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Meeting-report

Visualizing Defects and Amorphous Materials in 3D with Mixed-State Multislice Electron Ptychography

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As devices continue to get smaller, reliable and dose-efficient 3D visualization of structures at the atomic scale is ever more critical. Here we show through both simulations and experiments how mixed-state multislice electron ptychography meets this challenge. Moreover, we show that ptychography outperforms both annular dark field (ADF) and integrated differential phase contrast (iDPC) through-focal imaging modes at recovering the structure of both crystalline and amorphous materials in 3D, even in the presence of aberrations and noise. Experimentally, we demonstrate the use of multislice ptychography for imaging crystalline materials, amorphous materials, interfaces in 3D, and buried defects. With a deep sub-Å lateral resolution and a depth resolution of a few nanometers, these results enable a new perspective for understanding the formation and nature of defects in materials and the nature of amorphousness in various compounds.

Generating a 3D image using conventional Scanning Transmission Electron Microscopy (STEM) imaging modes like ADF or iDPC requires acquiring a through-focal series from multiple scans of the same area at different defocus values, reducing the electron dose budget per scan. These methods are susceptible to multiple scattering and tilt artifacts, reducing the reliability and interpretability of features in depth [1]. In contrast, multislice ptychography enables a 3D reconstruction with better resolution in all dimensions from just a single scan in a more dose efficient manner.

For a quantitative comparison of these methods in recovering a known structure, we simulated 4D-STEM datasets of a model rough interface of c-Si/a-SiO_2/a-HfO_2 [1] using abTEM [2]. We compared the performance of multislice ptychography to through-focal ADF and iDPC for realistic experimental conditions. The atomic potential of the model interface was used as the ground truth for evaluating the different imaging methods (Fig. 1(A, B)). Ptychographic reconstructions were done using the fold_slice package [3].

Fig. 1(B) shows a comparison of the depth-sectioning capabilities of ptychography, through-focal iDPC, and through-focal ADF at both infinite and realistic dose. Not only do the simulation results show that ptychography outperforms both ADF and iDPC but also that it is robust to noise and aberrations. From intensity profiles along a single column and their FWHM values (Fig. 1(C)), we see that ptychography (green circles) provides the best resolution and accuracy in depth, while iDPC suffers from broadening and smearing artifacts and ADF is very sensitive to dose.

Experimentally, we imaged a c-Si/a-SiO_2/a-HfO_2 interface using an EMPAD-G2 detector [4]. Fig. 2(A) shows an ADF image focused on the surface of the sample, revealing heavy atoms there. In Fig. 2(B), the projection of the ptychographic reconstruction illustrates structural contrast within the amorphous regions. The internal structure of the amorphous hafnium oxide can be seen along with features in the amorphous SiO_2 layer in the depth section (Fig. 2(C)) corresponding to the dashed red line in Fig. 2(B). Moreover, heavy atoms on the surfaces of the sample (red arrows) are resolved in depth.

We have also imaged an extended defect in an ion-implanted bulk silicon sample using an EMPAD detector [5]. Fig. 2(D) showcases the ptychographic reconstruction of a zoomed in area of the defect. The leftmost panel is a z-projection of the 3D stack where the irregularities in the crystal might be misconstrued as tilt – a shortcoming of most projective imaging methods. Depth slices of the 3D reconstruction reveal defects embedded within the sample (Fig. 2(D) middle, blue) and show the regular crystal further into the bulk (Fig. 2(D) rightmost, green). A depth section along the yellow dashed line (Fig. 2(E)) exposes defects within the Si bulk. Yellow arrows point to the local re-arrangement of the Si dumbbells, information that is lost in projection. There have been many proposals for the possible structure of these defects, including a local hexagonal silicon phase [6]. Now, with 3D information from multislice electron ptychography we are able to obtain new detailed knowledge about these defects. In Fig. 2(D) we see a structure that is indeed consistent with a local region of hexagonal silicon phase. With this level of depth-resolved detail, we are now ready to delve into the realm of 3D defect structures in devices and answer long-standing questions about them [7].

