

**Electron Microscopy and Instrumentation** 

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11 – 12 December, 2023 | City University of Hong Kong, Hong Kong SAR, China
13 – 14 December, 2023 | City University of Hong Kong Shenzhen Futian Research Institute, Shenzhen, China

#### HOST INSTITUTES

Hong Kong Institute for Advanced Study, City University of Hong Kong City University of Hong Kong Shenzhen Research Institute Department of Materials Science and Engineering, City University of Hong Kong City University of Hong Kong Shenzhen Futian Research Institute

#### **FORUM CO-CHAIRS**

Prof. Dr. Rafal E. Dunin-Borkowski, Research Centre Juelich Prof. Dr. Wolfgang Jaeger, Kiel University Prof. Dr. Fu-Rong Chen, City University of Hong Kong Prof. Dr. Xiaoyan Zhong, City University of Hong Kong

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# Programme Rundown HKIAS Forum and the 5<sup>th</sup> Sino-German Symposium on Advanced Electron Microscopy and Instrumentation

11 - 12 December, 2023 | City University of Hong Kong, Hong Kong SAR, P. R. China

13 - 14 December, 2023 | City University of Hong Kong Shenzhen Futian Research Institute, Shenzhen, P. R. China

| Day 1: 11 Dec 2023 (Monday)<br>Senate Room, 19/F, LAU Building, City University of Hong Kong, Hong Kong SAR, China |   |  |
|--|---|--|
| 09:00-09:10  | Opening Remarks   |  |
| Session 1: Elec  | ctron Microscopy in Materials Physics   |  |
| Chair: Prof. Q   | Diang Xu (Guangzhou Laboratory)   |  |
|  | Prof. Knut Urban (Forschungszentrum Jülich)                                     |  |
| 09:10-10:00  | Topic: Precision measurements of atomic positions and displacements in          |  |
|  | aberration-corrected conventional transmission electron microscopy (CTEM)       |  |
| 10:00-10:30  | Tea Break (Group photo)   |  |
|  | Prof. Wolfgang Jäger (Kiel University)  |  |
| 10:30-11:00  | <b>Topic:</b> Advanced Transmission Electron Microscopy for the Development of  |  |
|  | High-Efficiency Solar Cells   |  |
| 11.00-11.30  | Prof. Ye Zhu (The Hong Kong Polytechnic University)                             |  |
| 11.00 11.00  | <b>Topic:</b> Resolving exotic structure and polar ordering using advanced STEM |  |
|  | Prof. Philipp Pelz (Friedrich Alexander - Universität Erlangen- Nürnberg)       |  |
| 11:30-12:00  | <b>Topic:</b> Large-Scale 3D Atomic Resolution Phase-Contrast Imaging from 4D-  |  |
|  | STEM Measurements   |  |
| 12:00-12:30  | Free Discussion   |  |
|  | Lunch Break (by invitation only)  |  |
| 12:30-14:00  | Faculty Lounge, 9/F, BOC Building, City University of Hong Kong, Hong           |  |
|  | Kong SAR, China   |  |

| Day 1: 11 Dec   | 2023 (Monday)  |  |
|---|--|--|
| Senate Room, 19/F, LAU Building, City University of Hong Kong, Hong Kong SAR, China |  |  |
| Session 2: Advanced Instrumentation   |  |  |
| Chair: Prof. L  | iang Jin (Bioland Lab)   |  |
| 14:00-14:50   | Prof. Maximilian Haider (CEOS Company)                                     |  |
|   | Topic: Prospects of future instrumental developments for advanced electron |  |
| 14:50-15:20   | Dr. Petra Specht (University of California, Berkeley, USA)                 |  |
|   | Topic: Applied low current microscopy                                      |  |
| 15:20-15:50   | Tea Break  |  |
| 15:50-16:20   | Prof. Wenxin Tang (Chongqing University)                                   |  |

|             | Topic: Tailoring two dimensional materials in LEEM                            |
|-------------|---|
| 16 20 16 50 | Prof. Zhu-Jun Wang (ShanghaiTech University)                                  |
| 16:20-16:50 | electron microscope   |
| 16.50 17.05 | Dr Maarten Wirix (Thermo Fisher Scientific Co.)                               |
| 10:50-17:05 | Topic: Spectra for sample integrity, custom automation and field free imaging |
|             | Dr Takeo Sasaki (JEOL Co.)  |
| 17:05-17:20 | Topic: New features of 300 kV aberration-corrected TEM/STEM and               |
|             | applications for materials science  |
| 17:20-17:50 | Free Discussion   |
| 17:50-20:00 | Dinner  |

| Day 2: 12 Dec 2023 (Tuesday)                 |  |  |
|--|--|--|
| Senate Room, 19                              | /F, LAU Building, City University of Hong Kong, Hong Kong SAR, China           |  |
| Session 3: Electron Energy Loss Spectroscopy |  |  |
| Chair: Prof L                                | in Gan (Tsinghua University, Shenzhen International Graduate School)           |  |
| 00.00 00.30                                  | Prof. Wu Zhou (University of Chinese Academy of Sciences)                      |  |
| 09:00-09:50                                  | Topic: Single-atom microscopy and spectroscopy for 2D materials                |  |
|  | Prof. Xiaoyan Zhong (City University of Hong Kong)                             |  |
| 09:30-10:00                                  | Topic: Progress, prospects and challenges for achromatic imaging of electron   |  |
|  | energy loss spectroscopy   |  |
| 10:00-10:30                                  | Tea Break  |  |
|  | Dr Xingxu Yan (University of California, Irvine)                               |  |
| 10:30-11:00                                  | Topic: Probing Nanoscale and Atomic-Level Exotic Vibrational Modes and         |  |
|  | Phonon Dynamics using Monochromated Electron Energy-Loss Spectroscopy          |  |
| 11.00 11.20                                  | Dr. Benedikt Haas (Humboldt University Berlin)                                 |  |
| 11:00-11:50                                  | <b>Topic:</b> Extending the Capabilities of Energy- and Momentum-Resolved STEM |  |
| 11:30-12:00                                  | Free Discussion  |  |
| 12:00-14:00                                  | Lunch Break (by invitation only)   |  |
|  | City Chinese Restaurant, 8/F, BOC Building, City University of Hong Kong,      |  |
|  | Hong Kong SAR, China   |  |

#### Day 2: 12 Dec 2023 (Tuesday)

Senate Room, 19/F, LAU Building, City University of Hong Kong, Hong Kong SAR, China **Session 4: Electron Microscopy in Materials Physics** Chair: Dr. Qi Wang (City University of Hong Kong) Prof. Rafal E. Dunin-Borkowski (Forschungszentrum Jülich) Topic: Progress, prospects and challenges for in situ transmission electron 14:00-14:30 microscopy of electrical and magnetic switching processes in functional materials and nanoscale devices Prof. Songhua Cai (The Hong Kong Polytechnic University) Topic: Ex situ and in situ Scanning Transmission Electron Microscopy Studies 14:30-15:00 of Functional Perovskite Materials and Devices **Prof. Jiong Zhao** (The Hong Kong Polytechnic University) 15:00-15:30 **Topic:** In situ transmission electron microscopy on two-dimensional ferroic chalcogenides

| 15:30-16:00 | Tea Break   |
|-------------|---|
| 16:00-16:30 | <b>Dr. See Wee Chee</b> (Fritz-Haber-Institute of the Max-Planck Society)<br><b>Topic:</b> Understanding Electrocatalyst Re-structuring during Reaction with<br>Electrochemical Cell Transmission Electron Microscopy |
| 16:30-17:00 | <ul><li>Prof. Manling Sui (Beijing University of Technology)</li><li>Topic: In situ electron microscopy for electron radiolysis effect and dose dependence of functional metal oxides</li></ul>                       |
| 17:00-17:30 | <ul><li>Prof. Shih-Wei Hung (City University of Hong Kong)</li><li>Topic: Accurate Retrieval of Three-Dimensional Atomic Dynamics of Moiré Materials</li></ul>  |
| 17:30-18:00 | Free Discussion   |
| 18:00-21:00 | Dinner  |

| Day 3: 13 Dec 2023 (Wednesday) |   |  |
|--------------------------------|---|--|
| Building A, Sher               | nzhen-Hong Kong International Science and Technology Park, Futian District, Shenzhen, |  |
| 1/100-1/10                     | Opening remarks   |  |
| 14.00-14.10<br>Session 5: Ime  | opening remarks   |  |
| Chair: Drof I                  | uphan Lin (Southern University of Science and Technology)                             |  |
|                                | <b>Draf Angua Vinkland</b> (University of Oxford)                                     |  |
| 14:10-14:40                    | <b>Froi.</b> Angus Kirkiand (University of Oxford)                                    |  |
|                                | <b>Topic:</b> Making every electron count – Ptychography at low dose                  |  |
| 14:40-15:10                    | Prof. Fu-Rong Chen (City University of Hong Kong)                                     |  |
|                                | <b>Topic:</b> Atomic Resolution 3D Dynamics of Helix Materials: Present and Future    |  |
|                                | Prof. Xiaodong Han (Southern University of Science and Technology)                    |  |
| 15:10-15:40                    | Topic: Developing Atomic Resolved Mechanical Testing System and                       |  |
|                                | Measuring Grain/Twin Boundary Plasticity at Atomic Level                              |  |
| 15:40-16:10                    | Tea Break   |  |
|                                | Prof. Ute Kaiser (Ulm University)   |  |
| 16.10.16.40                    | <b>Topic:</b> From functionalizing inorganic two-dimensional materials on the level   |  |
| 10:10-10:40                    | of single atoms towards molecular imaging of organic two-dimensional                  |  |
|                                | materials   |  |
|                                | <b>Prof. Peivi Wang</b> (Southern University of Science and Technology)               |  |
| 16:40-17:10                    | <b>Topic:</b> The Principle of Aberration Corrected 300 kV Cryo-EM and Its            |  |
|                                | Application to Thick Specimens in Biology   |  |
|                                | <b>Prof Tao Ni</b> (The University of Hong Kong)                                      |  |
| 17.10-17.40                    | <b>Tonic:</b> Cryo-electron tomography and subtomogram averaging for high-            |  |
| 1/.10-1/.40                    | resolution structure determination of macromolecules                                  |  |
| 17.40-18.10                    | Free Discussion   |  |
| 19.10 21.00                    | Dispar  |  |
| 10:10-21:00                    | Dimer   |  |

#### Day 4: 14 Dec 2023 (Thursday)

Building A, Shenzhen-Hong Kong International Science and Technology Park, Futian District, Shenzhen, China

# Session 6: Ultrafast Imaging

Chair: Prof. Wanpeng Li (Beijing Institute of Nanoenergy and Nanosystems, Chinese Academy of Sciences)

| 09:00-09:30 | <b>Dr. Xihang Shi</b> (Israel Institute of Technology)<br><b>Topic:</b> Tunable X-ray Radiation from Quantum Free-electron Radiation  |  |  |
|-------------|---|--|--|
| 09:30-10:00 | <ul> <li>Dr. Murat Sivis (Max Planck Institute for Multidisciplinary Sciences and University of Gottingen)</li> <li>Topic: Mapping and controlling of optical near fields in an ultrafast transmission electron microscope</li> </ul> |  |  |
| 10:00-10:30 | <b>Dr. Roy Shiloh</b> (Hebrew University)<br><b>Topic:</b> Nanophotonic electron accelerators towards electron microscopy   |  |  |
| 10:30-11:00 | Tea Break   |  |  |
| 11:00-11:30 | <b>Prof. Xuewen Fu</b> (Nankai University)<br><b>Topic:</b> 4D electron microscopy and its applications in non-equilibrium dynamics   |  |  |
| 11:30-12:00 | <ul><li>Prof. Sascha Schäfer (Oldenburg University)</li><li>Topic: Instrumental developments in ultrafast transmission electron microscopy</li></ul>  |  |  |
| 12:00-12:30 | Free Discussion   |  |  |
| 12:30-14:00 | Lunch Break   |  |  |

