

Nondestructive, high-resolution, chemically specific 3D nanostructure characterization using extreme ultraviolet, coherent diffractive imaging reflectometry

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Abstract: A grand challenge in semiconductor metrology has been the nondestructive characterization 3D nanostructures and their multilayer structure, interfaces, and dopant concentrations. We combine extreme ultraviolet reflectometry with state-of-the-art ptychography imaging algorithms to achieve this goal. © 2023 The Author(s)

1. Introduction

The successful design and fabrication of next-generation semiconductor and quantum devices relies on an unmet metrology need. These devices are often carefully designed, doped multilayer structures, with critical dimensions of only a few nanometers. As critical features shrink below $\sim 10\text{nm}$, the device's functional properties deviate from the bulk and are no longer well-described by macroscopic models. This is because surface/interface effects such as roughness, oxide layers, and exchange interactions have thicknesses in this regime and therefore can significantly impact device performance, as can the particular concentrations and gradients of dopants within these devices. As devices shrink further and their complexity yet increases, it can be expected that even more properties of future devices will become dominated by surface and interface effects. Unfortunately, this connection between nanoscale structure and functional transport is extremely difficult to measure. Moreover, non-destructive measurements (or even better, in-situ) in working devices, though previously not possible for general samples, will be critical to further understanding its effect and optimizing the synthesis and integration of these systems.

To meet this need, we have developed a technique that combines extreme ultraviolet reflectometry (EUVR) with high-resolution ptychographic coherent diffractive imaging (CDI) [1,2], and can measure structure, dopant concentrations, and surface/interface quality in thin films and nanodevices, using a tabletop light source. We call this technique extreme ultraviolet coherent diffractive imaging reflectometry (EUV-CDIR). This technique builds on EUVR and ptychography, and has the benefits of each. EUVR measures the magnitude-intensity as a function of incidence angle and is sensitive to sample composition due to the many atomic transitions in this range of photon energies (12-120eV). Unfortunately, the spatial resolution of EUVR is limited to the beam size on the sample, typically 10's to 100's of microns. Ptychography provides the two-fold benefit of achieving an imaging resolution limited only by the numeric aperture of the camera, and also measuring the phase shift of the light reflected by the sample (dramatically improving sensitivity to composition and topography [3]). By combining the compositional and structural sensitivity of EUVR with the phase-sensitivity and resolution-enhancement of ptychographic imaging, EUV-CDIR is a unique, nondestructive, 3D sample-characterization technique whose resolution approaches the diffraction limit. Furthermore, we have developed two enhancements to the data collection and analysis that together can improve its achievable throughput by 100x or more.

To demonstrate the utility of EUV-CDIR, we have developed a reflectometer that can perform this technique in addition to more traditional reflectometry and diffractometry (see Fig. 1). The three modes of operation are complementary. Traditional, intensity-measuring EUVR (Fig. 1d) is useful for determining layer thickness and surface roughness in unstructured multilayer samples. To improve its efficacy, we designed and implemented a removable beamsplitter that can be used to improve the SNR of these measurements to nearly the shot-noise limit. Diffractometry (Fig. 1e) is useful for measuring a periodic array of nanostructures, and can determine their average shape and composition by measuring their diffraction efficiency as a function of incidence angle. Finally, coherent

diffractive imaging reflectometry (Fig. 1f) is able to measure the complex reflectance vs incidence angle at each transverse location on the sample (including non-periodic samples), with high resolution. This enables us to deduce the composition as a function of depth at each location on the sample (see Fig. 1c, [4]).

