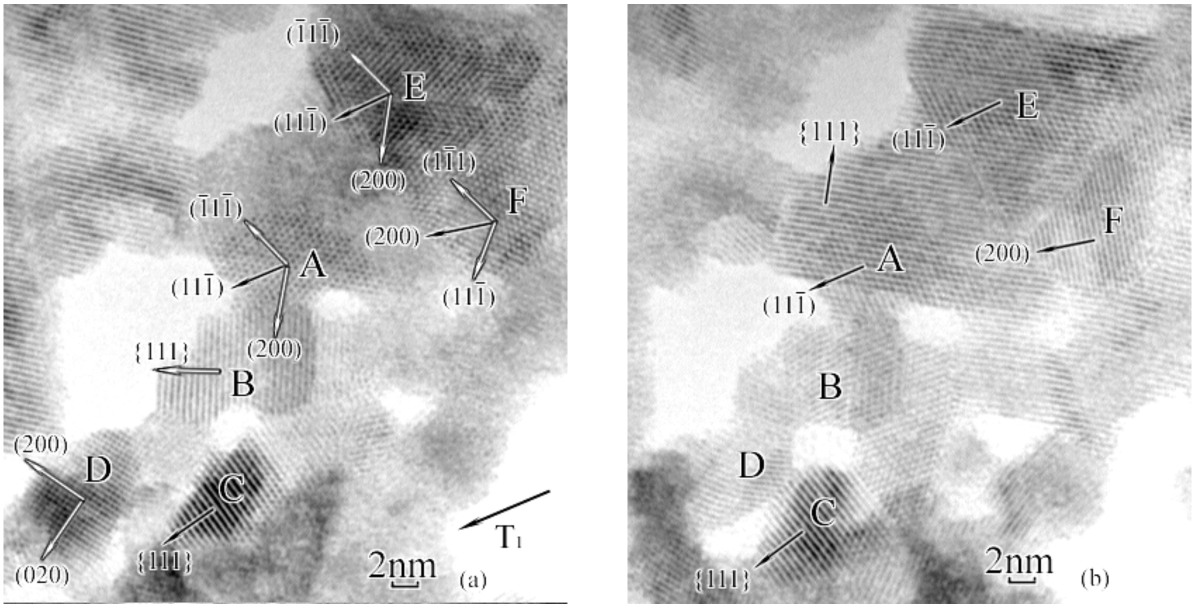
4. Private communication from W. James and W. Shih
5. Acknowledgements: Dr. James and Dr. Shih at U. Missouri-Rolla for the WC film



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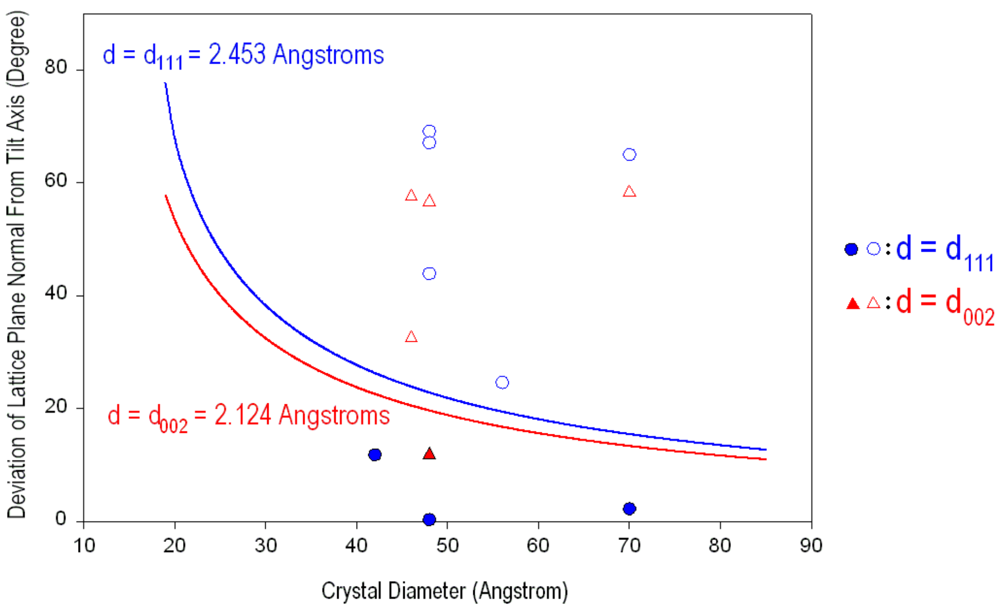

FIG. 1. HREM images showing lattice fringes in WC_{1-x} nanocrystals before, as shown in (a), and after, as shown in (b), tilting the specimen by 14.5°. In (a) a solid arrow along the normal of a set of lattice fringes denotes that this fringe set is predicted to remain visible, while a hollow arrow is used for a fringe set predicted to become invisible, after tilt. The arrow marked "T_1" denotes the direction of the tilt axis. A good consistency between the model prediction and the experimental observation exists, since all the fringe sets labeled with solid arrows in (a) remain visible after tilt, as shown in (b).

FIG. 2. Comparison of the model prediction of lattice fringe visibility after tilt, with the experimental observation shown in Figure 1. Two plots of the angular limits between the normals of {111} and {002} WC_{1-x} lattice planes and the tilt axis are shown as functions of crystal diameter, together with the experimental data. When the angle between a lattice plane normal and the tilt axis is less than the corresponding angular limit, this set of lattice fringes is predicted to remain visible after tilt, and the data point is represented with a solid symbol. Otherwise the lattice fringe set is predicted to become invisible, and a hollow symbol is used for data point representation. Please note that all the solid symbols are below, while all the hollow symbols are above, the corresponding curves.