Annual Report FY2025

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1. Introduction

My work involves electron microscopy and holography of functional materials. The main focus is on novel high-Z semiconductor materials and devices, of particular interest for next-gen medical imaging and astrophysics applications. I am collaborating with external industrial partners for these semiconductor material studies. I also have active academic partnerships with Xi'an Jiaotong University, University of Alberta, and internally within OIST, involving electron microscopy and holography studies of various functional materials.

2. Activities and findings

2.1 Tellurium oxide microscopy and spectroscopy [1]

The first manuscript this year related to the study of tellurium oxide. Tellurium oxide is a critical layer in next-generation high-Z semiconductor devices, but prior studies are limited and many of its properties are poorly characterized and understood. Part of the challenge here is that tellurium oxide appears to decompose in a very erratic and unpredictable fashion, whether under material processing steps, or under exposure from the electron beam. With this in mind, I conducted a systematic experimental study of the electron beam induced phase-changes in tellurium oxide, as a function of various electron beam parameters.

Numerous unusual phenomena were observed, which had not been reported before for this material, and are documented in the paper. The mean fee path for 300kV electron scattering in [001]-oriented α-TeO_2 was measured as $\lambda = 155 \pm 6$m. A critical finding was that the material essentially became "immortal", tolerating an infinite cumulative electron dose, as long as the electron dose *rate* was kept below a defined threshold. A phase change from tellurium oxide to tellurium metal was also observed under certain exposure conditions, as illustrated in Fig. 1 (an example figure copied from the manuscript). Full details are included in the manuscript.

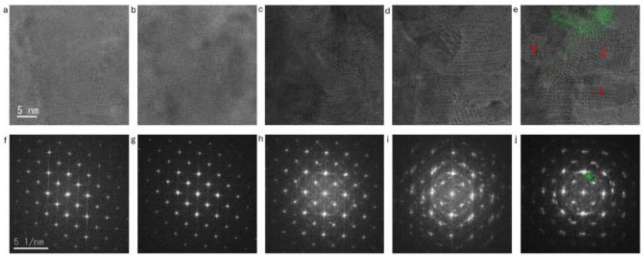

Fig. 7. HRTEM images and FFTs as a function of electron dose (snapshots from an 8.3 min extended exposure). (a,f) The pristine specimen at the beginning of the study. The ordered array of sharp white dots corresponds to oriented single crystal α-TeO_2 in [001] orientation. (b,g) At the early stages of the exposure, the spots quickly broadened, indicating that the crystal domain size is decreasing in real space, but otherwise the material remained [001]-oriented α-TeO_2. (c,h) There are very faint additional spots appearing in the FFT. (d,i) Now, there are very clear additional spots in the FFT. (e,j) The final frame in the image series, after 8.3 min (2.6×10^4 C/cm^2) exposure. Numerous small spots are present, which match randomly-oriented grains of Te and TeO_2. In (j), one labeled FFT spot, which matches elemental Te, has been mapped back into real space (e), showing the location of the elemental Te grain. Overall, a key observation is that the baseline single crystal [001] pattern also survived throughout the entire exposure. The labels 1, 2, 3 in (e) are mentioned in the text.

Figure 1 – an example figure from [1]. Atomic resolution HRTEM images showing a controlled phase change from oriented single crystals of tellurium oxide, to small grains of tellurium oxide and tellurium metal in a mosaic structure.

Overall, I proposed that the dominant factor in causing the unpredictable phase changes in tellurium oxide was the accumulation of beam-induced electric charge, and the creation of associated electric fields, in the material.

The manuscript was published in November 2025, as *Electron-beam induced damage in tellurium dioxide*, C. Cassidy, Micron 198 (2025) 103875. This was my first time to publish a single author experimental paper, which was an enjoyable new experience (if a lot of work 😊). It serves as an important foundation for more detailed quantitative microscopy studies on tellurium oxide, which have been progressing well in the meantime.

2.2 Electron holography studies of electrostatic charging under gas atmospheres [2]

Long-range electric and magnetic potentials are usually invisible with standard microscopy techniques. With electron holography, however, such potentials can be directly mapped and fully quantified. Electron holography is thus quite a powerful technique for nanoscale mapping of electric potentials in semiconductor devices.

The work reported here was performed in collaboration with Dr. Makoto Schreiber at University of Alberta in Canada. In conducting electron holography studies of specimens containing dielectric support films, we noted anomalous phase profiles which varied as a function of the electron beam illumination dose rate. Preliminary calculations suggested that the anomalous phase ramps were caused by charging of the dielectric films. We decided to explore flowing inert gas over the specimens in the TEM, to see if the charging could be actively compensated. The underlying idea is that the electron beam ionizes the inert gas, and the free charges can then move to the specimen-trapped charges via Coulomb attraction, and neutralize the charge on the specimen.

