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Nanobelt Thickness and Mean-free Path Determination by CBED and PEELS

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Recently, long ribbon-like one dimensional nanostructures of semiconducting oxides (see Fig. 1) were successfully synthesized using a physical vapor deposition technique [1]. Termed nanobelts, these structures can grow to lengths of up to 1mm, and have uniform thickness and virtually defect free structure, making them ideal candidates for electronic applications. It is known that the properties of nanostructures depend strongly on their size and shape [2]. Due to the material's nano-scale dimension, transmission electron microscopy (TEM) techniques on individual nanobelts are required to investigate their properties. In this study, thickness and mean-free path (λ) determinations for ZnO nanobelts were undertaken with the conjunction use of convergent beam electron diffraction (CBED) and parallel electron energy loss spectrometry (PEELS).

The details of the CBED procedure have been described by Allen [3]. Individual, hexagonal-structured ZnO nanobelts were first tilted under dynamical diffraction in the presence of Kikuchi lines, to two-beam conditions. The CBED (*0000*) and (*hkil*) discs were recorded for accurate measurement and indexed. Using the parallel, symmetric dark and light fringes found in the chosen diffracted disc (see Fig. 2), and their distances from the center of the diffracted disc (deviation from the Bragg angle, represented by ΔX), divided by the distance between the centers of the two discs (X), s_i was calculated, followed by the crystal thickness from the equation first described by Kossel and Möllenstedt:

$$(s_i^2 + 1/\xi_g^2)t^2 = n_i^2$$

where
s_i represents the deviation from the Bragg angle of the ith fringe
n_i is an integer for minima and a non-negative real number for maxima
ξ_g represents the extinction distance
t is the crystal thickness.

The values of n_i were determined by trial and error based on the best-fit straight line of the plotted s_i^2/n_i^2 vs. $1/n_i^2$ values. Rearranging the equation, the y-intercept is equal to $1/t^2$ and the slope is $1/\xi_g^2$. To confirm the accuracy of the thickness determination by CBED for nanobelts, the thickness was calculated using three separate indexed reflections from the same nanobelt (Fig. 3) and compared to the other two. The thickness was accepted when the three values were within 10% of each other. PEELS may be related to thickness by recalling that during beam-specimen interactions, inelastic scattering events increase with increasing specimen thickness. This is demonstrated as thickness dependence by the intensity of PEELS spectra. To extract the sample thickness information, we must measure the intensity of the zero-loss peak (I_o) and the entire low loss portion of the spectrum (I_T). The relative intensity of the zero-loss peak and the total intensity of the spectrum can be related to the mean-free path (λ) and sample thickness (t) by the following equation:

$$t = \lambda \ln\left(\frac{I_T}{I_o}\right)$$

Therefore, after determining the thickness of individual nanobelts via CBED, the electron mean-free

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path of the material was also calculated from a PEELS spectrum of the same nanobelt. The plot of Mean-free Path vs. Thickness (Fig. 4) indicates that within experimental error, the mean-free path of ZnO nanobelts is independent of thickness, and found to be 161 ± 15 nm at 200 kV.

References:

[1] Pan, Z., Dai, Z., Wang, Z., *Science,* **291**(2001), 1947-1949.
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[3] Allen, S.M., *Phil. Mag*., **A43:2**(1981), 325-335.
[4] This research was partially supported by the Georgia Tech Molecular Design Institute under contract N00014-95-1-1116 from the Office of Naval Research and NSF under DMR-9733160.

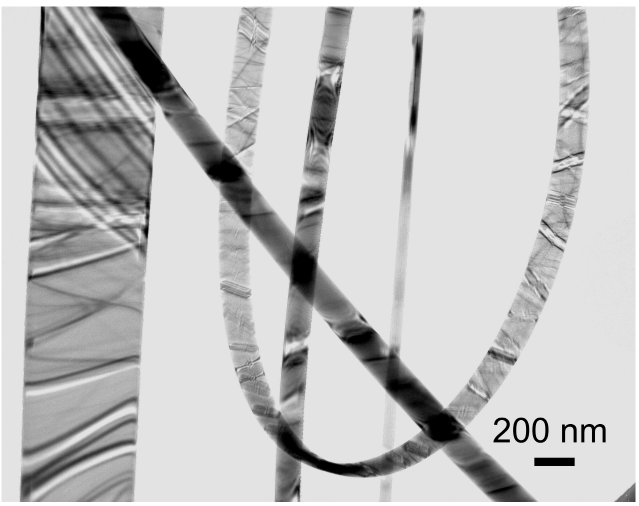

FIG. 1. Typical TEM image of ZnO nanobelts that exhibits a "ribbon" geometry.

