Scanning Nanobeam Electron Diffraction for Atomic Scale Tomography

Megan E. Holtz[1,*], Andrew A. Herzing[2], and Brian Gorman[1]

[1]Department of Metallurgy and Materials Engineering, Colorado School of Mines, Golden, CO, United States

[2]Materials Measurement Laboratory, National Institute of Standards and Technology, Gaithersburg, MD, United States

The goal of atomic scale tomography (AST) is to fully characterize the crystal structure and composition of a material atom-by-atom in three dimensions, enabling structure-property determination for materials. The combination of structural data from transmission electron microscopy (TEM) and compositional and structural information from atom probe tomography (APT) is routinely used as a powerful tool in materials characterization, and leveraging these techniques is an avenue towards AST [1]. Here we discuss characterization of crystal structure using scanning nanobeam electron diffraction (NBED) for atom probe specimens, and their strengths and limitations for AST.

To measure local interatomic positions in the crystal, we use a previously reported transform on scanning nanobeam electron diffraction: the exit wave power cepstrum (EWPC) [2]. The EWPC transform provides similar information to a pair distribution function (PDF). However, whereas the PDF is produced by calculating the Fourier transform of reciprocal space diffraction data, the EWPC is produced by first taking the logarithm of the diffraction data prior to applying the Fourier transform. Taking the logarithm suppresses artifacts related to the specimen orientation and thickness, which makes it robust across the depth of a cylindrical specimen. The resulting data give the spatially resolved lattice parameters and strain maps to picometer precision (Figure 1). The spatial resolution is limited by the probed volume, which in NBED mode is typically defined by the lateral spread of a 1 nm diameter probe as it transmits through the thickness of the specimen. The amount of this beam spreading depends upon the beam energy, specimen composition, and density.

We explore this strain mapping technique for cylindrical APT samples compared to conventional TEM cross-sectional lamella. The geometry of the specimen can potentially impact many strained materials of interest because once a small specimen is removed from the bulk material it may undergo strain relaxation as it is no longer constrained by the surrounding material. Lift-out and thinning the TEM lamella geometry can introduce bending and shearing to the specimen which may not be present in the high-symmetry cylindrical specimens for APT. This difference may enable us to see strain and dislocations running through materials without warping that may be present in thin TEM lamella.

To obtain crystallographic information about the sample in three dimensions, tomographic data using nanobeam electron diffraction is acquired (Figure 2). The strengths and limitations of using EWPC on tomographic NBED data to reconstruct strain profiles in three dimensions will be discussed. In particular, the criteria that imaging is projection-based for accurate tomographic reconstruction does not hold true for some types of peak-fitting algorithms within EWPC and we will investigate the implications of this for the fidelity of the reconstructed data.