Day 4: 14 Dec 2023 (Thursday) Building A, Shenzhen-Hong Kong International Science and Technology ParkFutian District, Shenzhen, China Session 7: Quantum Electron Microscopy Chair: Prof. Lin Xie (Great Bay University)

|             | An Are (Great Day Oniversity)  |
|-------------|--|
| 14.00 14.20 | Prof. Pieter Kruit (Delft University of Technology)                      |
| 14:00-14:50 | Topic: Contrast formation in Quantum Electron Microscopy                 |
| 14.20 15.00 | Prof. Hiroshi Okamoto (Akita Prefectural University, Japan)              |
| 14:30-15:00 | Topic: Entanglement-Enhanced Electron Microscopy and Its Generalizations |
| 15.00 15.20 | Prof. Yu-Chun Hsueh (City University of Hong Kong)                       |
| 15:00-15:50 | Topic: Quantum Resonator in A Time-resolved Electron Microscope          |
| 15:30-16:00 | Tea Break  |
|             | Prof. Vincenzo Grillo (National Research Council (Italy))                |
| 16:00-16:30 | Topic: A few "quantum" and spooky ideas and experiments in electron      |
|             | microscopy   |
|             | Dr. Christian Kisielowski (Lawrence Berkeley National Laboratory, USA)   |
| 16.30-17.00 | Topic: Probing Dynamic Responses of Nano-Materials at the Boundary       |
| 10.30-17.00 | between Classical and Quantum Mechanics detecting Coherent-Inelastic     |
|             | Electron Self-interferences  |
| 17:00-17:30 | Free Discussion  |
| 17:30-17:40 | Closing remarks  |
| 17:40-21:00 | Dinner   |

# Abstract, Biography and Photo

# Precision measurements of atomic positions and displacements in aberration-corrected conventional transmission electron microscopy (CTEM)

#### Knut W. Urban<sup>1</sup>, Lei Jin<sup>1</sup>, Chun-Lin Jia<sup>2</sup>

# <sup>1)</sup>Ernst Ruska Center (ER-C) for Microscopy and Spectroscopy with Electrons, <u>Research Center Juelich, 52425 Juelich, Germany</u>

<sup>2)</sup> School of Electronic and Information Engineering and State Key Laboratory for Mechanical Behavior of Materials, Xi'an Jiaotong University, Xi'an 710049, China

With the introduction of aberration-corrected electron optics, transmission electron microscopy has achieved single-atom resolution. Moreover, atomic structures can now be measured, atom by atom, with picometer-level precision. This is of outstanding importance, since atomic displacements in these dimensions are responsible for corresponding local changes in physical properties. This access to locally varying properties, e.g. at the unit cell level or at an internal interface or at a surface make transmission electron microscopy a unique tool in the study of materials.

The objective of this paper is to provide an overview of ultra-high precision measurements in the aberration-corrected conventional transmission electron microscope (CTEM) [1]. The starting point of such measurements is the fact that the high precision can only be achieved indirectly, by proper refinement of the experimental data. A central element is the calculation of the image by iterative solution of the Schrödinger equation, where the unknown sample parameters as well as the only imprecisely known optical parameters enter as variables. The model, both of the specimen and of the imaging process itself, obtained on the basis of an optimal fit (at absolute value of the image contrast) to the intensity distribution in the experimental image, represents the actual result of the investigation.

The value of atomic resolution electron microscopy as a quantitative measuring technique for physical investigations depends crucially on the extent to which it is possible to quantify the measurement errors [2,3,4,5]. The key measure here is the precision, expressed by the standard deviation, which refers to the closeness of agreement between the individual measurement results.

[1] K.W. Urban et al., Progress in atomic-resolution aberration corrected conventional transmission electron microscopy (CTEM). Prog. Mat. Sci. 133, 101037 (2023).

[2] S. Van Aert et al., Maximum likelihood estimation of structure parameters from high resolution electron microscopy images. Part II: A practical example. Ultramicroscopy 104, 107 (2005).

[3] L. Houben, A. Thust, K. Urban, Atomic-precision determination of the reconstruction of a 90° tilt boundary in YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> by aberration corrected HRTEM. Ultramicroscopy 106, 200 (2006).

[4] C.-L. Jia, et al., Atomic-scale evidence for displacive disorder in bismuth zinc niobate pyrochlore. Ultramicroscopy 192, 57 (2018).

[5] Z.-H. Ge et al., Atomic-scale observation of off-centering rattlers in filled skutterudites. Adv. Energy Mater. 12, 2103770 (2022).

| Full name   | Knut Wolf Urban  |
|-------------|--|
| Affiliation | Professor, Ernst Ruska-Center for Microscopy and<br>Spectroscopy with Electrons (ER-C), Helmholtz<br>Research Center Jülich, Germany   |
| Distinction | <ul> <li>1986 Acta Metallurgica Award</li> <li>1996 Research Award of the Japanese Soc. for Promotion of Science</li> <li>1999 Heyn Medal of German Society for Materials Science</li> <li>2000 Honorary Member Materials Research Society of India</li> <li>2000 Medal for Scientific Publishing of the German Physical Society</li> <li>2006 Von-Hippel Award of the US Materials Research</li> <li>Society (MRS)</li> <li>2006 Honorary Member of US Materials Research Society (MRS)</li> </ul>  |
|             | 2006 Beckurts-Award for Scientific and Technical Innovation<br>2008 HONDA Prize for Ecotechnology (Honda Foundation, Japan)<br>2009 Appointment Member of the Academy of Arts and Sciences of<br>the State of North Rhine-Westphalia, Germany<br>2009 Honorary Professor at Xi'an Jiaotong University, Xi'an, China<br><b>*2011 Awarded the Wolf Prize in Physics</b>  |
|             | <ul> <li>2012 Honorary Member German Electron Microscopy Society</li> <li>2014 Frontiers of Knowledge Award in Basic Sciences (BBVA<br/>Foundation, Madrid, Spain)</li> <li>2015 Honorary Member German Physical Society</li> <li>2015 Honorary Member Japanese Institute of Metals and Materials</li> <li>2015 NIMS Award (National Institute of Materials Science, Tsukuba,<br/>Japan)</li> <li>2018 Doctor honoris causa, Tel Aviv University, Israel</li> <li>*2020 Awarded the Kavli Prize in Nanoscience</li> <li>2020 Appointment Foreign Member of the Norwegian Academy of<br/>Science and Letters</li> </ul> |
| Biography   | Knut Urban is a German physicist. He studied at the University of<br>Stuttgart where he obtained his doctor degree in physics in 1972, before<br>moving to the Max Planck Institute of Metals Research in Stuttgart.   |
|             | In 1986 he was appointed a professor in materials sciences at<br>Erlangen–Nuremberg University, and just one year later became Chair<br>of Experimental Physics at RWTH Aachen University and the Director<br>of the Institute of Microstructure Research at Forschungszentrum<br>Juelich. The institute's research focuses on phases and phase<br>transformations as well as lattice defects in metals and alloys, HTc<br>superconductivity and superconducting devices, and atomic structures<br>in complex oxides. From 1990 he collaborated with Harald Rose   |

and Maximilian Haider to construct the first aberration-corrected transmission electron microscope. With this Urban realized atomic resolution in electron microscopy in materials for the first time (published in 1998).

Urban then worked on the application of aberration-corrected atomically resolving transmission electron microscopy to materials science. In particular he focused on the connection between the precise arrangement of atoms within a lattice and the physical properties of a material. In 2004 he was chosen as one of the two directors of the Juelich-Aachen Ernst Ruska Center for Microscopy and Spectroscopy with Electrons and since 2012 has been a JARA Senior (Distinguished) Professor at RWTH Aachen University. Urban served as President of the German Physical Society from 2004 to 2006.

Urban has been awarded a number of honors. These include the Von Hippel Award of the US Materials Research Society, and jointly with Rose and Haider, the Wolf Prize in Physics, the Kavli Prize in Nanoscience, the HONDA prize in Ecotechnology and the BBVA Foundation Frontiers of Knowledge Award in Basic Sciences. He is an honorary member of several scientific bodies, including the US Materials Research Society, the German Physical Society and the Japanese Institute of Metals and Materials.

# Advanced Transmission Electron Microscopy for the Development of High-Efficiency Solar Cells

Andras Kovács<sup>1</sup>, Felix Predan<sup>2</sup>, Jens Ohlmann<sup>2</sup>, David Lackner<sup>2</sup>, Frank Dimroth<sup>2</sup>, Rafal E. Dunin-Borkowski<sup>1</sup>, Dietrich Haeussler<sup>formerly 3</sup>, Wolfgang Jaeger<sup>3</sup>

<sup>1</sup>Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons, Forschungszentrum Juelich, Germany

<sup>2</sup>Fraunhofer Institute for Solar Energy Systems ISE, Freiburg, Germany

<sup>3</sup>Institute for Materials Science, Christian-Albrechts-Universitaet zu Kiel, Germany

High-efficiency multi-junction solar cells based on hetero-epitaxial III-V compound semiconductor layers reach currently highest solar-electric conversion efficiencies of more than 46% under concentrated sunlight (1). Such cells are fabricated by metal-organic vapor phase epitaxy (MOVPE) and are primarily used in terrestrial concentrator photovoltaics, in power generation for satellites and spacecraft and for solar generation of hydrogen (e.g., ref. 1). Being important also for their integration with cells based on Ge and Si, the challenges in heteroepitaxial layer growth of different III-V semiconductor materials are the reduction of crystal defects and the control of layer strains, caused by the differences in lattice constant and thermal expansion. Successful approaches used for processing technologies are the direct epitaxial growth on substrates involving buffer layer concepts (2, 3), and the layer transfer combined with semiconductor wafer bonding (4-7). The imaging and spectroscopic methods of advanced and in situ transmission electron microscopy (S)TEM are instrumental in developing concepts for engineering and controlling layer strains and in understanding electrical properties of interfaces and their behaviour during thermal processing, thus contributing relevant analytical information for the development of successful processing technologies in solar cell fabrication. Current developments, aiming at solar cell efficiencies of 50 % and more, explore new combinations of materials systems and employ novel approaches for high-resolution characterization of electrical interface properties.

1. F. Dimroth et al., Four-Junction Wafer-Bonded Concentrator Solar Cells, IEEE Journal of Photovoltaics, 6, 343 (2016).

2. F. Dimroth, T. Roesener, S. Essig, Ch. Weuffen, A. Wekkeli, E. Oliva, G. Siefer, K. Volz, Th. Hannappel, D. Häussler, W. Jäger, A. W. Bett: *Comparison of Direct Growth and Wafer Bonding for the Fabrication of GaInP/GaAs Dual-Junction Solar Cells on Silicon*. IEEE Journal of Photovoltaics 4 (2), 620 - 625 (2014).

3. J. Schöne, E. Spiecker, F. Dimroth, A.W. Bett, W. Jäger, *Defect Formation and Strain Relaxation in graded GaPAs/GaAs, GaNAs/GaAs and GaInNAs/Ge Buffer Systems for high-efficiency Solar Cells,* Journal of Physics: Conference Series, DOI: 10.1088/1742-6596/471/1/012008, 471, 012008 (2013).

4. K. Derendorf, S. Essig, E. Oliva, V. Klinger, T. Roesener, S.P. Philipps, J. Benick, M. Hermle, M. Schachtner, G. Siefer, W. Jäger, F. Dimroth, *Fabrication of GaInP/GaAs//Si Solar Cells by Surface Activated Direct Wafer Bonding*, IEEE Journal of Photovoltaics 3(4), 1423 (2013).

