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Energy-Loss Characteristics for EFTEM Imaging with a Liquid Flow Cell

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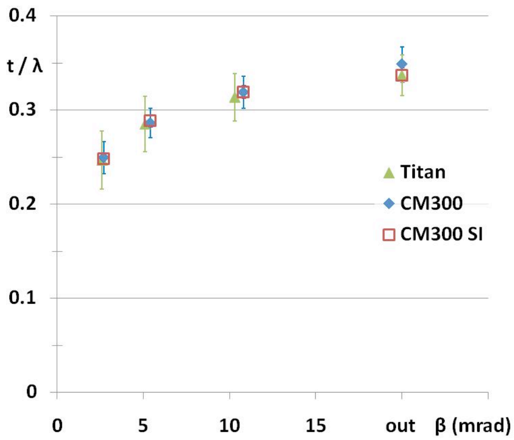
The imaging of microscopic structures at nanometer-scale spatial resolution in a liquid environment is of interest for a wide range of studies, with applications from nanotherapeutics to homogeneous catalysis. We have recently demonstrated that bright-field imaging in the transmission electron microscope (TEM) using a prototype liquid flow cell specimen holder provides a promising method for meeting this challenge [1]. However, the imaging characteristics for TEM are rapidly degraded with increasing projected thickness of the liquid cell, as the incident electron beam undergoes multiple elastic and inelastic scattering. In particular, a small collection semi-angle β and energy-selecting slit ΔE are required to maintain nanometer-scale spatial resolution in the face of spherical and chromatic aberrations, respectively, which greatly limits the available signal. In this paper, we report on various challenges for performing energy-filtered TEM (EFTEM) imaging in a liquid cell.

EFTEM zero-loss (ZL), low-loss (LL) and spectral images (SI) were acquired with incident energy $E_0 = 300$ keV and a small effective beam convergence semi-angle $\alpha < 1$ mrad. In order to separate the contributions of the amorphous silicon nitride (SiN_x) membranes from those of the encapsulated fluid, we began by measuring the variation of t / λ with β, as shown in Fig. 1. Data acquired with two instruments, and with t / λ maps formed both from the simple ratio of LL and ZL images and through extraction from a SI ($\Delta E = 5$ eV; $0 \leq E \leq 100$ eV), indicated a significant and reproducible variation, indicating that $\lambda(\beta)$ varies by ≈ 30 % as β increases from $\beta = 2.7$ mrad to $\beta = 10.8$ mrad. The higher image intensity that results from increasing the collection angle is thus rendered less useful by the increased proportion of inelastically scattered electrons.

The elastic deformation of the 50-nm-thick SiN_x membrane under the pressure differential from the liquid-filled cell to the vacuum presents a more significant challenge for liquid-cell imaging. A plot of the calculated bulge height h as a function of membrane radius r and thickness t, shown in Fig. 2, indicates that reducing the lateral dimension of the electron-transparent window provides the most effective strategy for minimizing the thickness variation across the window. An electron energy-loss (EEL) spectrum profile illustrates the dramatic effect of the bulging membrane on the energy-loss spectrum for an electron-transparent window of width 90 µm, as shown in Fig. 3. The EEL spectral shape evident near the edge of the membrane (2b) degrades into a featureless distribution of intensity with a peak energy and energy breadth of several hundred eV at the membrane center. The correspondingly large variation in intensity and specimen thickness are evident in Fig. 4 [3].

[1] K.L. Klein, I.M. Anderson, and N. de Jonge, *J. Microsc.* (2011) available online Jan. 20, 2011. doi:10.1111/j.1365-2818.2010.03484.x
[2] F.S. Tsai, *Characterization of Mechanical and Optical Properties of X-ray Mask Membranes*, RLE Technical Report No.564, Massachusetts Institute of Technology, Cambridge MA (1991).
[3] We are grateful to Protochips Inc. for access to their prototype TEM holder and for support from the National Research Council Postdoctoral Research Associateship Program (KLK).