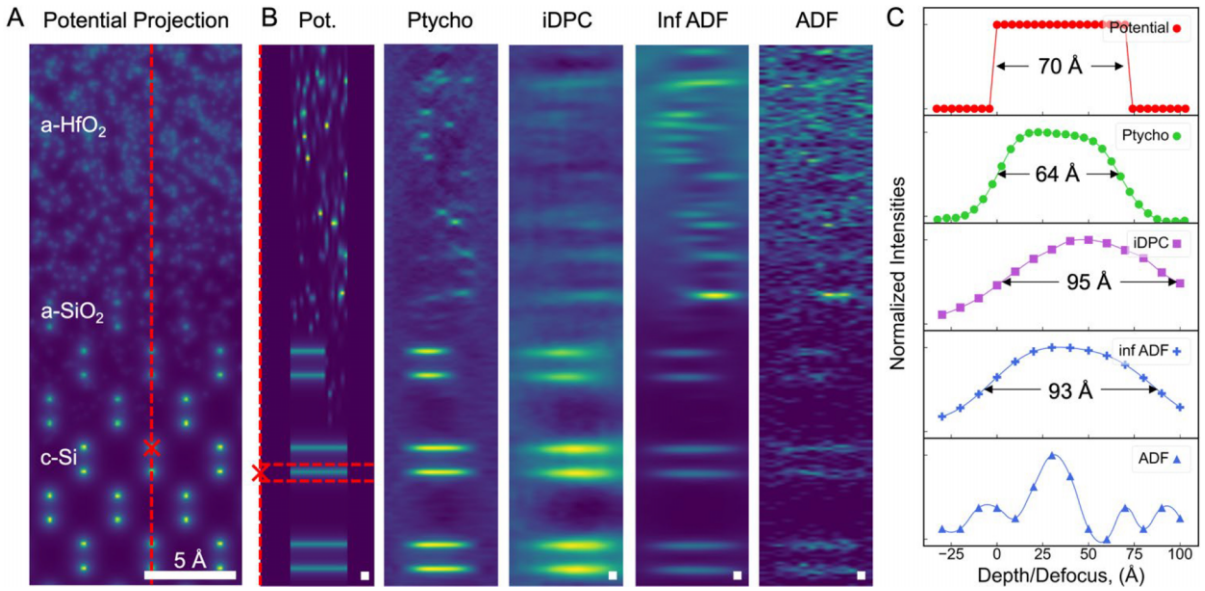

Fig. 1. Comparison of the depth sectioning capabilities of multislice electron ptychography, through-focal iDPC, and through-focal ADF. (A) Projection of the atomic potential of the model a-HfO_2/a-SiO_2/c-Si rough interface. (B) Depth section along the dashed red line in (A) (with 3.8 Å blurring in depth) and simulated results along the same line using various imaging modes. Spherical and chromatic aberrations and Poisson noise were added to the simulations for more realistic results; Inf ADF has no Poisson noise added. (C) Intensity profiles along the column marked with an x in (A) and a dotted red box in (B) with their FWHM values. Ptychography (green circles) gives the best resolution and accuracy in depth. Unannotated scale bars are 10 Å.

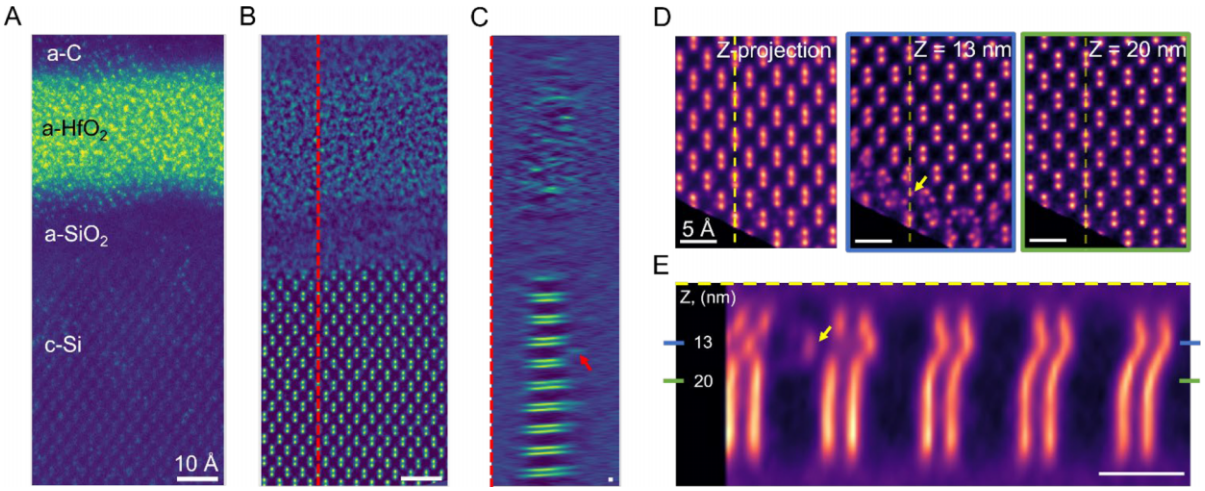

Fig. 2. Experimental results from a-HfO_2/a-SiO_2/c-Si interface (A-C) and extended defects in recrystallized Si (D-E). (A) Defocused ADF image of the interface showing heavy atoms on the surface of the thin lamella. (B) Projection of the ptychographic reconstruction showing structure in the amorphous regions. (C) Depth section along the dashed red line in (B) shows the internal structure of the a-HfO_2 amorphous region as well as some structure in the a-SiO_2 layer. Heavy atoms on the surfaces of the sample can be seen in depth (red arrow). (D) Ptychographic reconstruction of an extended defect in Si. Left panel is a z-projection of all the layers showing irregularities in the crystal that can be mistaken for tilt, next are slices of the 3D reconstruction revealing defects buried inside the sample (middle, blue) and the intact crystal further into the bulk (right, green). (E) Depth section along the yellow line in (D) reveals the defects in the Si bulk, including an out-of-phase boundary. The yellow arrows point at the local misorientation of the Si dumbbells, expected for the hexagonal phase. Blue and green lines indicate the depth of the corresponding slices in (D). (A-C) scale bars are 10 Å; (D-E) scale bars are 5 Å.

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