5. D. Häussler, L. Houben, S. Essig, M. Kurttepeli, F. Dimroth, R.E. Dunin-Borkowski, W. Jäger, *Aberration-corrected transmission electron microscopy analyses of GaAs/Si interfaces in wafer-bonded multi-junction solar cells.* Ultramicroscopy, 134, 55 - 61 (2013).

6. F. Predan, A. Kovács, J. Ohlmann, D. Lackner, R. E. Dunin-Borkowski, F. Dimroth, W. Jäger: *Effects* of thermal annealing on structural and electrical properties of surface-activated n- GaSb/n-GaInP direct wafer bonds. Journal of Applied Physics 122, 135307 (2017).

7. F. Predan, D. Lackner, E. Oliva, A. Kovács, W. Jäger, F. Dimroth., "Developments for Wafer-bonded Four-Junction Solar Cells based on GaSb," in 2018 IEEE 7<sup>th</sup> World Conference on Photovoltaic Energy Conversion (WCPEC, Waikoloa Village, HI, USA, 2018, pp. 1–6.

It is a pleasure to acknowledge the contributions of all colleagues mentioned in the references.

| Full name   | Wolfgang Jäger   |  |
|-------------|--|--|
| Affiliation | Prof. Dr.<br>Institute for Materials Science, Christian-<br>Albrechts-Universität zu Kiel<br>Street: Kaiserstr. 2<br>Code, City: 24143 Kiel<br>State: Germany EU   |  |
| Distinction | Member of international award committees         Member of international award committees         Member of numerous search committees for academia and for research institutions         Member of EMS Outstanding Paper Award Committee of the European Microscopy Society 2011-2013         Member of EU-funded MACAN Consortium 2008 - 2013         Advisory board member for institutes and research institutions         Since 2015, Long-term Guest Scientist, Ernst-Ruska Centre, Forschungszentrum Jülich, Germany         2015, Institute of Engineering Innovation, The University of Tokyo (JSPS Fellowship Award)         Since 2013, Guest Professor, Chalmers University of Technology, Dept Physics, Gothenburg, Sweden         Wolfgang Lagger received his Diploma and his Doctor degree (Dr rer   |  |
| Biography   | Wolfgang Jaeger received his Diploma and his Doctor degree (Dr rer<br>nat, 1976) in Physics from the University of Stuttgart based on his<br>research work at the Max-Planck-Institute for Metal Research,<br>Stuttgart, Germany under the guidance of Professor Alfred Seeger. He<br>joined the Materials Science Division at Argonne National Laboratory<br>USA (1977-1979) as a post-doc scientist. From 1979 to 1996 he took<br>positions of a staff scientist and of a group leader of a microstructure<br>research group at the Institute for Solid State Research at the Research<br>Centre Juelich, Germany. In 1996, he was appointed Professor and<br>Head of the Microanalysis of Materials Group at the Christian-<br>Albrechts-Universität zu Kiel / Kiel University, Germany. He<br>contributed to establishing the infrastructure and the study course<br>system in Materials Science and initiated and coordinated several |  |

interdisciplinary activities at the Kiel University.

In 2013, he retired from his university teaching duties and since continues with research and further professional activities, such as coorganizing scientific conferences and workshops on advanced transmission electron microscopy and its applications to current topics in materials and life science. Currently, he enjoys being guest scientist at the Ernst Ruska - Center of the Forschungszentrum Juelich, Germany, and at the Department of Physics of the Chalmers University of Technology, Gothenburg Sweden.

His research interest focuses on transmission electron microscopy and microstructure research in materials science of functional thin films, nanomaterials and materials for energy. He published more than 300 scientific publications, including book chapters and review articles, and has given numerous invited and plenary presentations at international conferences, in advanced scientific schools, and in institutes.

# **Resolving exotic structure and polar ordering using advanced STEM**

<u>Chao Xu</u><sup>1</sup>, Ye Zhu<sup>1</sup>

<sup>1</sup>Department of Applied Physics, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong SAR, China

Novel functional materials are usually characterized by emerging ordering beyond the conventional unit-cell level. Examples include artificial superlattices, self-assembled nanostructures, ferroic domain structures, and charge-density waves. Such complex ordering, even though occurring collectively, commonly suffers nanoscale fluctuations that destroy the longrange periodicity that is required for conventional diffraction-based structure analysis, posing a formidable challenge for accurate structure determination. On the other hand, the maturation of aberration-corrected TEM/STEM presents an alternative real-space approach to probe the local complex ordering, through directly imaging the atomic structure with picometer precision. In this talk, I will give several examples demonstrating the power of advanced STEM on resolving the complex atomic and polar ordering in perovskite oxides and 2D materials: i) By developing an imaging condition optimized for oxygen contrast, we can image sensitively the octahedral structure in perovskite oxides with picometer precision. It further enabled us to reveal an extraordinary 2D ordered octahedral tilting in the solid electrolyte Li<sub>0.5-3x</sub>Nd<sub>0.5+x</sub>TiO<sub>3</sub> (Fig. 1a and 1b), and to demonstrate its dependence on the competition between Li content and lattice strain.[1] ii) Through atomic displacement mapping based on high-resolution imaging, and electric polarization mapping based on 4D-STEM, we made the first experimental discovery of 2D antiferroelectricity in vdW In<sub>2</sub>Se<sub>3</sub> (Fig. 1c), and resolved the true nature of its superstructure that had been under debate for over four decades.[2] We also demonstrated the 2D ferroelasticity coupled with this antiferroelectricity through the spontaneous lattice strain.[3] iii) Lastly, iDPC technique in STEM allowed us to unravel the exotic polar textures associated to the modulated octahedral tilting in complex perovskites. The characterization strategy and capability in our work demonstrate a powerful tool to probe the structure-property interplay in emerging functional materials at the atomic scale.

[1] Zhu, Y.\*, Withers, R. L., Bourgeois, L., Dwyer, C. & Etheridge, J.\* (2015). *Nat. Mater.* 14, 1142.

[2] Xu, C., Chen, Y., Cai, X., Meingast, A., Guo, X., Wang, F., Lin, Z., Lo, T. W., Maunders, C., Lazar, S., Wang, N., Lei, D., Chai, Y., Zhai, T., Luo, X. & Zhu, Y.\* (2020). *Phys. Rev. Lett.* **125**, 047601.

[3] Xu, C., Mao, J., Guo, X., Yan, S., Chen, Y., Lo, T. W., Chen, C., Lei, D., Luo, X., Hao, J., Zheng, C. & Zhu, Y.\* (2021). *Nat. Commun.* **12**, 3665.

Y. Z. thanks the financial support from the Research Grants Council of Hong Kong (N\_PolyU531/18, C5029-18E) and the Hong Kong Polytechnic University grant (1-ZVRP).

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## Large-Scale 3D Atomic Resolution Phase-Contrast Imaging from 4D-STEM Measurements

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Spurred by the development and widespread availability of fast pixelated direct electron detectors (DEDs), scanning transmission electron microscopy (STEM) is undergoing a computational imaging renaissance. Experiments envisaged more than ten years ago can finally be realized with modern detector technology and reconstruction algorithms for 4D-STEM datasets.

We show the experimental demonstration of one such experiment relying on fast DEDs: ptychographic electron tomography at atomic resolution. Using ptychographic electron tomography, we have reconstructed an atomic-resolution 3D volume of a double-wall carbon nanotube encapsulating a complex core-shell  $Zr_{11}Te_{50}$  structure from 34 million diffraction patterns. From this volume, the atomic structure was determined using atom tracing methods known from annular dark-field atomic electron tomography. Ptychographic electron tomography relies on the projection approximation, such that samples have to be ~10nm or less in thickness. Multi-slice ptychographic tomography alleviates this constraint by modeling multiple scattering and allowing 3D atomic resolution phase-contrast imaging beyond the depth of focus limit. I will show first results using multi-slice ptychographic electron tomography to reconstruct samples with thickness beyond the depth of focus limit. With further technical developments, this method will be able to solve the 3D atomic structure of general TEM samples like FIB lamellas or needle specimens at atomic resolution in 3D. Finally, the limitations of current algorithms will be discussed, and developments towards imaging much larger volumes with 4D-STEM will be detailed. I will show preliminary reconstructions of a Co<sub>3</sub>O<sub>4</sub> nanocube that break the depth-of-field limit of conventional electron microscopy by a factor of 3.

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| Biography   | Philipp Pelz received Bachelor degrees in Physi<br>Informatics (2012), and Master degrees in Applied<br>Physics, Materials Science & Chemistry (2013). In 20<br>his Ph.D. in Physics from the University of Hambur<br>Planck Institute for the Structure and Dynamics of Ma<br>Subsequently, he spent three years as a postdoctoral re<br>University of California, Berkeley and the National Cen<br>Microscopy. Since August 2022 he is Tenure-Track<br>Computational Materials Microscopy at FAU Erlangen | cs (2011) and<br>& Engineering<br>018 he obtained<br>rg & The Max<br>atter, Germany.<br>esearcher at the<br>tter for Electron<br>& Professor for<br>-Nürnberg. |

## Prospects of future instrumental developments for advanced electron

#### microscopy

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The generally advancement of the electron microscope requires a continuous development of all components which are mandatory for high performing TEM and STEM applications. For high resolution imaging and analytical purposes with high energy resolution some components are crucial, however, the overall stability and sensitivity against external fields are equally important because finally, the weakest part defines the attainable performance.

A detailed investigation of future prospects of instrumental developments has to cover all components from the source of the electrons to the recording detectors in order to get the most out of the interaction process between the electrons and the object. At the electron source not only the electron emitter and its shape has to be optimized but also the electron optical components of the gun which affect the size of the virtual source. The smaller the virtual source size the higher the brightness one can achieve.

A limitation within the electron optical column which was not known before 2013 [1] is the so-called Johnson noise which causes an image spread and, therefore, a fundamental limitation of the attainable spatial resolution. This Johnson noise depends mainly on the distance of the electrons to metal surfaces of pole pieces or vacuum tubes. This distance cannot be lowered arbitrarily because of the required strength of the multipole fields for the compensation of aberrations and, at the same time, the field strengths are limited by saturation of magnetic pole pieces or the breakthrough voltage of electrodes when applying high voltages. This limitation set by the free electrons in metals was first noticed when the chromatic aberration C<sub>c</sub> of a 300 kV TEM had to be compensated and the theoretically achievable resolution could not be attained. The required beam diameter within the counteracting electrostatic and magnetic quadrupole fields caused a loss of resolution due to an increased image spread. The only way to reduce the resolution limiting effect of the Johnson noise is to reduce the temperature of all metallic surfaces within the objective lens and the multipole elements of a corrector. This would be possible with super-conducting lenses and multipole elements. However, the effort would be extremely high and also the operation costs later due to the needed liquid Nitrogen or Helium coolant would make the efforts for such a system questionable.

An additional component which needs a certain attention too is a monochromator to reduce the energy spread of the illuminating electrons [2] which would be advantageous for high spatial resolution and, even more important, for high resolution electron energy loss spectroscopy.

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Acknowledgements:

We are grateful of having received a Synergy Grant *with substantial* funding from the European Union's Horizon2020 research and innovation Program under Grant No 951215 (MoRe-TEM).

