SLS Symposium on

High resolution Imaging and Soft X-rays

Tuesday, March 13, 2018

10:00 to 12:15, WBGB/019

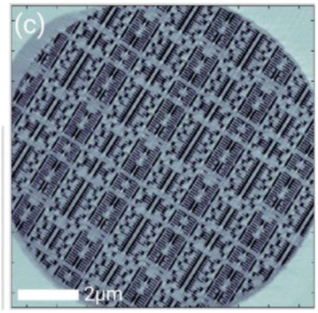
10:00 High resolution 3D imaging of integrated circuits by X-ray ptychography
Michal Odstrcil, M. Holler, J. Raabe, M. Guizar-Sicairos

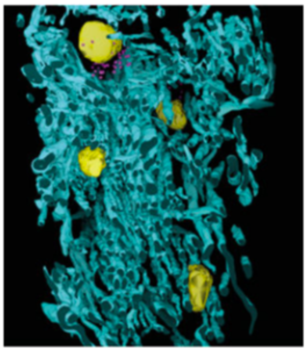
10:30 3D structures revealed by X-ray ptychographic tomography
Esther H. R. Tsai, M. Holler, A. Diaz, A. Menzel, and M. Guizar-Sicairos

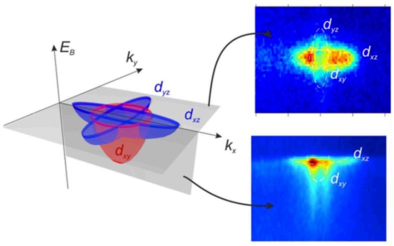
11:00 Coffee break

11:15 Orbital character of the mobile and localized electron states at the LAO/STO
Alla Chikina, F. Lechermann, M. A. Husanu, M. Caputo, Th. Schmitt, M. Radovic, and V. N. Strocov

11:45 Combining Resonant Inelastic X-ray Scattering (RIXS) with spatial resolution using a new zone-plate analyser setup
Florian Döring, F. Marschall, Z. Yin, B. Rösner, M. Beye, P. Miedema, K. Kubiček, L. Glaser, J. Soltau, V. A. Guzenko, J. Buck, M.Risch, D. McNally, M. Dantz, X. Lu, V. Strokov, T. Schmitt, S. Techert, C. David







High resolution 3D imaging of integrated circuits by X-ray ptychography

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Significant advances in lithography and chip manufacturing in recent years have resulted in new challenges in metrology and analysis of electronic microdevices. Manufacturing processes for the 10 nm node are already available and in combination with complex three-dimensional structured interconnections, there is a lack of methods for verification that the final products correspond to the original specifications. The traditional metrology methods such as destructive techniques based on delayering of ICs and imaging by scanning electron microscopy are close to their limits already for the current lithography processes and additionally delayering and imaging of a large chip is a time-consuming and destructive process, where important features of the IC can be lost.

One of the methods that have the potential to alleviate these issues is high resolution X-ray nanotomography. Due to high penetration of the X-ray light, it is currently possible to image interior structures of the IC at 15 nm isotropic 3D resolution[1] with future outlook to sub-10nm resolution by ptychographic X-ray computed tomography (PXCT). PXCT is a locally nondestructive imaging method that enables to obtain detailed device geometries and corresponding elemental maps. However, in order to tackle the experimental challenges and improve the imaging quality, new computational methods need to be developed for both ptychography and tomography that can account for a wide range of imperfections such as sample drifts, illumination changes or sample changes during the scan. In our presentation, we will discuss some of the potential directions for future developments and current challenges that need to be tackled in order to reach reliable sub-10nm 3D resolution for highly scattering samples such as ICs.

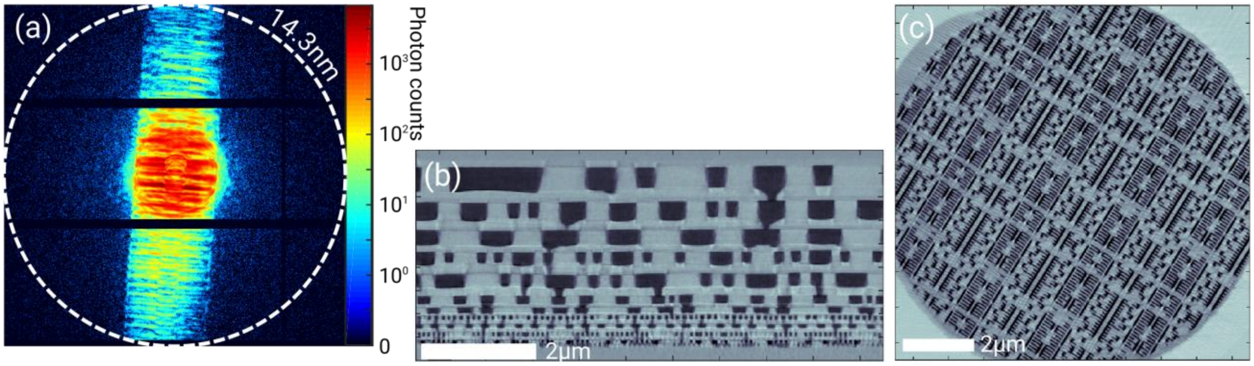

Figure 1 (a) Example of measured diffraction pattern for 100ms exposure time at 6.2keV for an Intel chip produced by 22nm technology[1] measured at cSAXS, (b,c) cuts through PXCT reconstruction of electron density of the same sample