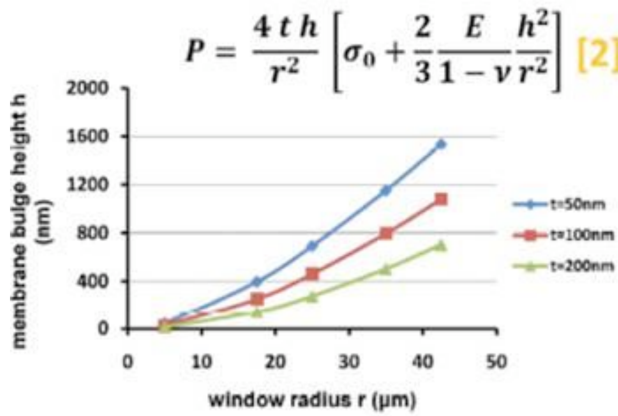


FIG. 1. Plot of measured t/λ values for 50-nm-thick amorphous SiN_x as a function of collection aperture semi-angle β. Since thickness t is constant, clearly $\lambda = \lambda(\beta)$.

FIG. 2. Pressure differential (P) dependence and calculated bulge height h as a function of radius r of electron-transparent SiN_x window of thickness t. Here, σ_0 is the residual ($P = 0$) stress, E is Young's modulus, and ν is Poisson's ratio for the SiN_x membrane [2].

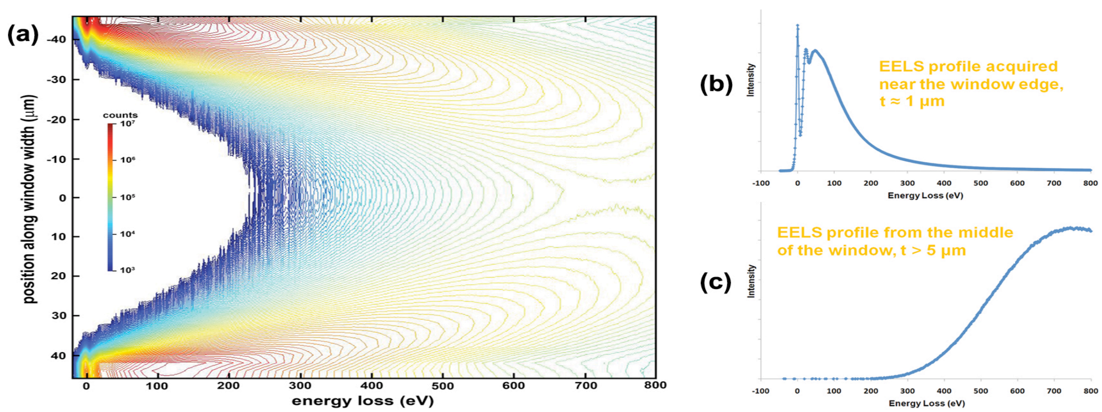


FIG. 3. Effect of membrane bulging on EEL characteristics of liquid cell: (a) spectrum profile contour plot, showing intensity as a function of energy loss and position relative to the edge of a 90-μm-wide SiN_x window; (b,c) EEL spectra from (b) edge (-45 μm) and (c) center (0 μm) of window.

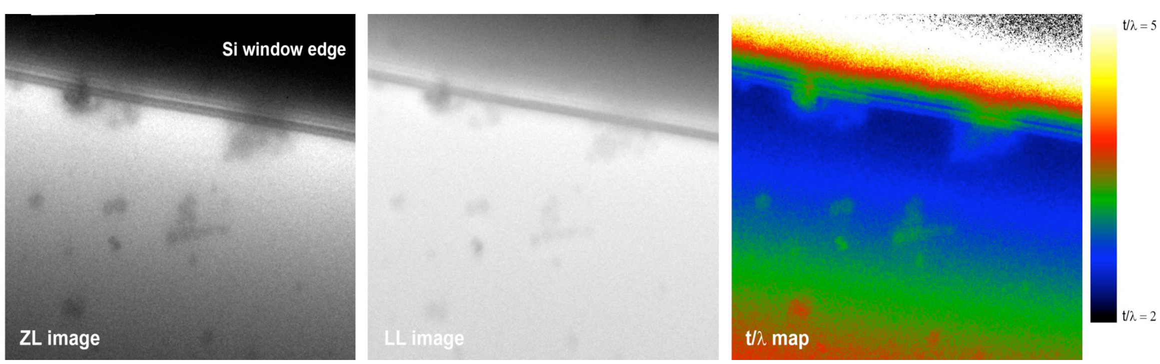


FIG. 4. Zero loss (ZL), low loss (LL) and t/λ images of citrate-stabilized gold nanoparticles suspended in the liquid cell of Fig. 2. For scale, images are 2.5 μm in height. The bulging increases the thickness of the cell by a factor of 2 within 2 μm of the window's edge, from $t/\lambda \approx 2$ to $t/\lambda \approx 4$.

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