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|             | resolution TEM finished   |
|             | 2005: the Innovation Award of the State of Baden Württemberg  |
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|             | 2006: the Karl Heinz Beckurts Preis together with Prof. Rose and  |
|             | Prof. Urban   |
|             | 2008: an Honorary Professorship at the University of Karlsruhe  |
|             | 2008: the Honda Prize together with Prof. Rose and Prof. Urban for  |
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|             | microscopy for high-resolution visualization of atomic structure  |
|             | 2011: Wolf Prize in Physics together with Prof. Harald Rose and   |
|             | Prof. Knut Urban.   |
|             | 2013: BBV Foundation Frontiers of Knowledge Award in Physics  |
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|             | 2015: Honorary Fellowship of the Royal Microscopical Society.   |
|             | 2015: NIMS Award 2015.  |
|             | 2019: Member of the Class 2019 Fellows of the Microscopy Society  |
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| Biography   | Maximilian Haider studied physics in Kiel and Darmstadt and   |
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|             | (EMBL). In 1989 he became a Group Leader within the Physical  |
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|             | set up a Cs-corrector for a 200 kV TEM together with Prof. Rose in  |
|             | Darmstadt and Prof. Urban in Juelich. In 1996 he founded CEOS   |
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# **Applied Low Current Microscopy**

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Since the DoE funded TEAM project ended in the United States with the development of spherical and chromatic aberration correction in 2009 single atom sensitivity is established for transmission electron microscopy (TEM) imaging. Theoretically, every material could be imaged in full atomic resolution, and every atom could be resolved within all material structures. In an experimental setup, however, electron beam sensitivity of a given material determines which original structures can be resolved. Therefore, our focus in the last decade has been on minimizing electron beam induced material damage, and to find alternative ways to image beam sensitive materials without altering their original structure. At least, if a material is altered under the electron beam the dynamics of this behavior should be recorded, too. Such dynamic considerations led to the development of current-controlled TEM.

Low dose rate and pulsed electron beams were applied to image pristine properties of MgCl<sub>2</sub>, a Ziegler-Natta catalyst which is both air and electron beam sensitive. The choice of pulse and delay times allowed to mitigate damage caused by phonon accumulation. The total electron dose can be extended more than ten times before material alterations set in. Changing electron currents while imaging polymers or various catalytic nanocrystals by varying dose rates and / or the irradiated areas had similar effects. Using direct electron detectors facilitated those experiments, and allowed for the use of ultra-low currents where single electrons are delivered to interact with the material under investigation. The importance of reproducible imaging and sample preparation conditions will be discussed.

With the application of current-controlled TEM the onset of material changes is found to be generally delayed. The experimental setup may be used in combination with cryo-microscopy, and could further postpone material alterations, even in soft materials.

#### Acknowledgment:

Electron microscopy at the Molecular Foundry is supported by the Office of Science, the Office of Basic Energy Sciences, the U.S. Department of Energy, Contract No. DE-AC02-05CH11231.

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| Biography   | Dr. Petra Specht is an Associate Scientist at the MSE Department of UC Berkeley. In 1996 she was awarded her PhD in Natural Sciences (Dr.rer.nat.) from the Technical University in Aachen, Germany while working at the Max-Planck Institute for Iron Research in Duesseldorf, on mechanical properties of single crystalline NiAl and FeAl intermetallics. In April 1996 she joined the Eicke Weber group, Mat.Sci.&Eng. Department at UC Berkeley. There, she worked on MBE-grown III-V semiconductor production, material characterization and prototype electronic device development. She invented a radiation-hard buffer layer for satellite electronics, and later specialized in radiation effects of high bandgap semiconductors. Since 2008 her research has been continuing in the Oscar Dubon group, with a focus on TEM characterization of semiconductor and catalytic materials. She collaborates with Dr. Christian Kisielowski since 2010. Dr. Specht has published over 100 peer-reviewed articles, her h-index is 25. |

#### Tailoring two dimensional materials in LEEM

#### Wen-Xin Tang

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By using in situ low energy electron microscope (LEEM) technique, we show that tailored twin grains, which are defined in a face-centered cubic structure with stacking sequence of (111) planes reversed in the two sides of (111) mirror plane, are expected to have epitaxial lattice for single crystal graphene growth because of the  $C_{6V}$  symmetry of the graphene lattice. Using first principle calculation, we reveal the orientation mechanism of a graphene domains on arbitrary Cu surface and build the quantitative lattice match relation of all the twinned copper crystals. We further experimentally realized the predicted twinned copper by introducing microhardness indent and external induced driving force, which is proved to be ideal substrate for single crystalline graphene epitaxy. [1]

We also use LEEM to explore the dynamic intercalation of lithium into bilayer graphene on SiC. Stacking engineering in van der Waals (vdW) materials is a powerful method to control topological electronic phases for quantum device applications. Atomic intercalation into the vdW material can modulate the stacking structure at the atomic scale without a highly technical protocol. Here we report that lithium intercalation in a topologically structured graphene/buffer system on SiC(0001) drives dynamic topological domain wall (TDW) motions associated with stacking order change by using an in situ aberration-corrected low-energy electron microscope in combination with theoretical modelling. We observe sequential and selective lithium intercalation that starts at topological crossing points (AA stacking) and then selectively extends to AB stacking domains. Lithium intercalation locally changes the domain stacking order to AA and in turn alters the neighbouring TDW stacking orders, and continuous intercalation drives the evolution of the whole topological structure network. Our work reveals moving TDWs protected by the topology of stacking and lays the foundation for controlling the stacking structure via atomic intercalation. These findings open up new avenues to realize intercalation-driven vdW electronic devices.[2]

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- [2] Nature Nanotechnology 2023 Oct 18(10):1154-1161 (2023)

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| Affiliation | Professor Dr. Chongqing University, Chongqing,<br>China   |   |
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# **Observing while it happens: CVD growth of graphene inside a scanning**

#### electron microscope

#### Zhu-Jun Wang<sup>1</sup>

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In my presentation, I will elucidate the capabilities of environmental scanning electron microscopy (SEM) and champion its use as an invaluable in-situ surface science instrument. Following a showcase of illustrative examples centered on gas-phase reactions on metallic substrates, I will direct attention to the chemical vapor deposition (CVD) synthesis of graphene. Through this lens, I aim to emphasize how direct observational techniques have afforded critical understanding into the growth dynamics and etching characteristics of both mono-layered and multi-layered graphene.

Drawing from direct observational data, we have recently postulated a plausible mechanism for the assisted self-assembly of twisted layer graphene, as illustrated in Fig.1. This procedure, seamlessly integrable into conventional chemical vapor deposition (CVD) growth, can be aptly analogized to the artful practices of Origami and Kirigami with paper. The process encompasses the deliberate induction of wrinkle formation in monolayer graphene, followed by the intricate folding, tearing, and subsequent adlayer growth of these wrinkles. A salient feature of this method is the generation of intertwined graphene spirals and the transformation of the chiral angle inherent to one-dimensional (1D) wrinkles into a two-dimensional (2D) twist angle, interposing layers within a three-dimensional (3D) superlattice. Employing techniques such as seeded growth and substrate engineering allows for the precision-driven formation of layered stacks with predefined twist angles. The foundational principle is not only robust but also universally applicable, permitting its extension to other foldable 2D materials. Such an approach holds the promise of enabling the fabrication of miniaturized electronic components, encompassing capacitors, resistors, inductors, and superconductors.



Fig.1. Spirals of twisted layer graphene with pre-defined twist-angle. Comparison between simulation and in-situ observation during CVD growth.

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|-------------|---|
| Affiliation | Professor, ShanghaiTech University, China   |
| Biography   | Zhu-Jun Wang is an expert on both electron microscopy and<br>heterogeneous catalysis. He has successfully constructed the surface<br>sensitive near-ambient-pressure in-situ scanning electron microscopy<br>(SEM) during his doctoral research. With this technique, the detailed<br>atomic-scale information obtained by in-situ transmission electron<br>microscopy (TEM)/ scanning tunneling microscopy (STM) can be<br>embedded within the global picture obtained at lower magnifications<br>by in-situ SEM and subsequently correlated with the spectroscopic data<br>from near ambient pressure in-situ X-ray photoelectron spectroscopy<br>(NAP-XPS). This multi-scale approach enables to investigate the<br>dynamic nature of catalyst during the ongoing work, bridges the<br>pressure-gap, and links atomistic details to collective processes. One<br>typical work is the analysis of surface dynamics, from the micrometer-<br>to the atomic-scale, at well-controlled experimental environments to<br>obtain a fundamental understanding of the mechanism of graphene and<br>two dimensional (2D) materials growth on metal catalyst. |

# Spectra for sample integrity, custom automation and field free imaging

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Modern high end transmission electron microscopes (TEMs) are great instruments for atomic observation. Resolutions below 50pm can be achieved and with advanced techniques like ptychography even the atomic-resolution limit set by lattice vibrations can be reached.<sup>1</sup>

A lot of new materials investigations focusing on the fields of energy storage, catalysis, polymers, and quantum materials require the characterization of specimens which degrade fast under the electron beam. The result is low quality micrographs or in the worst-case misinterpretation of beam induced effects. It is therefore necessary for an Analytical TEM to be able to analyze a wide range of materials as dose efficient as possible.

Spectra is a high-end TEM which has many advantages that can help users to observe beam sensitive materials. The Velox acquisition software has new capabilities to predict the electron dose and bring the specimen into the correct orientation with minimal dose required. The single electron sensitive panther stem detector is segmented and is well integrated in Velox. This combination makes it possible to do imaging in STEM at extremely low electron doses with integrated differential phase contrast imaging.<sup>2,3</sup> For optimizing the electron specimen interactions the Spectra Ultra can switch high tensions in a matter of minutes, completely transforming the way that the TEM is done. Lastly the super high solid angle Ultra-X EDX detector helps to maximize the collection of X-rays. This makes that new materials can be observed with STEM-EDX and EDX tomography becomes significantly faster and more dose efficient.

In conclusion, Spectra is a great TEM to observe materials while preserving the sample integrity.

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3. Lazić, I. *et al.* Single-particle cryo-EM structures from iDPC–STEM at near-atomic resolution. *Nat Methods* **19**, 1126–1136 (2022).

#### New features of 300 kV aberration-corrected TEM/STEM and

#### applications for materials science

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Aberration-corrected TEM and STEM have been widely used to

investigate atomic structures of materials. Their performances have already reached to sub-ångström level, enabling precise understanding of static atomic structures. From the hardware point of view, next target would be real-time imaging capability in in-situ observations such as heating/cooling, biasing, stressing, and gas/liquid-cell experiments, because in-situ TEM/STEM characterization could unveil correlation between materials properties and structural change in atomic level. A high frame rate TEM/STEM imaging is of great importance to chronologically capture structural change.

Frame rate of TEM micrograph depends on each camera and is typically over few tens frames per second. In STEM imaging, however, frame rate is few frames per second, depending on the scan pixel sizes. Only making both scan dwell time and fly-back time shorter is not sufficient to achieve a faster scan, because acquired STEM images would be distorted due to a voltage ringing caused by slow response of the original scan coil [1]. Bottlenecks are not only due to slow response of scan coil, but also long afterglow of detector scintillator and detector electronics.

We developed a high-speed scan system on JEM-ARM300F2 by improving those bottlenecks in order to make real-time atomic motion imaging possible [2]. Figure 1

shows an ADF-STEM image of SrTiO<sub>3</sub>[100] taken with a high-speed scan system at 300 kV. Dwell time of 83ns/pixel and fly-back time of 20  $\mu$ s (31 fps at 512 x 512 pixels) were used. 100 images were superimposed (accumulated during 32 seconds) to make signal-to-noise ratio better. No ringing distortion observed in the lattice fringes, meaning that the inductance of the scan coil was successfully optimized. FT of the STEM image shows 1Å spot, meaning that a high-speed scan system works successfully and stably. This technology could improve capability of real-time STEM imaging in insitu experiments. In addition to development of highspeed scan system, recent applications using JEM-ARM300F2 will be presented in this talk.

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Figure 1 (a) ADF-STEM image of  $SrTiO_3[100]$  taken with 31 fps, and 512 x 512 pixels, showing no ringing distortion observed. (b) FT of the image.



