

Confessions of a Ptychopath: Dose, Dimensions, Damage and Despair

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Electron ptychography has recently shown great promise as an atomic-resolution imaging technique for all elemental species in both two and three dimensions. However, each ptychographic step forward is typically a result of months (or years) of sample preparation, data acquisition and reconstruction refinement. In this presentation, we will summarize three of the main experimental difficulties associated with performing electron ptychography, and provide several examples of unsuccessful experiments which emphasize these challenges.

Detection: The correct use of fast pixelated detectors at appropriate electron dose levels and experimental settings is crucial to obtaining data that yields high-fidelity reconstructions. For example, by lowering the dynamic range of certain detectors, acquisition speeds can be increased tenfold [1]. Initial ptychography experiments using a 1-bit dynamic range were obtained from an MoS₂ monolayer (80 kV, 22.5 mrad) at an electron dose of 10,000 e⁻ Å⁻², resulting in low signal-to-noise ratios and poor fidelity phase reconstructions (Figure 1A). However, upon choosing a thicker sample with larger periodic features (e.g. ZSM-5) and using a smaller convergence angle (7.5 mrad at 300 kV), the contrast improved significantly, even at electron doses of 1,000 e⁻ Å⁻² (inset of Figure 1A) [2]. For 2D materials such as graphene and MoS₂, multiple frame acquisition and alignment can provide necessary signal to image single atoms [3]. With larger defocus values of the probe, 1-bit data acquisition becomes insufficient for sampling the fine features in the diffraction patterns.

Dimensions: The extension of ptychography to three dimensions has provided the promise of imaging single light atoms inside materials via inverse multislice methods, tomographic routines, or a combination of both [4, 5]. Focused-probe ptychography experiments of monolayer graphene yielded an undiscovered ‘defect’ revealed by electron ptychography and optical sectioning, as shown in Figure 1B. However, the depth resolution was insufficient to determine accurate atomic z-coordinates, and data acquisition speeds may have been slower than the movement of atoms within the defect system.

Damage: Obtaining sufficient signal while avoiding beam-induced sample changes is one of the greatest challenges to the ptychographic researcher. Recent experiments on gold nanoparticle superlattice assemblies were performed in an attempt to reveal the inter-particle ligand distribution (Figure 1C). Although some regions suggest ordering of ligands close to the nanoparticles (white arrows), gradual aggregation resulted in ligand motion during data acquisition. Future work will involve performing experiments under cryogenic conditions to mitigate ligand movement and prevent damage.

To conclude the presentation, recent progress on ptychographic atomic electron tomography experiments will be discussed.

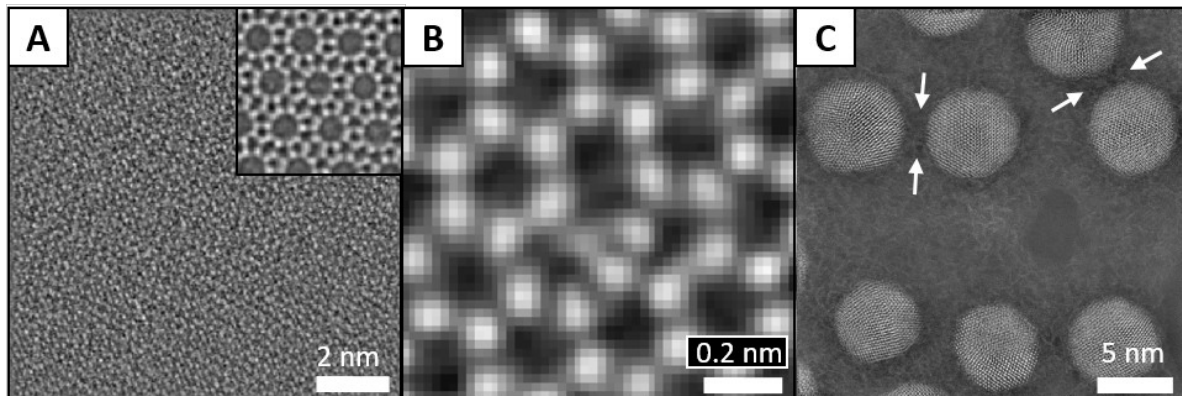


Figure 1. Ptychographic phase reconstructions of: **A.** 1-bit data obtained from an MoS₂ monolayer (main) and ZSM-5 zeolite (inset); **B.** a graphene defect analyzed via optical sectioning; and **C.** a ligand-capped gold nanoparticle superlattice assembly. Arrows in C indicate possible ordering of ligands.

References:

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