

3D Tomography for Multiple-scattering Samples Using Phase Contrast Electron Microscopy

David Ren¹, Colin Ophus², Jihan Zhou³, Hannah DeVyldere², Michael Whittaker², Benjamin Gilbert², Jillian Banfield¹, Mary Scott², Jianwei Miao³ and Laura Waller¹

¹University of California-Berkeley, Berkeley, California, United States, ²Lawrence Berkeley National Laboratory, Berkeley, California, United States, ³University of California, Los Angeles, Los Angeles, California, United States

Electron Tomography is a crucial imaging technique that has made many contributions to understanding the structure and properties of chemical materials and biological samples. Previous works have demonstrated the ability to solve structures at very high resolution [1,2]. For example, high angle annular dark field scanning transmission electron microscopy (HAADF-STEM) has been used to reconstruct a sample at atomic resolution in 3D during different annealing times [2,3]. However, these existing tomographic methods often cannot uniquely solve the structure of dose-sensitive materials due to electron beam-induced damage at the dose required to resolve thick sample structure. In a recent work, we proposed a highly dose-efficient phase contrast imaging modality that can solve for 3D structures with high accuracy and a large field of view [4]. First, we combine phase contrast focal series high resolution transmission electron microscopy (HRTEM) at different tilt angles to lower the total dose needed during acquisition compared to HAADF-STEM. Then, we use the multi-slice algorithm to model the dynamical electron scattering process. The multi-slice algorithm allows us to eliminate the projection assumptions used in previous work, and to account for multiple scattering processes. We then formulate an inverse problem that is robust to low SNR images, and the pipeline is illustrated in Fig. 1. In simulation, we have shown that the method can retrieve 3D structure of both amorphous and crystalline structures at atomic resolution.

In this talk, we show application of our proposed method to image 3D samples at different resolution. Figure 2 (a) shows selected HRTEM measurements of montmorillonite (MMT) clays suspended in LiCl solution. They are of special interest because their 3D topology is tightly related to their behavior. In Fig. 2 (b), we reveal the 3D curvature of the clay layers. Next, we apply our method to atomic resolution HRTEM measurements of an oxidized tungsten tip, as shown in Fig. 2 (c). In this case, the sample is a composed of tungsten crystal core and a tungsten oxide shell. Figure 2 (d) shows the 3D tungsten tip atomic potentials solved using our proposed framework. Note the clearly resolved tungsten crystal core structure in the reconstruction. In order to reconstruct the 3D volume of the sample within a reasonable time, we deploy our algorithm to multiple GPUs, where each GPU covers a small patch of the full field-of-view. In order to achieve a more accurate reconstruction, we will explore methods to align the tilt-defocus series and to further validate the results.

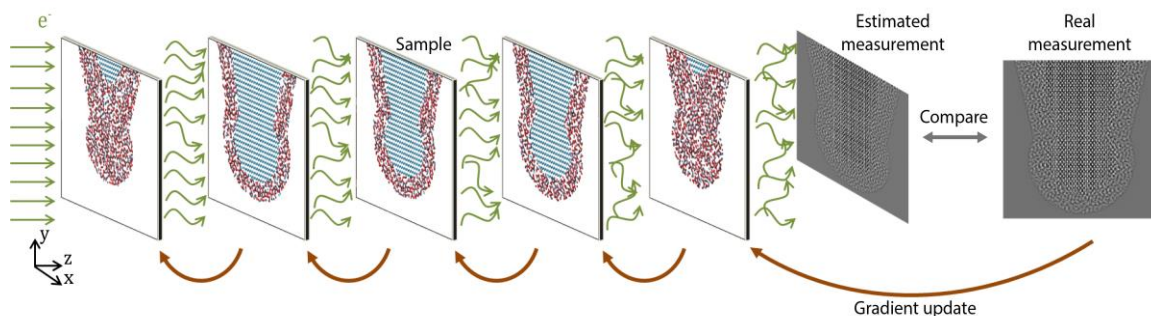


Figure 1. Schematic for multi-slice scattering algorithm. At each sample tilt, the 3D sample is modeled by many discrete layers. A plane wave incident on the sample propagates through layer by layer, as shown by the green arrows. A gradient step is taken from the data discrepancy with respect to each layer during reconstruction, as shown in red arrows.