# Single-atom microscopy and spectroscopy for 2D materials

<u>Wu Zhou</u><sup>1</sup>, Mingquan Xu<sup>1</sup> and Aowen Li<sup>1</sup>

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Aberration-corrected scanning transmission electron microscopy (STEM) operated at low accelerating voltage can now provide real-space imaging and spectroscopy analysis at the atomic scale and even with single atom sensitivity. Reducing the energy of the incident electron has been proven crucial for the study of two-dimensional materials and carbon-based nanomaterials. While imaging at the single atom level is becoming routine operation in many state-of-the-art STEMs, pushing the sensitivity of spectroscopy techniques down to the single atom level would provide new opportunities to probe the material functionalities, but remains relatively challenging. In this regard, 2D materials, especially doped graphene samples, could provide an ideal platform for exploring the new limit of single-atom spectroscopy techniques.

I will discuss our recent results on pushing the sensitivity of single-atom spectroscopy techniques using dopants in monolayer graphene as a model system. With this relatively stable single-atom model system, we can explore the ultimate sensitivity of EELS at 60 kV where the dose level is no longer the limiting factor. We show that the sensitivity of single-atom vibrational spectroscopy analysis can be pushed to the chemical-bonding level and this technique could be applied to explore local vibrational signatures at defects and interfaces in 2D materials [1]. If by the time of this symposium we have updated results, I should also discuss briefly the progress of atom-by-atom isotope mapping using vibrational spectroscopy technique. As for core-loss excitations, we show that electronic states contributed by specific unoccupied pz orbital around a four-fold coordinated Si point defect in graphene can be mapped out using atomic-resolution energy-loss near-edge fine structure (ELNES) spectroscopy [2].

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Acknowledgement:

We are grateful to all the collaborators who contributed to the work discussed here. This work was supported in part by the Beijing Outstanding Young Scientist Program (BJJWZYJH01201914430039) and the National Key R&D Program of China (2018YFA0305800).

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| Biography   | Wu Zhou is a Professor in School of Physical Scien<br>electron microscopy laboratory at the University of C<br>of Sciences (UCAS) in Beijing, China. He received h<br>2006 from Tsinghua University and his Ph.D. in 20<br>University. His research is mainly focused on the<br>cutting-edge electron microscopy techniques with 1<br>energy resolution, and the application of electron micro<br>the origin of functionalities in 2D materials, heterogen<br>quantum materials. | tces and leads the<br>Chinese Academy<br>is B.S. degree in<br>010 from Lehigh<br>development of<br>high spatial and<br>roscopy to unveil<br>tous catalysts and |

# Progress, prospects and challenges for achromatic imaging of electron energy loss spectroscopy

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Energy-filtered transmission electron microscopy (EFTEM) is an effective method to acquire chemical information from samples with high spatial resolution in the transmission electron microscope (TEM) with parallel beam illumination. However, the main challenges of EFTEM are low dose efficiency, poor energy resolution (which is related to the slit width of the energy window), poor signal-to-noise ratio, and limited spatial resolution caused by chromatic/spherical aberration. Spatially resolved energy loss electron spectroscopy (SR-EELS) [1] is the method that collects the spatial information and spectrum simultaneously at an energy-dispersive plane under parallel beam illumination. However, the spatial resolution of spectrum imaging is limited by spherical (Cs) and chromatic (Cc) aberrations. Thanks to the development of the Cs/Cc corrector, the spatial resolution of inelastic scattering imaging is promoted to the atomic scale and the atomicplane-resolved EELS (APR-EELS) can be achieved [2]. In this work, we achieved the achromatic APR-EELS imaging of SrTiO<sub>3</sub>, CaTiO<sub>3</sub>, and SrTiO<sub>3</sub>-CaTiO<sub>3</sub> superlattice with different experimental conditions, such as crystal orientation, focus values. Combined with dynamic diffraction calculation and experiment data analysis, we explored the optimized experimental conditions to access atomic-plane-resolved chemical information in APR-EELS. We also show the application of APR-EELS on collecting electron magnetic circular dichroism with atomic plane resolution to understand the magnetic, chemical, and structural information in the examples of complex oxides Sr<sub>2</sub>FeMoO<sub>6</sub> [2]. We would like to further discuss the progress, prospects, and challenges of APR-EELS.

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#### Acknowledgments

Thanks to B. Lin, Q. Wang, J. Rusz, L. Jin, A. Tavabi, G. H. Huang, H. B. Jiang, and R. E. Dunin-Borkowski. This work was financially supported by National Natural Science Foundation of China (52171014), the Sino-German Mobility Programme by the Sino-German Center for Research Promotion (M-0265), Science, Technology and Innovation Commission of Shenzhen Municipality (SGDX20210823104200001, JCYJ20210324134402007, HZQB-KCZYB-2020031), Innovation and Technology Fund (ITS/365/21), the Research Grants Council of Hong Kong Special Administrative Region, China (Project No. E-CityU101/20, CityU 11302121, CityU 11309822, G-CityU102/20), the European Research Council (Grant No. 856538, project "3D MAGiC"), CityU Strategic Interdisciplinary Research Grant (7020043), the City University of Hong Kong (Projects no. 9610484, 9680291, 9678288, 9610607), and the City University of Hong Kong Shenzhen Research Institute.

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| Biography   | Dr. Xiaoyan Zhong is currently an associate Professor in the Department of Materials Science and Engineering at City University of Hong Kong. He received his B.S. degree in Materials Science and Engineering in 2001 and PhD degree in Materials Science and Engineering in 2007 at Tsinghua University. After three-year postdoctoral research at Argonne National Laboratory in USA, he began his independent academic career as assistant professor and associate professor at Tsinghua University from 2010 to 2020. He joined Department of Materials Science and Engineering at City University of Hong Kong since May 2020. His current research interests involve methodology development of transmission electron microscopy and spectroscopy and their application in solving new challenges in magnetic materials. Recently Zhong's group has developed the quantitative atomic-plane resolved electron magnetic circular dichroism into atomic level by achromatic electron microscopy, which was published in the peer-reviewed journals such as <i>Nature Materials, Nature Communications</i> and <i>Advanced Functional Materials</i> . He received "Ten Major Scientific and Technological Progress of China's Colleges and Universities" awarded by Ministry of Education of the People's Republic of China and the Excellent Young Scholar awarded by Chinese Electron Microscopy |

# Probing Nanoscale and Atomic-Level Exotic Vibrational Modes and Phonon Dynamics using Monochromated Electron Energy-Loss Spectroscopy

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Imperfections such as stacking faults and interfaces are recognized as controlling factors in modifying thermal properties and heat transport by scattering phonons and changing vibrational spectra. They play a more pivotal role when the material's dimension is reduced to the nanoscale. However, it is traditionally difficult to obtain experimental evidence of defect-induced phonon modes and phonon dynamics due to the lack of effective tools with sufficient spatial resolution. Meanwhile, in a perfect crystal, the vibration of individual atoms, known as thermal ellipsoids, could exhibit strong anisotropies and fundamentally affect the orientation dependency of dielectric, thermal and optical properties. The averaged thermal ellipsoids were conventionally estimated by diffraction methods, which encounter critical drawbacks of lacking spatial and energy resolutions. Monochromated electron energy-loss spectroscopy (EELS) in the advanced electron microscope reached an unprecedented energy resolution of a few millielectronvolts (meV), enabling the detection of phonon modes in materials with nanoscale and even atomic resolution. In this talk, I will present several examples that utilize a series of space- and angle-resolved vibrational EELS methods to investigate exotic vibrational states and phonon dynamics in diverse scenarios. (1) At a single stacking fault in SiC, the acoustic vibration modes at the X point undergo a red shift of 3.8 meV with increased spectral intensity and such defect phonons are confined to within a few nanometers of the defect. (2) Localized interfacial phonon modes were revealed in both Si-Ge heterojunctions and monolayer MoS<sub>2</sub>-WSe<sub>2</sub> heterointerfaces and could promote the thermal transport across solid-solid interfaces. (3) A differential phonon momentum mapping method was developed and utilized to observe a strong specular reflection of Si optical phonons at compositionally abrupt interfaces between Si and SiGe quantum dots. (4) We recently developed a novel momentum-selective dark-field EELS method with a desired momentum exchange and probed unexpectedly frequency-linked thermal ellipsoids of individual oxygen atoms in SrTiO<sub>3</sub>. Our work charts a definitive course for investigating phonon propagation in perfect crystals and near defects and provides guidance for the thermal nanoengineering of nanotransistors, power electronics, and thermoelectric devices.

This work was supported by the Department of Energy, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering (DE-SC0014430), and partially by the NSF under grant number DMR-2034738. The authors acknowledge the use of facilities and instrumentation at the UC Irvine Materials Research Institute (IMRI) supported in part by the National Science Foundation through the Materials Research Science and Engineering Center program (DMR-2011967).

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# **Extending the Capabilities of Energy- and Momentum-Resolved STEM**

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Although vibrational electron energy-loss spectroscopy had already been demonstrated in the 1960s [1], the possibility to do so in an actual electron microscope with a sub-nm probe was only established in 2014 [2]. While specialized optical vibrational spectroscopy techniques have attained sub-nm resolution by now [3], the electron microscope is still unique in being the only instrument capable of atomically resolving bulk-like specimens not just surfaces. Recently, we have demonstrated atomically-resolved phonon EELS of extended defects – in excellent agreement with calculations [4]. This study has shown that grain boundaries are not only barriers to phonon transport but can also support localized phonon modes and thus potentially act as waveguides for phonons in future devices.

Another promising application is momentum-resolved vibrational EELS first demonstrated by Hage et al. in 2018 [5]. Here, we experimentally map phonon dispersion surfaces (in 2D) from momentum-resolved vibrational EELS [6] combined with quantitative comparison with suitable theory [7]. The technique could be used to visualize anisotropies in phonon transport, e.g. in steady states, or to investigate mode softening.

The utilization of vibrational EELS to probe biologically-relevant materials without damage (in aloof geometry) was shown by Rez et al. in 2016 [8]. We have advanced this technique and will demonstrate the first combination of vibrational spectroscopy and cryo-transfer to investigate beam damage in vitrified samples [9]. We observe that hydrogen atoms are break off first and we also observe the partial reforming of bonds.

Lastly, we will demonstrate how to energy-resolve 4D-STEM at the atomic scale, leading to rich 5-dimensional data sets. The practical implementation using the registration of 4D-STEM [10] and first results for core-loss [11] as well as vibrational energies will be shown and potential applications discussed.

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# Progress, prospects and challenges for *in situ* transmission electron microscopy of electrical and magnetic switching processes in functional materials and nanoscale devices

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The local electrical and magnetic properties of nanoscale materials and devices such as transistors, field emitters and memory cells can be affected by the presence of defects, dopants, interfaces and surfaces, as well as by local variations in morphology, chemical composition and crystallographic orientation. We are developing both diffraction contrast and phase contrast techniques in the transmission electron microscope, in combination with model-based and model-independent data analysis, for the study of electrical and magnetic switching processes in functional materials and nanoscale devices. I will illustrate how such measurements can be made in the presence of external stimuli such as applied voltage, temperature, reactive gas and light. I will highlight key experimental issues that need to be considered, including the influence of sample preparation, dynamical diffraction and electronbeam-induced charging. As a representative example, Fig. 1 illustrates the switching of an individual Ag-In-Sb-Te resistive-switching-based phase change memory device between a low resistance state and a high resistance state in the transmission electron microscope using short (<50 ns) current pulses. I will conclude with a perspective on future developments in instrumentation and techniques that may allow such measurements to be made with greater reliability, sensitivity, precision and accuracy.