References

1. Holler, M. *et al.* High-resolution non-destructive three-dimensional imaging of integrated circuits. *Nature* **543,** 402–406 (2017).

3D structures revealed by X-ray ptychographic tomography

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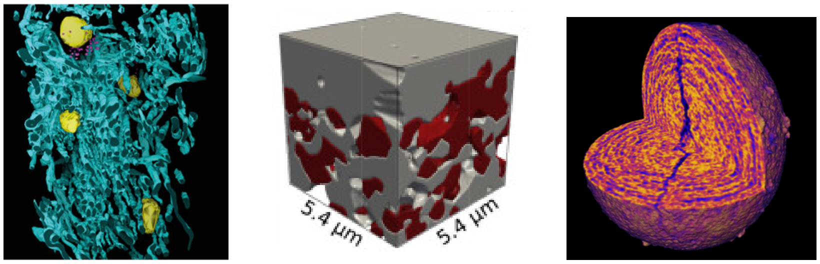
Ptychography is a coherent diffractive imaging method that offers a high resolution that is not limited by the focusing or imaging optics. The sample is illuminated at overlapping regions while at each sample position a diffraction pattern is recorded. An iterative algorithm is then used to retrieve the amplitude and phase information of both the illumination and the sample. ptychographic X-ray computed tomography (PXCT) provides quantitative electron density tomograms of extended system at spatial resolution [1,2] levels hardly achievable by common X-ray microscopy techniques. Here we show examples where the method allows for the characterization of various materials, from physiological-relevant features in bulk frozen-hydrated tissues [3] to tracking morphological changes in energy materials [4]. We also discuss on-going developments, including the depth-of-field limitation and multi-slice methods [5,6].



(a) (b) (c)

Fig. 1. Examples of 3D structures obtained via PXCT. (a) Thick biological brain tissue imaged in frozen-hydrated state without staining for identifying physiologically relevant features. (b) Tracking structural evolution of solid oxide cell electrode during a complete redox cycle. (c) Observation of morphological changes for aging lithium-ion battery cathode materials.

Acknowledgment

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References

1. M. Holler, A. Diaz, M. Guizar-Sicairos, P. Karvinen, E. Färm, E. Härkönen, M. Ritala, A. Menzel, J. Raabe, and O. Bunk, "X-ray ptychographic computed tomography at 16 nm isotropic 3D resolution," Sci. Rep. **4**, 3857 (2014).
2. M. Holler, M. Guizar-Sicairos, E. H. Tsai, R. Dinapoli, E. Müller, O. Bunk, J. Raabe, and G. Aeppli, "High-resolution non-destructive three-dimensional imaging of integrated circuits," Nature **543**, 402 (2017).
3. S. H. Shahmoradian, E. H. R. Tsai, A. Diaz, M. Guizar-Sicairos, J. Raabe, L. Spycher, M. Britschgi, A. Ruf, H. Stahlberg, and M. Holler, "Three-dimensional imaging of biological tissue by cryo x-ray ptychography," Sci. Rep. **7**, 6291 (2017).
4. S. De Angelis, P. S. Jørgensen, V. Esposito, E. H. R. Tsai, M. Holler, K. Kreka, E. Abdellahi, and J. R. Bowen, "Ex-situ tracking solid oxide cell electrode microstructural evolution in a redox cycle by high resolution ptychographic nanotomography," J. Power Sources **360**, 520–527 (2017).
5. E. H. R. Tsai, I. Usov, A. Diaz, A. Menzel, and M. Guizar-Sicairos, "X-ray ptychography with extended depth of field," Opt. Express **24**, 29089–29108 (2016).
6. P. Li and A. Maiden, "Multi-slice ptychographic tomography," Sci. Rep. **8**, 2049 (2018).

Orbital character of the mobile and localized electron states at the LAO/STO interface

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The discovery of high mobility two-dimensional electron system (2DES) emergent at the interface between two wide band-gap insulators $LaAlO_3$ (LAO) and $SrTiO_3$ (STO) has put forward new perspectives of oxide electronics where interfacing different materials tunes the interplay of electron, spin, orbital and lattice degrees of freedom characteristic of the transition-metal oxides. Oxygen vacancies (V_Os) at the LAO/STO interface leave two vacant electrons, one of which stays localized at the Ti^{3+}ion and another joins the mobile 2DES. This forms a dichotomic electron system where the localized strongly correlated in-gap electrons coexist with the less correlated mobile ones.