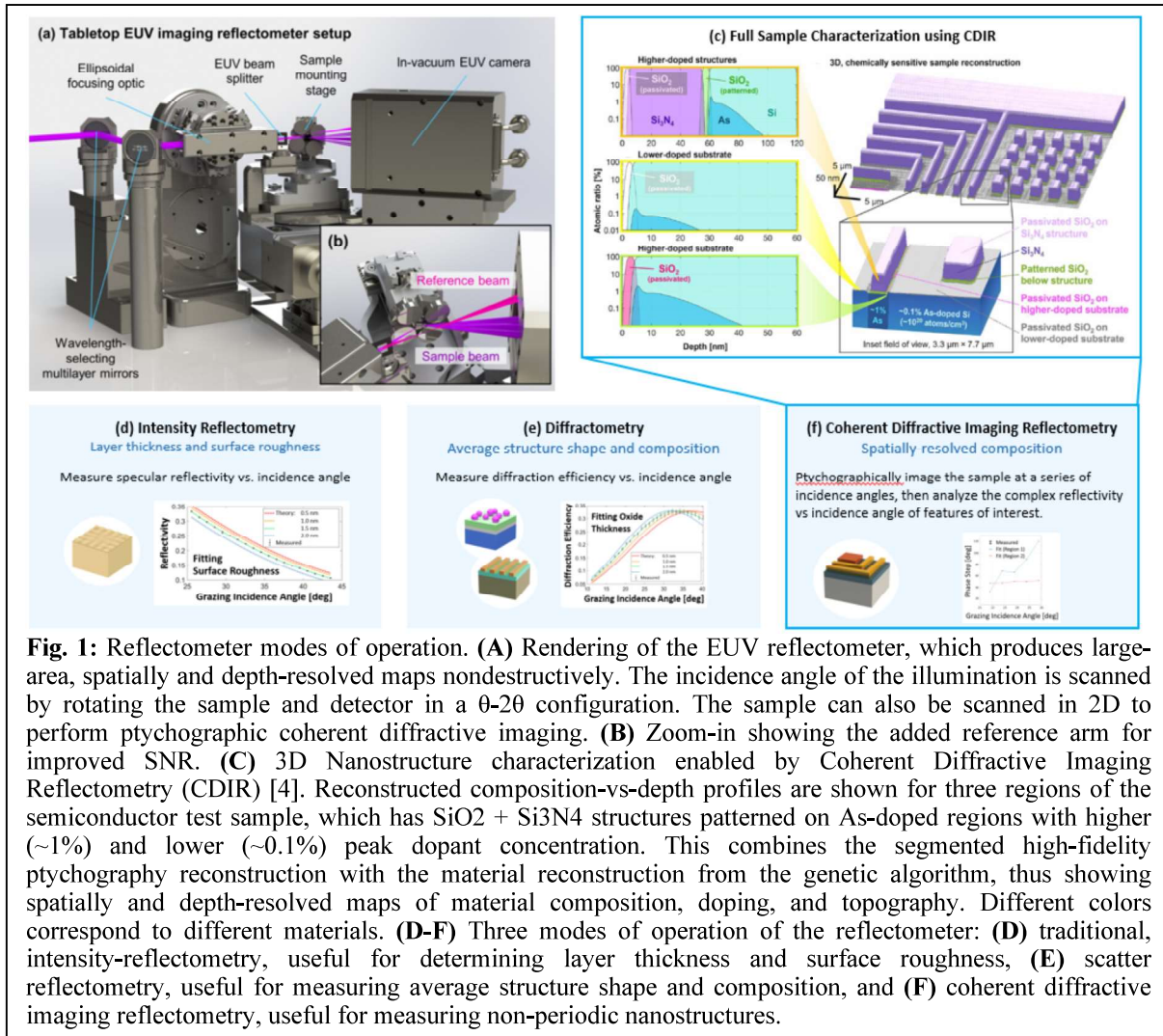


Fig. 1: Reflectometer modes of operation. **(A)** Rendering of the EUV reflectometer, which produces large-area, spatially and depth-resolved maps nondestructively. The incidence angle of the illumination is scanned by rotating the sample and detector in a θ - 2θ configuration. The sample can also be scanned in 2D to perform ptychographic coherent diffractive imaging. **(B)** Zoom-in showing the added reference arm for improved SNR. **(C)** 3D Nanostructure characterization enabled by Coherent Diffractive Imaging Reflectometry (CDIR) [4]. Reconstructed composition-vs-depth profiles are shown for three regions of the semiconductor test sample, which has SiO₂ + Si₃N₄ structures patterned on As-doped regions with higher (~1%) and lower (~0.1%) peak dopant concentration. This combines the segmented high-fidelity ptychography reconstruction with the material reconstruction from the genetic algorithm, thus showing spatially and depth-resolved maps of material composition, doping, and topography. Different colors correspond to different materials. **(D-F)** Three modes of operation of the reflectometer: **(D)** traditional, intensity-reflectometry, useful for determining layer thickness and surface roughness, **(E)** scatter reflectometry, useful for measuring average structure shape and composition, and **(F)** coherent diffractive imaging reflectometry, useful for measuring non-periodic nanostructures.

Our instrument has already been critical for measuring samples from industry, academe, and national laboratories, and fills a major gap in metrology for next-generation semiconductor and quantum devices. Furthermore, the success of this instrument has already inspired the development of similar devices in other universities, national laboratories, and in industry.

2. References

- [1] S. Döring et al. "EUV reflectometry for thickness and density determination of thin film coatings." *Applied Physics A* **107**, 795-800 (2012).
- [2] A. M. Maiden and J. M. Rodenburg. "An improved ptychographical phase retrieval algorithm for diffractive imaging." *Ultramicroscopy* **109.10**, 1256-1262 (2009).
- [3] E. R. Shanblatt et al. "Quantitative chemically specific coherent diffractive imaging of reactions at buried interfaces with few nanometer precision." *Nano letters* **16.9**, 5444-5450 (2016).
- [4] M. Tanksalvala et al, "Nondestructive, high-resolution, chemically specific 3D nanostructure characterization using phase-sensitive EUV imaging reflectometry," *Science Advances* **7.5**, eabd9667 (2021).