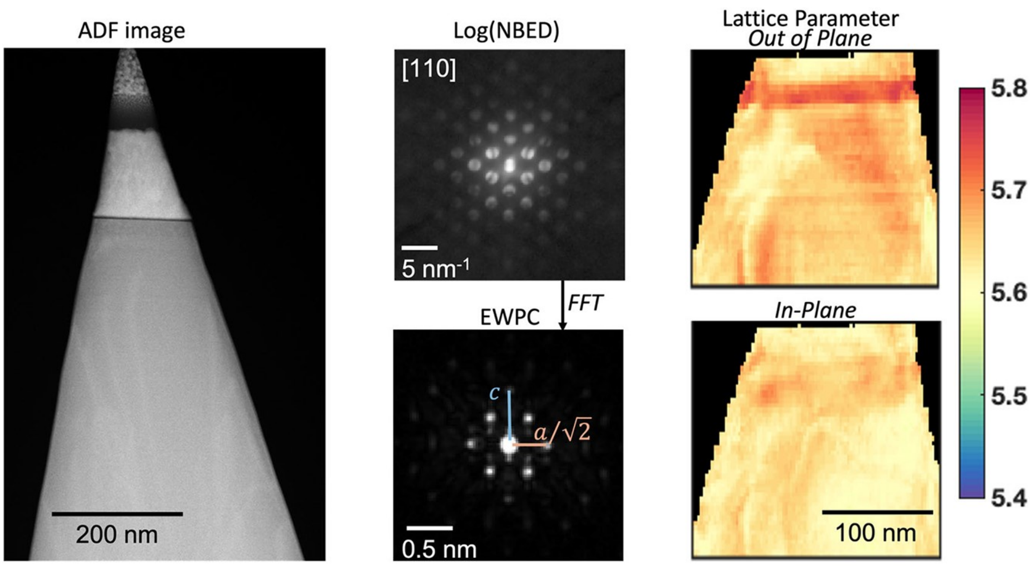

Figure 1: EWPC analysis for a quantum well structure. NBED and EWPC determines the out of plane (c) and in plane (a) lattice parameters.

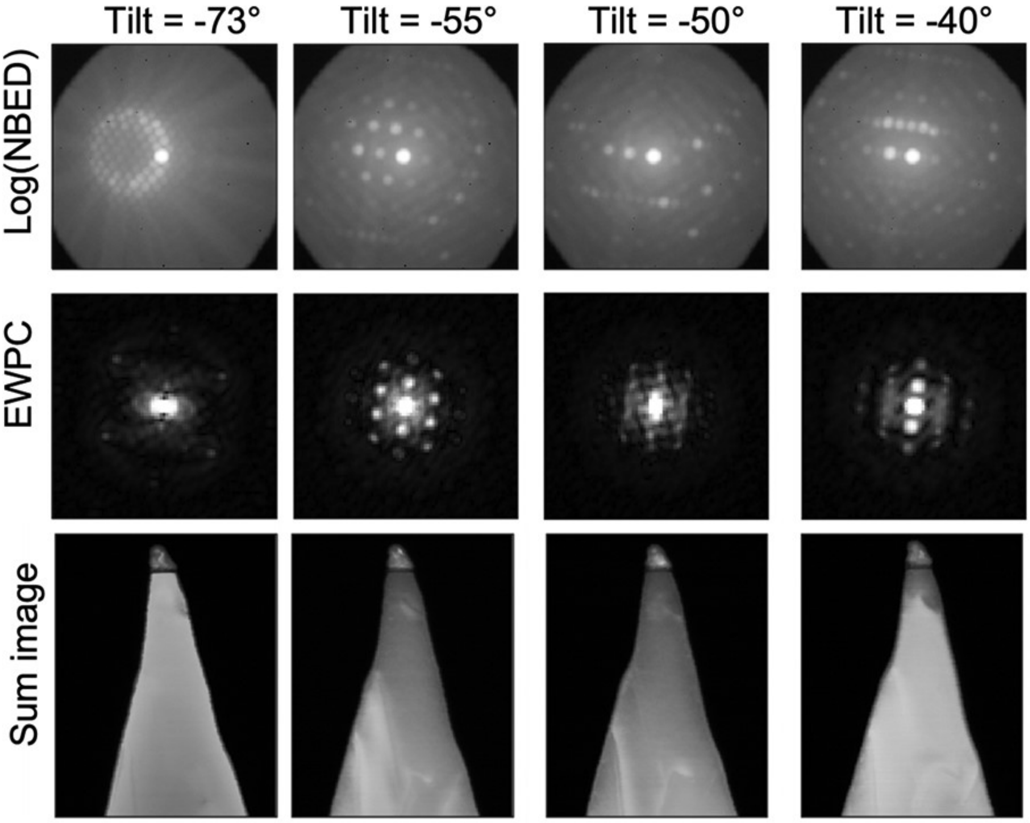

Figure 2: Excerpts from a tilt series of NBED data (top). Middle row: EWPC transform of the NBED data, showing spots for fitting even far away from zone axis. Bottom: Dark field images, with 100 nm horizontal field of view.

References

[1] Thomas F. Kelly, Brian P. Gorman, and Simon P. Ringer. In: *Atomic-Scale Analytical Tomography: Concepts and Implications*. Cambridge University Press, 2022.

[2] Elliot Padgett et al. In: *Ultramicroscopy* 214 (2020), p. 112994. DOI: `10.1016/j.ultramic.2020.112994`.