Figure 1. Annular dark-field scanning transmission electron microscopy images recorded during the switching of a Ag-In-Sb-Te phase change memory line cell. a) Pristine crystalline low resistance state (LRS) switched using a square pulse of duration 50 ns to b) a high resistance state (HRS). An amorphous region is outlined by a red dashed line. Images recorded after first and second "sweep" pulses (of duration 100 ns) are shown in c) and d) for the HRS and LRS, respectively. A crystalline conducting channel is marked with a blue dashed line. e, f) Subsequent switching from the HRS to the LRS. The scale bar is 80 nm.

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# *Ex situ* and *in situ* Scanning Transmission Electron Microscopy Studies of Functional Perovskite Materials and Devices

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Functional perovskite materials, especially oxide-based and halide-based perovskites, offer abundant physical properties for the development of information and energy conversion devices. Characterization techniques such as aberration-corrected scanning transmission electron microscopy (STEM) and differential phase contrast (DPC) provide crucial insights into the atomic structures and various physical information of materials, playing an important role in material and device development. To investigate the bending-related phenomena in ultrathin freestanding oxide perovskites, advanced STEM approaches were adopted to characterizing the cross-sectional atomic structures of wrinkled BiFeO<sub>3</sub> (BFO) and SrTiO<sub>3</sub> (STO) membranes. Our results show that both membranes can accommodate extremely high strain gradients up to  $10^7$ /m. Moreover, the maximum polarization reached approximately 146.6  $\mu$ C/cm<sup>2</sup> and 35.3  $\mu$ C/cm<sup>2</sup> in BFO and STO, respectively, contributed by flexoelectricity. Additionally, we discovered a substantial change in membrane thickness in bent BFO, implying an unusual flexural deformation in ferroelectric membranes.

The polarization switching behaviors in ferroelectric materials play a significant role in information storage. Here, we present an *in situ* biasing approach for the direct atomic-scale STEM observations of domain nucleation and sideways motion in ferroelectric capacitors with uniform electric field. At lower electric field, the motion of ferroelectric domain walls exhibited creeping characteristics accompanied by continuous deformations. Atomic-resolution imaging of creeping domain wall also revealed concurrent variations in both the magnitude and orientation of polarization during the ferroelectric polarization switching process.

As emerging optoelectronic materials, halide perovskites have been widely applied in devices such as solar cells. However, their sensitivity to high-energy electrons still poses challenges for the study of atomic structure and dynamic behaviors. To realize the high-resolution characterizations of halide perovskites, post-protection treatment with ultra-thin carbon film was adopted to enhance the structural stability of perovskite solar cell cross-sectional specimens, optimizing the imaging conditions for STEM characterization. Low-dose STEM imaging revealed the atomic structures of grain boundaries, stacking faults, and twin boundaries within halide perovskite grains, corresponding theoretical calculations indicated that the interaction between point defects and intragrain interfaces could have detrimental influences on device performance. Furthermore, to investigate the photo-induced structural evolution and degradation process of halide perovskites, an *in situ* S/TEM observations under light illumination. During the photo-induced degradation process, nanoscale halide particles first appeared within the halide perovskite grains, then gradually expanding with accumulated

illumination time. The generation of halide particles also introduced significant intragrain strain, which could accelerate the performance degradation of solar cell devices.

**Key words**: oxide perovskite thin films; perovskite solar cell; scanning transmission electron microscopy; *in situ* TEM

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# *In situ* transmission electron microscopy on two-dimensional ferroic chalcogenides

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Current electronic devices have encountered grand challenges — How to further scale down the device size, upgrade the integration of input/output, memory and computing units, and reduce the energy consumption. In particular, the massive communication between the computational unit and memory is hard to afford within current architecture of electronic devices and computers. New paradigm of in-memory-processing architecture has been put forward recently. Beyond current von Neumann systems, two-dimensional (2D) ferroelectrics (FE) with miniaturized dimension, high speed and high sensitivity, and robust ferroic order with memory functionalities, are superior candidates for next-generation in-memory computing devices. Moreover, the facile phase transition in 2D materials potentially offers another degree of freedom to manipulate the non-volatile memory states. Therefore, the 2D polymorphism and 2D ferroelectrics and ferroelastics indeed provide promising solutions to the aforementioned challenges.

In this presentation, we will clarify the ferroic ordering and their physical origin, and introduce how to control/manipulate the phase transition and the ferroelectricity as well as ferroelasticity in 2D and build novel devices. We applied a variety of in situ transmission electron microscopy techniques (Fig.1A-C), specifically employing in situ mechanical manipulation, in situ mechanical testing, in situ electrical testing, in situ heating, and in situ electron beam control, to conduct comprehensive investigations of ferroic phase transitions and ferroic ordering in two-dimensional (2D) chalcogenides (Fig. 1D). The diverse phases observed in these ferroic 2D materials showcase unique mechanical, electrical, and other captivating physical properties. Through our study, we successfully established a direct correlation between atomic-scale structures and device-level performance (Fig. 1E), thereby enhancing our understanding and enabling practical applications of 2D functional materials.

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*Fig.1 A-C,* in situ TEM study on 2D materials. *D*, phase engineering on 2D In2Se3. *E*, memory transistor devices build on 2D In2Se3.

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# Understanding Electrocatalyst Re-structuring during Reaction with Electrochemical Cell Transmission Electron Microscopy

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Understanding the nature of functional materials in their operating environments is crucial for unravelling their structure-property relationships and enabling the design of materials with better performance. In recent years, there have been significant strides in instrumentation that allow us to study samples within liquid and gas environments inside a transmission electron microscope (TEM) [1]. Particularly. electrochemical cell TEM (EC-TEM) is emerging as a powerful technique for studying the morphology of electrocatalysts in an electrolyte and under applied potential. In this seminar, I will discuss the work in my group using EC-TEM to reveal the structural transformations that take place in catalysts under reaction conditions. First, I will briefly cover basic concepts of EC-TEM. Then, I will show using examples from our work on Cu-based electrocatalysts for electrochemical CO<sub>2</sub> reduction how complex structural motifs are created due by the restructuring of pre-catalysts under applied potential and the impact that these motifs have on the electrocatalysts' properties [2. 3]. Lastly, I will briefly touch on our current work combining operando EC-TEM and time-resolved energy-dispersive X-ray spectroscopy to track both catalyst morphology and chemical state during reaction [4].

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# In situ electron microscopy for electron radiolysis effect and dose dependence of functional metal oxides

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Advancements of aberration-corrected electron optics system and data acquisition schemes have enabled TEM to acquire images with sub-Ångström resolution and single-atom sensitivity. However, bottleneck of radiation damage is still challenging TEM imaging of soft materials and Knotek-Feibelman effect materials at the atomic level. For the radiation damage, it has been shown that the electron beam may not only alter the shape and surface structure at atomic level, but also induce radiolytic artifacts in chemical reactions at nanoscale, e.g., photocatalysis, metal-insulator transformation (MIT), super-dissolution of oxides [1, 2, 3]. The electron beam effects at nanometer resolution are discussed in this work. One example shows a dose-rate dependence of hydrogen bubble generation accompanying with water dissociation of Pt loaded TiO<sub>2</sub> nanoparticles in a liquid cell during in situ TEM observations. As the dose rate of electron beam is setup to be order of  $1 e^{-A^{-2} \cdot \sec^{-1}}$ , the hydrogen bubble appears at around 240 seconds, while as the dose rate is increased to the order of  $10^3 \text{ e}^{-\text{Å}^{-2}} \cdot \text{sec}^{-1}$ , the appearance of hydrogen bubble is faster by 4 times (at around 60 seconds). It clearly shows that the in-situ observations of photocatalysis can really mix up with radiolysis from the electron beam. Another example, an insulator phase of VO<sub>2</sub> is known to transform to metal phase at 68°C. A nano probe at high electron dose-rate can stimulate phase transformation from insulator phase to metal phase in a VO<sub>2</sub> nanowire even at room temperature.

The electron beam effect must be taken into account for interpretation of in-situ EM observation. The dose-rate of incident electrons becomes a very critical and inevitable degree of freedom that must be controlled to surpress radiolysis artifacts and to reveal pristine structure at atomic resolution. In the present paper, we present a systematical analysis of electron microscopy methodology towards probing in-situ dynamics at high temporal resolution and emphasize in particularly the role of controlling the electron delivery.

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| Biography   | <b>Manling Sui</b> is a Chair Professor and the Director of Academic<br>Committee of Faculty of Materials and Manufacturing at Beijing<br>University of Technology (BJUT). She received her Ph.D. in 1991 from<br>Institute of Metal Research, Chinese Academy of Science, and<br>successively worked at Northeastern University (China), University of<br>Wisconsin-Madison (USA), and Institute of Metal Research, Chinese<br>Academy of Sciences. Then she joined BJUT as a Chair Professor of<br>Cheung Kong Scholars Programme in 2009. Her research is focused on<br><i>in situ</i> electron microscopy <b>for</b> structure and property relationship in<br>advanced materials, mechanisms for the performance and structure<br>evolution of energy and catalytic materials in the applied fields (heat,<br>electricity, light, etc.) and environment (liquid and gas), and low dose<br>electron microscopy for the study of electron beam sensitive materials.<br>She has published more than 200 papers in the peer-reviewed journals,<br>including <i>Science, Nature Materials, Nature Photonics, Nature<br/>Communications, Physical Review Letters, Advanced Materials, ASC<br/>Nano, Nano Letters, Acta Materialia etc.</i> Her papers have been cited<br>more than 10000 times by SCI papers. |  |

# Accurate Retrieval of Three-Dimensional Atomic Dynamics of Morié Materials

#### Shih-Wei Hung<sup>1</sup> and Fu-Rong Chen<sup>2</sup>

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Determination of the three-dimensional (3D) atomic positions of materials is the key factor to understand the physical properties. Visualization of 3D atomic structure of nano-scale crystalline objects has been demonstrated from in-line holography by low dose rate electron microscopy. However, this method needs to resolve individual atom column along projection of electron beam. Here, an analytic method based on simulated annealing and energy minimization algorithms is proposed to retrieve the 3D atomic dynamics of moiré materials, such as single-walled carbon nanotube and twisted bilayer graphene, in which atomic column is not well-resolved and moiré fringe exists. The accurate determination of atomic positions can reveal the detailed structural properties, such as deformation field and surface roughness. This ability to probe structural change in time series is pivotal to understand the structurefunction relationship of nanomaterials.

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| Biography   | Dr.Shih-Wei Hung received his PhD degree from National Tsing<br>Hua University in 2012. He then worked as a postdoc at Tokyo<br>University, Japan in 2014 before joining City University of Hong Kong<br>in 2018.<br>He conducts research on methodology development of three-<br>dimensional atomic resolution dynamics reconstruction for the soft<br>materials imaging with aberration corrected environmental<br>transmission electron microscope. In addition to electron microscopy,<br>he also investigated material properties at atomic scale, such as<br>biomolecule adsorption, wettability, and thermal boundary<br>conductivity, using molecular dynamics simulations. |

# Making every electron count – Ptychography at low dose

Angus Kirkland<sup>1,2</sup>

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This talk will describe recent developments in the use of Electron Ptychography for studies of materials, including biological structures at low electron fluence.

Importantly for this application, ptychography is an inherently dose efficient technique, enabling effective phase reconstruction of radiation sensitive samples. I will particularly highlight recent developments in optimised scanning and data processing using machine learning for reductions in radiation damage and noise reduction in low fluence data sets.

In the life sciences Cryo-electron ptychography holds much promise particularly when used with a defocused probe to scan across a specimen with highly overlapped probe positions. This can be applied in a variant of conventional single particle analysis to provide 3D structures taking advantage of the known resolution variation of the effective ptychographic transfer function with convergence angle to provide wide spatial frequency bandwidth transfer. This geometry also allows datasets from wide fields of view to be collected that are suitable for studies of biologically relevant structures in a cellular context.