This worked beautifully – we could obtain direct visualization and quantification of the charge neutralization under gas atmosphere. Full details are described in the manuscript (an example figure is shown in Fig. 2 below).

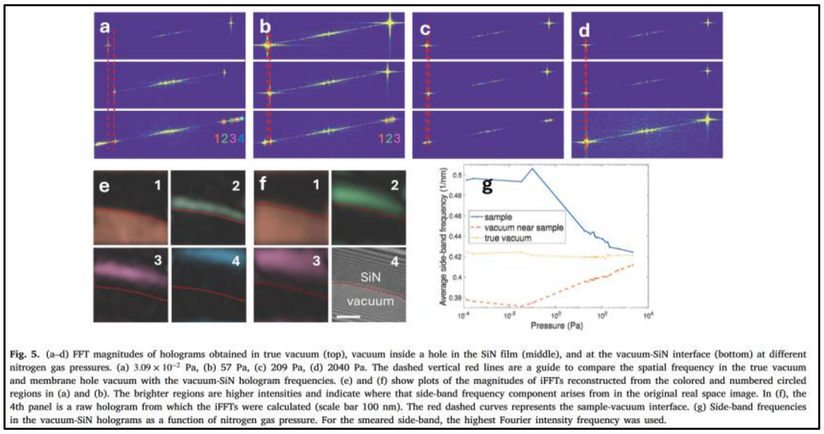

Fig. 5. (a–d) FFT magnitudes of holograms obtained in true vacuum (top), vacuum inside a hole in the SiN film (middle), and at the vacuum-SiN interface (bottom) at different nitrogen gas pressures. (a) 3.09×10^{-2} Pa, (b) 57 Pa, (c) 209 Pa, (d) 2040 Pa. The dashed vertical red lines are a guide to compare the spatial frequency in the true vacuum and membrane hole vacuum with the vacuum-SiN hologram frequencies. (e) and (f) show plots of the magnitudes of iFFTs reconstructed from the colored and numbered circled regions in (a) and (b). The brighter regions are higher intensities and indicate where that side-band frequency component arises from in the original real space image. In (f), the 4th panel is a raw hologram from which the iFFTs were calculated (scale bar 100 nm). The red dashed curves represents the sample-vacuum interface. (g) Side-band frequencies in the vacuum-SiN holograms as a function of nitrogen gas pressure. For the smeared side-band, the highest Fourier intensity frequency was used.

Figure 2 – an example figure from [2]. Electron holographic data acquired from a charging dielectric material, as a function of the flow of inert gas through the electron beam. We obtained clear proof that nitrogen gas flow reduces dielectric charging, and quantified the magnitude of the effect. Full details are included in the manuscript.

2.3 Other activities in FY2025 (work in progress)

Several other studies have progressed well during FY2025, and are at various stages of maturity:

- Microscopy and spectroscopy of diamond nanostructures (submitted).
- Analysis of Te-M/O-K peak overlaps in EDS studies of α-TeO_2 (experiments completed, manuscript in preparation)
- Cryogenic microscopy and spectroscopy of iridium oxide nanocatalysts (experiments completed, manuscript in preparation).
- Optimized lamella preparation for high-Z semiconductor device junctions (new protocol developed and documented)
- High resolution EELS studies of tellurium dioxide (in progress).
- Electron holography studies of Mean Inner Potential values in tellurium dioxide (in progress, but the results are quite unexpected, so it may take some time to figure out what is going on here).

3. Collaborations

- High-Z semiconductors
  Dr. B. Vogt-Wolz, Dr. M. Labayen de Inza, Dr. J. Wrege (Siemens Healthineers, Germany); Dr. J. Ellwood. T. Unten, N. Kishi (Acrorad Ltd, Okinawa)
- Gas electron holography
  Dr. M. Schreiber (NRC, University of Alberta)
- Holography of magnetic monopoles
  Prof. N. Shannon (OIST); Dr. A. Dhar (Stanford)
- Diamond microstructure electron microscopy
  Dr. Stoffel Jansens (OIST)
- Iridium oxide catalyst investigations
  Dr. Chenfeng Ding (Xi-an Jiaotong University), Dr. Alec LaGrow (OIST)

4. Publications and other output

Peer reviewed publications:

[1] C. Cassidy, *Electron-beam induced damage in tellurium dioxide*, Micron 198 (2025) 103875.

[2] M. Schreiber and C. Cassidy, *Study of gas-based charge compensation in an open-cell environmental TEM by off-axis electron holography*, Micron 199 (2025) 103896.

(both manuscripts were contributed by invitation to a special issue: *Quantitative and precise measurements in (scanning) electron microscopy and electron tomography*, in memory of a colleague, Dr. Misa Hayashida, who passed away in 2024).

5. Funding

External: Collaborative Research Agreement with external industrial partners (CRA4, 10/24-09/25). Details and research plan can be provided on request.