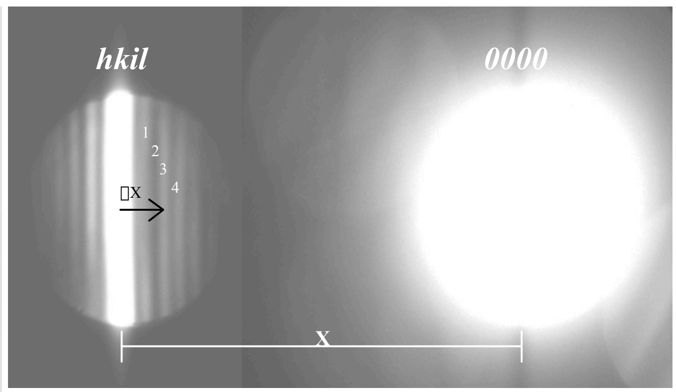

FIG. 2. Illustration of the measurements of X and ΔX in CBED.

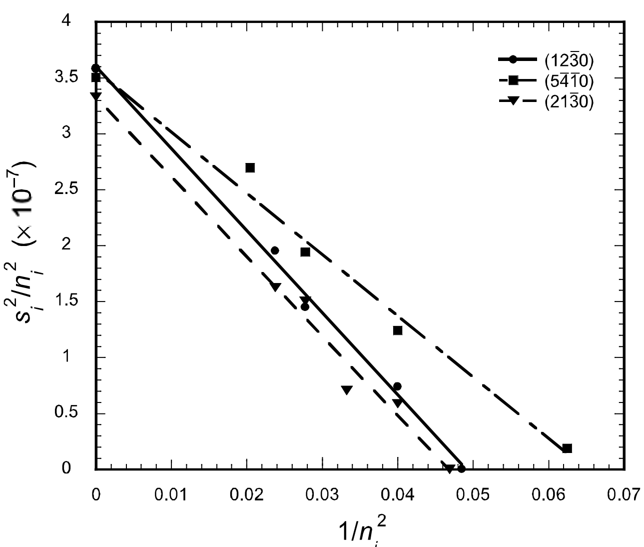

FIG. 3. s_i^2/n_i^2 vs. $1/n_i^2$ plot for three reflections acquired from the same nanobelt.

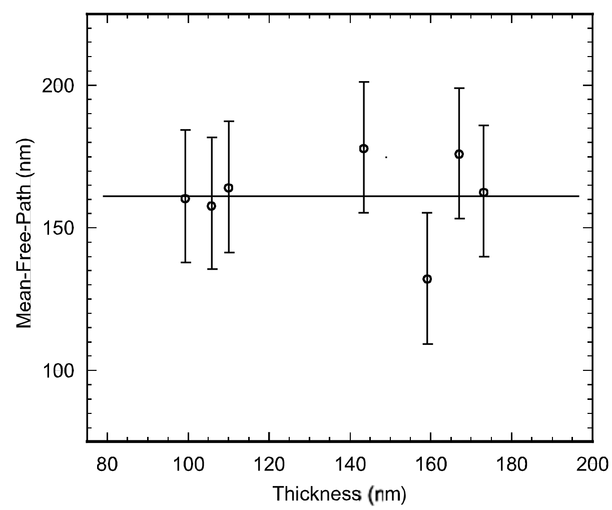

FIG. 4. Plot of experimentally determined mean-free path (λ) values versus thickness, for individual nanobelts; data are consistent and λ is independent of nanobelt thickness.