Finally Fourier Ptychography as an alternative data acquisition and processing strategy will be discussed.

| Full name      | Angus Kirkland   |  |
|----------------|--|--|
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| Distinction    |  |  |
| D's succession | Professor Angus Kirkland was awarded his MA and PhD from the<br>University of Cambridge and has held the posts of Professor of<br>Materials at Oxford since 2005 and JEOL Professor of Electron<br>Microscopy since 2013. In 2016 he was appointed as Director of the<br>National Physical Sciences Imaging Centre at Diamond Lightsource<br>and is Science Director at the recently established Rosalind Franklin<br>Institute. |  |
| Biography      | He was awarded the MSA prize in 2005, the Rose prize in 2015, the<br>Quadrennial prize of the European Microscopy Society in 2016 and the<br>Agar Medal for Electron Microscopy in 2017.<br>He served as General Secretary of the International Federation of<br>Societies for Microscopy in from 2014 -2018 and was elected President<br>in 2018.   |  |
|                | He has also served as Editor in Chief of Ultramicroscopy since 2010  |  |

# Atomic Resolution 3D Dynamics of Helix Materials: present and future

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Advancements of aberration-corrected electron optics and data acquisition schemes have made TEM capable of delivering images with sub-Ångström resolution and single-atom sensitivity. However, three bottlenecks, namely, radiation damage, static imaging and 2D projection geometry are still challenging TEM imaging of soft materials at the atomic level. For a crystalline material, the 3D atomic information of was determined from the maximum propagation intensity of the atomic column wave relative to the vacuum wave and/or refined by the big-bang method.[1,2,3] The dynamics is obtained from tracking the 3D atom positions deduced from time-resolved exit wave functions reconstructed from sub-sets of defocus series of images [3]. This method well applied to crystalline materials where the atom columns are isolatedly resolved. However, because most of the projected spacings between atoms from an individual helix C60, carbon nanotube and DNA are beyond the resolution of the TEM, it is still difficult to directly resolve its atomic columns in the image mode. Furthermore when rather isolated atomic columns are resolved, the moiré pattern formed by the rolled-up graphene layer is clearly visible between two intense bright lines corresponding to the vertical tube walls. Each bright spot in the images may be associated with a cluster of atoms in the projection view.

In the present paper, we present advancements of electron microscopy methodology towards probing 3D atomic resolution dynamics of helix materials, CNT, C60/ CNT and DNA, at high temporal resolution and emphasize in particularly the role of controlling the electron delivery. We develop a genetic evolution method utilizing iterative simulation annealing and energy minimization (SA-EM) to extract all spatiotemporal information encoded in the transmission electron microscopy (TEM) data up to the limits of the counting statistics. In my talk, atom dynamics in 3D of CNT, C60 and DNA will be presented. And the future trend of noninvasive TEM on the 3 D atom dynamics will be also presented.

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#### The authors would like to thanks support from all UCG projects in Hong Kong

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| Distinction | 2019, Microscopy Society of American Innovation aw<br>Ultrafast/ Ultra High Voltage Desktop Electron Micro   | scope           |
|             | 2016, The 51th San-Yat Sen Academic and Cultural F<br>Award: Science Category  | Foundation      |
|             | 2015, Institute for Biotechnology and Medicine Indus<br>national prize for enterprise innovation   | try: The 12th   |
|             | 2013, Ministry of Science and Technology (MOST): I<br>Research and Academic Award in Nanotechnology  | Excellent       |
|             | 2013, MOST "Excellent Start-Up Company award in and "Excellent Start-Up Company Award"   | Nanotechnology" |
|             | 2013, European Microscopy Society Outstanding pape   | er Award        |
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|           | Prof. Fu-Rong Chen<br>Associate Vice-President, City University of Hong Kong<br>Director, City University of Hong Kong, Shenzhen Futian Research   |
|-----------|--|
|           | Institute<br>Director, City University of Hong Kong Shenzhen Research Institute<br>Director, City University of Hong Kong Chengdu Research Institute   |
|           | Professor Fu-Rong Chen received his bachelor's degree in Materials<br>Science and Engineering at National Tsing Hua University in Shinchu in<br>1980. He obtained his Ph.D. in Materials Science from Stony Brook<br>University, USA in 1986 and started his postdoctoral career as research<br>associate at MIT from 1986 to 1988. From 1988 to 1990, he worked as<br>research assistant professor at Northwestern University, USA.   |
|           | 1990, Prof. Chen Furong joined National Tsinghua University and was promoted to the position of Chair Professor in 2017.   |
|           | 2018, Prof. Chen joined City University of Hong Kong as Chair<br>Professor and served as Director of City University of Hong Kong<br>Shenzhen Futian Research Institute.   |
| Biography | Prof. Chen has long been engaged in the research field of materials<br>science and electron microscopes. His research interests are in low-dose<br>3D atomic resolution dynamics, soft materials dynamics imaging,<br>quantum electron microscopy, and solar energy tunable (SET) glass.   |
|           | During his research career, Prof. Chen published more than 300 SCI papers in high-impact journals such as Nature, Nature Materials, Nature Communications, PNAS, etc. and he received many awards and honors, including The best paper in the year in Europea Microscopy Society 2012, The Microscopy Society of American Innovation Award for the Design of an Ultrafast/ Ultra High Voltage Desktop Electron Microscope in 2019 and The Distinguished Professor of Tsing Hua University in 2012. He received an average of 5 invited talks for international conferences per year. |
|           | In the field of industrialization of scientific achievements, he has<br>founded two high-tech companies, engaged in the research and<br>development of electrochromic energy-saving smart glass and compact<br>electron microscope.  |

# Developing Atomic Resolved Mechanical Testing System and Measuring Grain/Twin Boundary Plasticity at Atomic Level

#### Xiaodong Han<sup>1, 2</sup>

#### <sup>1</sup>Beijing University of Technology, 100081, Beijing, P. R. China <sup>2</sup>Southern University of Science and Technology, Shenzhen, China

How to characterize and measure the interface phenomena on the microscopic level is one of the most fundamental questions [1]. We report here the characterizing and measuring of plasticity properties of grain and twin boundaries at microscopic level, particularly at nano and atomic scale. Mechanical-thermal-electrical functional instruments are developed to accommodate the sub-A spatial resolution with time-resolved abilities [3]. By monitoring and measuring grain and twin boundaries' plasticity at atomic level, it is discovered that: large angle unsymmetrical tilt grain boundaries slide by intrinsic dislocations climb and extrinsic disconnection slide [4]. The interactions of sliding extrinsic disconnections with intrinsic GB dislocation-disconnection pairs accommodate GB sliding by GB atom transfers. For the TB, dislocations pin, pile up and cross-slip are directly revealed and uncovered. Finally, we report a new nucleation route of deformation twin through alternated stacking faults to detour the extremely high twin fault energy in nano-crystalline Pt, which is in contrast to the classic layer by layer emission stacking fault route for twin nucleus.

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|             | Chair Professor,  |
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|             | Beijing, China.   |
|             | Researches include:   |
|             | 1.Developing atomic-resolved in situ microscopy techniques and        |
|             | methods;  |
|             | 2. X-ray detection and photon/electron detection materials and        |
|             | techniques;   |
|             | 3. Build materials structure-property relationships;                  |
| Biography   | 4. Atomistic mechanisms of dislocations and grain/twin boundaries;    |
|             | 5. Developing high strength yet ductile metals and alloys.            |
|             | Publishing more than 270 scientific papers including Science.         |
|             | Nature, Nature series journals, Phys. Rev. Letts., Adv. Mater., Acta  |
|             | Mater., etc. Awarded National Nature Science Prize (2nd-Class, 2021), |
|             | First-Class Prize of Beijing Science and Technology Progress.         |

# From functionalizing inorganic two-dimensional materials on the level of single atoms towards molecular imaging of organic two-dimensional materials

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Prerequisite for functionalizing two-dimensional inorganic materials using the electron beam in a transmission electron microscope is the detailed understanding of the beam electron - sample interactions. We derive this basic knowledge from atomically-resolved, time-dependent in-situ TEM imaging of inorganic two-dimensional (2D) transition metal dichalcogenides (TMDs) using the chromatic- and spherical-aberration-corrected low-voltage SALVE instrument operating in the voltage range between 80kV and 20kV.

We show for different single-layer TMDs that in dependence of the electron accelerating voltage and applied electron dose the defect formation can be initiated. Applying this knowledge, in-situ and ex-situ structural and chemical transformations of different freestanding TMDs and of more rarely reported TMPTs (TM phosphorus tri-chalcogenides) are performed and verified by complementary ab-initio calculations.

For lateral heterostructures we show near-atomically sharp junctions with a typical extent of 3 nm for the covalently bonded MoSe2-WSe2 interface and use this knowledge to explain the considerably narrowed optical transition linewidth in the PL and Raman spectroscopy. Further we show proof-of-principle experiments in which we transfer electron-exposed TMD flakes from a TEM grid to arbitrary substrates to directly relate the results of the photoluminescence and transport measurements to their structural origin.

For vertical few-layer heterostructures we study the effect of interlayer excitons in WSe2 located in the low-loss range of the EELS spectrum. In few-layer graphene heterostructure we discuss the Li crystal nucleation mechanism from in-situ studies of a miniaturized electrochemical cell, where reversibly single-crystalline bilayer graphene is lithiated and delithiated in controlled manner using an electrochemical gate confined to a device protrusion.

The knowledge gained for the study of 2D inorganic materials we apply to the study of 2D polymers and 2D metal-organic frameworks (MOFs), however, not surprisingly, functionalization and even atomically-resolved imaging is hindered due to much lower resilience of the organic material during electron irradiation. We present key strategies to achieve nevertheless higher resolution in high-resolution TEM images of imine-based 2D polymer films, which include the selection of a surprisingly low electron accelerating voltage of 120kV for achieving a resolution of 1.9A, enabling imaging the linker molecules. Further, we study experimentally and computationally the role of different organometallic bonds and hydrogen content on electron radiation stability, using a group of four structurally similar Cu-based 2D MOFs.