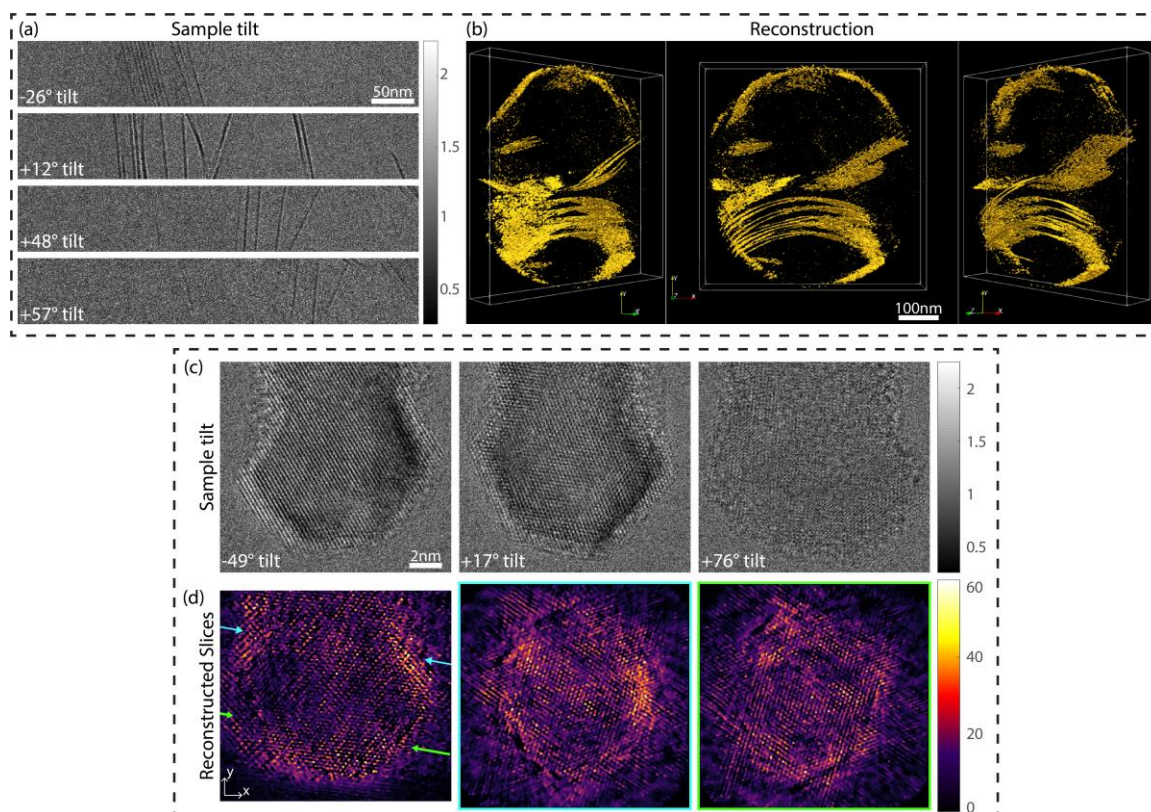


Figure 2. 3D reconstruction of montmorillonite (MMT) clay and atomic resolution tungsten tip from phase contrast high resolution transmission electron microscopy (HRTEM). (a) Images of clay recorded at various tilt angles with defocus of 500nm. (b) Reconstructed clay volume rendered in 3D. (c) Intensity images of tip recorded at various tilt angles with defocus of 75nm. (d) Slices of reconstructed tungsten tip volume showing individual atoms.

References

- [1] E Nogales, *Nature Methods* 13 (2016), p. 24-27.
- [2] Y Yang et al., *Nature* 542 (2017), 75.
- [3] J Zhou et al., *Nature* 570 (2019), 500-503.
- [4] D Ren et al., *Ultramicroscopy* 208 (2020), 112860.