We use resonant soft-X-ray ARPES experiments at the ADRESS beamline of Swiss Light Source to establish orbital character of the mobile and localized electron states at the LAO/STO interface created by the V_Os. We identify the predominantly Ti e_g- vs t_{2g}-derived orbital character of these two electron systems. DFT+DMFT calculations agree with the experiment on both energy position and orbital character of the localized and mobile electrons. This finding of a crosstalk between the localized and mobile electron systems sheds new light of the mechanism of magnetism and superconductivity at the LAO/STO interface.

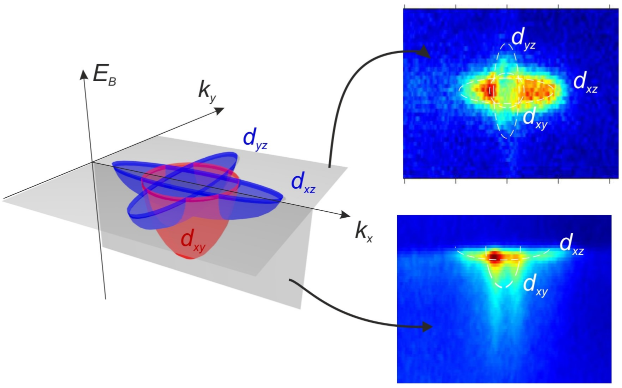

FIG. 1: Schematic dispersions of the 2DES 3d states including Fermi surface map and high-symmetry cut.

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[1] F. Lechermann et al., Physical Review B **90 (8)**, 85125 (2014).
[2] C. Cancellieri et al., Nature Communications **7**, 10386 (2016).

Combining Resonant Inelastic X-ray Scattering (RIXS) with spatial resolution using a new zone-plate analyser setup

F. Döring[1], F. Marschall[1], Z. Yin[1], B. Rösner[1], M. Beye[2], P. Miedema[2], K. Kubiček[2], L. Glaser[2], J. Soltau[3], V. A. Guzenko[1], J. Buck[2], M.Risch[4], D. McNally[1], M. Dantz[1], X. Lu[1], V. Strokov[1], T. Schmitt[1], S. Techert[2,3], C. David[1]

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We have implemented and successfully tested off-axis transmission Fresnel zone plates as spectral analyzers for resonant inelastic X-ray scattering (RIXS) [1]. The imaging capabilities of zone plates allow for analysis of independent information in both dimensions of the detector at the same time: one dimension is used for energy dispersing the emitted radiation and allows for sub-eV resolution RIXS, while the orthogonal axis images the sample surface with sub-µm resolution and consequently enables analyzing sample structures. Thus, by scanning a line focus across the sample in one dimension, we efficiently recorded RIXS spectra spatially resolved in 2D, increasing the throughput by two orders of magnitude.

Moreover, by varying the photon energy along a line focus on the sample, we were able to exploit the imaging capabilities of zone plates in order to simultaneously record the emission spectra over a range of excitation energies, enabling advanced two-dimensional (2D) mapping applications [2].

The presented scheme opens up a variety of novel measurements and efficient, ultra-fast time resolved investigations at X-ray Free-Electron Laser sources.

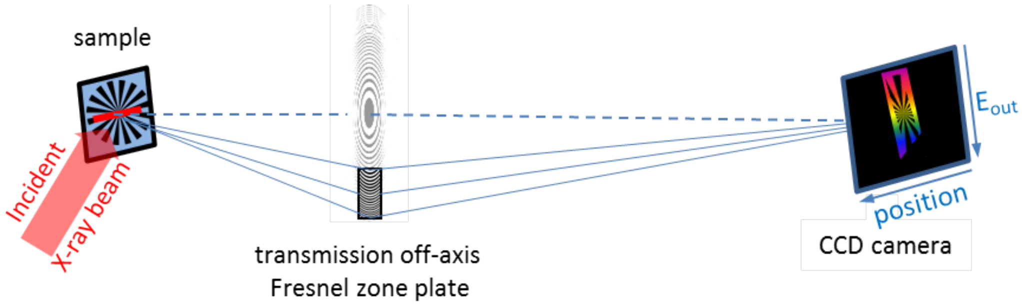

***Figure 1:** Sketch of the new RIXS implementation: A transmission off-axis Fresnel zone plate is used to collect the emitted light from the sample and disperse it along one axis of the detector while maintaining spatial resolution along the other axis.*

[1] F. Marschall et al., Opt. Express **25**, 14 (2017) (10.1364/OE.25.015624)
[2] F. Marschall et al., Sci. Rep. **7**, 8849 (2017) (10.1038/s41598-017-09052-0)