We summarize our results in one sentence that 2D materials and lower-voltage atomic (molecular) resolution TEM/STEM are just made for each other.

| Full name   | Ute Anneliese Kaiser   |
|-------------|--|
| Affiliation | Professor / University Ulm / German  |
| Biography   | Ute Kaiser received her Diploma (1976) and her PhD in<br>Crystallography (1993) from the Humboldt University Berlin, her<br>Habilitation in Experimental Physics from the Friedrich-Schiller<br>University, Jena, Germany, in 2002. From 2004 till 30 <sup>th</sup> of September<br>2023 she was full professor at Ulm University in the Physics<br>Department and Head of Ulm's Materials Science Electron<br>Microscopy Centre. Starting from 1 <sup>st</sup> of October she is senior professor<br>at Ulm University at the Institute for Quantum Optics. |

# The Principal of Aberration Corrected 300 kV Cryo-EM and Its Application to Thick Specimens in Biology

Jing Wu<sup>1</sup>, Yuanzhu Gao<sup>1</sup>, Lei Zhang<sup>1</sup>, Chuang Liu<sup>2</sup>, Peiyi Wang<sup>1</sup>

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High-resolution 3D structures of cells, tissue, virus, or other biological macromolecules complex in their native environment can be achieved by Cryo-ET in situ and is considered to be a bridging between molecular biology and cellular biology. Compared with Cryo-EM SPA (single particle analysis), Cryo-ET is not perfect in many aspects and has no mature workflow with some technical bottlenecks that need to be solved urgently. One major matter is the degradation of image quality when the sample thickness increase. In general, the thickness of biological sample suitable for Cryo-ET study is limited to 150~200 nm. This is because when the electron beam travel through a thick sample, the inelastic scattering occurs due to the energy loss. As sample thickness increases, more and more inelastic scattering will generate. The electrons with various wavelength arriving at the back focal plane of the objective lens have different cross-over, caused the chromatic aberration (Cc) which decrease the image quality dramatically. CEOS Cc-corrector is one of the most important inventions in the electron microscope history. It can significantly improve the information limit and reduce the focus spread caused by energy loss. Cc-corrector also affects the envelope function of contrast transfer of the instrument. The successful application of Cc-corrector in material science inspires us on introducing a Cc corrector to the Cryo-EM community. We report here the progress and preliminary results of the world's first 300kV cryo-electron microscope equipped with CEOS Cs/Cc aberration corrector and TFS selectris imaging filter in the Cryo-Electron Microscope Center at the Southern University of Science and Technology.

| Full name   | Peiyi Wang (王培毅)   |  |
|-------------|--|--|
| Affiliation | Professor, Cryo-EM Center, Southern<br>University of Science and Technology,<br>Shenzhen, China  |  |
| Distinction | A member of the Royal Microscopy Society, the British<br>Physical Society, the British Biochemical Society, the<br>Chinese Physical Society, the Chinese Electron Microscopy<br>Society.   |  |
| Biography   | Professor Wang is a Cryo-electron microscopist, Director<br>of Cryo-electron Microscopy Center at the Southern<br>University of Science and Technology. He studied for the<br>degree of D. Phil in the Department of Materials at the<br>University of Oxford in the early of 1990's, and then worked<br>as a research fellow at serval universities in France and the<br>UK. In 2000, he was granted a permanent position at the<br>University of Sheffield, and moved to the University of<br>Leeds, UK in 2007 before jointed the Southern University of<br>Science and Technology in 2018. His research interests<br>mainly in the application of high-resolution electron<br>microscopy in the field of materials science and the structural<br>biology. The research areas include macromolecular complex<br>and viruses. He has published more than 60 articles in<br>"Nature", "Science", "Cell" and other internationally<br>renowned journals. |  |

# Cryo-electron tomography and subtomogram averaging for highresolution structure determination of macromolecules

#### Tao Ni\*

\*School of Biomedical Sciences, LKS Faculty of Medicine, The University of Hong Kong, Hong Kong SAR, China.

Cryo-electron tomography and subtomogram averaging are rising and fast-evolving imaging techniques to study biological events, providing structural information at an unprecedented resolution while preserving spatial correlation in their native contexts. The latest technology and methodology development ranging from sample preparation to data collection and data processing, has enabled significant advancement in its applications to various biological systems. I will present an overview of the current technology development enabling high-resolution structural study *in situ*, highlighting the use of *a priori* information of biological samples to assess the quality of subtomogram averaging pipeline. I will exemplify the applications of this technique to understanding viruses and principles of macromolecule assembly using different biological systems, ranging from *in vitro* to *in situ* samples, which provide structural information at different resolutions and contexts. Finally, the challenges of cellular cryo-electron tomography will be discussed.

| Full name   | Tao Ni (倪濤)  |
|-------------|--|
| Affiliation | Dr, The University of Hong Kong, China   |
| Biography   | Dr. Tao Ni is an Assistant Professor in the School of Biomedical<br>Sciences at the University of Hong Kong from 2022. Prior to<br>joining to HKU, he was a postdoctoral research fellow at the<br>University of Oxford and visitor scientist at eBIC working on<br>HIV capsid and carboxysomes using cryoEM and cryoET<br>subtomogram averaging. He completed his Bachelor in<br>Biological Sciences degree in Nankai University (2008-2012)<br>and PhD in structural biology in the University of Oxford (2012-<br>2016). His research focuses on the interface between host and<br>pathogens to decipher their relationship in the molecular level<br>using cryo-electron tomography. |

#### **Tunable X-ray Radiation from Quantum Free-electron Radiation**

Xihang Shi<sup>1</sup>, Michael Shentcis<sup>1</sup>, Yaniv Kurman<sup>1</sup>, Liang Jie Wong<sup>2</sup>, F. Javier García de Abajo<sup>3</sup>, and Ido Kaminer<sup>1</sup>

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Tunable control of X-rays remains an open challenge with crucial implications for high- resolution X-ray spectroscopy, medical imaging, and more. However, the necessary optical elements in the X-ray regime are bulky and inefficient, strongly limiting the application of X- rays compared to the rich possibilities in the visible and IR regimes. In response, we propose a novel approach of generating tunable X-ray emission, relying on **coherent interactions between free electrons and crystalline materials**.

Coherent electron interactions are generally described by two separate processes – parametric X-ray radiation (PXR) and coherent Bremsstrahlung (CB)<sup>1</sup>. Both PXR and CB are intrinsically linked to the crystal structure, enabling control of the radiation via engineering of the crystal structure. We first demonstrated this scheme in transmission electron microscopes (TEMs) using van der Waals (vdW) materials<sup>2</sup>, serving as a versatile platform for engineering X-ray sources. Our most recent developments of this approach include two schemes for creating **structured X- ray beams** using vdW heterostructures: X-ray caustics<sup>3</sup> and focused-X-ray beams<sup>4</sup>. Such beams are created directly at the sources, bypassing the need for X-ray-optical elements.

Furthermore, we also discovered a regime of electron-driven X-ray radiation in which the quantum nature of electrons and light plays critical roles in the radiation process<sup>5</sup>. Until our work, the classical approach was sufficient for describing free-electron radiation, with only marginal quantum corrections. We identified conditions under which an entirely new radiation mechanism emerges, driven by the entanglement between the emitted photons and the electrons.



(a) Caustic X-ray beams created via parametric X-ray radiation from electrons passing through a strained crystal structures.

(b) Free electrons scatter elastically when passing through periodic crystals, generating highly entangled electron-photon joint states.

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| Distinction | I am conducting theoretical research on free-electron radiation and<br>light-matter interaction. My work focuses on studying the interactions<br>in the quantum regime and proposing novel light source schemes. |  |
| Biography   | I obtained my Bachelor's degree from Sun Yat-Sen University in<br>China, and PhD from Nanyang Technological University in Singapore.<br>Currently, I am engaged in postdoctoral research at Technion, Israel.    |  |

# Mapping and controlling of optical near fields in an ultrafast transmission electron microscope

Murat Sivis

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Electron microscopy allows for the mapping of optical properties of metallic and dielectric nanostructures via cathodoluminescence [1] or electron-energy-loss spectroscopy (EELS) [2]. In recent years, a new method, photon-induced near-field electron microscopy (PINEM) [3], has been established in ultrafast transmission electron microscopes, enabling quantitative measurements of near-field strengths [4]. In this new approach, ultrashort laser pulses (picosecond to femtosecond pulse duration) excite specific spectral modes of a sample, and pulses of high-energy electrons interacting with the associated near-fields experience stimulated energy gain and loss. In contrast to EELS, which probes the intrinsic properties of a nano-optical system with a spectral resolution limited by the electron microscope used (sub-100 meV with a monochromator), PINEM provides an access to the extrinsic optical modes with a spectral resolution limited only by the spectral bandwidth of the laser. This is achieved by electron-energy gain spectroscopy (EEGS), where the near-field strengths are measured for different laser wavelengths [5]. This makes PINEM and EEGS powerful additions to the electron microscopy toolbox.

In this talk I will give an overview of our efforts in the Göttingen UTEM project [6] to exploit these new capabilities of ultrafast transmission electron microscopy for mapping and controlling optical near-fields in metallic and dielectric nano- and microstructures [7,8]. I will present recent studies on mode selective reconstruction of plasmonic near fields [9], measurements of the time evolution of such near-fields with attosecond precision and illustrate how we intend to use these near fields for atomic gas excitation and for probing nonlinear optical excitations.

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| Affiliation | Senior scientist and project leader, Max-Planck<br>Institute for multidisciplinary sciences and University<br>of Göttingen Germany/Göttingen  |  |
| Distinction | Condensed matter physics, Ultrafast nano-optics, and Nonlinear strong-field phenomena and ultrafast electron microscopy   |  |
| Biography   | Education:<br>Dr. rer. nat. (Physics) 2013, Georg-August University, Götting<br>Germany<br>ŸDiploma (Physics) 2009, Georg-August University, Götting<br>Germany<br>Professional experience:<br>2009 – present: Georg-August University- 4th Physical Institu<br>Göttingen, Germany<br>Ÿ2016 (April- July): Research stay at the National Research Cour<br>Canada in Ottawa and University of Ottawa (Group of Prof. Dr. Paul<br>Corkum)<br>2020 – present: Max-Planck Institute for multidisciplinary sciences<br>Murat Sivis is a senior researcher and project leader at the University<br>Göttingen and the Max-Planck Institute for multidisciplinary sciences<br>Göttingen. He received his undergraduate education in physics at<br>University of Göttingen and earned a PhD in Physics in 2013 under<br>supervision of Prof. Claus Ropers. In 2016 he was a visiting research<br>at the National Research Council Ottawa in the group of Prof. P<br>Corkum.<br>He is part of the Göttingen Ultrafast Transmission Electron Microsco<br>(Gö-UTEM) project, where he studies electron-light interactions.<br>Dr. Sivis authored one book chapter and 23 journal articles. He receives<br>the Dr. Berliner-Dr. Ungewitter PhD thesis prize (2014) and the aw<br>for outstanding scientific publications by an early career scientist (20<br>of the University of Göttingen and the Lower Saxony science of<br>the University of Göttingen and the Lower Saxony science of the University of Göttingen and the Lower Saxony science of the University of Göttingen and the Lower Saxony science of the University of Göttingen and the Lower Saxony science of the University of Göttingen and the Lower Saxony science of the University of Göttingen and the Lower Saxony science of the University of Göttingen and the Lower Saxony science of the University of Göttingen and the Lower Saxony science of the University of Göttingen and the Lower Saxony science of the University of Göttingen and the Lower Saxony science of the University of Göttingen and the Lower Saxony science of the University of Göttingen and the Lower Saxony science of the University of Göttingen and the Low |  |

#### Nanophotonic electron accelerators towards electron microscopy

<u>Roy Shiloh</u><sup>1,2</sup>, Tomáš Chlouba<sup>2</sup>, Stefanie Kraus<sup>2</sup>, Leon Brückner<sup>2</sup>, Julian Litzel<sup>2</sup>, and Peter Hommelhoff<sup>2</sup>

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Nanophotonic dielectric laser acceleration (DLA) is a fast-evolving, emerging field aimed at providing a solution to the miniaturization of electron accelerators, down to the chip-scale [1–3]. Although the average acceleration gradients are limited by the material breakdown threshold (up to ~10 GV/m [4]), this technology currently offers acceleration of superb-quality single-electron pulses (normalized emittance ~100 pm-rad) at kHz and potentially MHz repetition rates, and can be easily implemented at academic-scale costs and settings. The opportunity to design accelerator structures and nanofabricate them in a university clean-room is a great advantage for cutting-edge research in quantum electron-light interaction [5], and to the recent proposals for the temporal modulation of electron wavepackets in the attosecond regime, potentially soon in the MeV energy range. In fact, so far, this experimental venture into DLA has been mostly based on electron microscopes, which provide the electron source, beam-forming and injector unit.

Considering this starting point, these chip-scaled accelerators can be naturally considered as a basis for advanced, high-energy, ultrafast electron microscopy of thick samples. As such, building-scale high-energy (e.g. the 3 MeV Hitachi microscope) electron microscopes could one day potentially be miniaturized and installed in any university lab.

In this talk, I will give an overview of the current state of nanophotonic DLA research, with the different schemes being pursued both theoretically and experimentally, some recent proposals and application directions, and the latest results in the sub-relativistic (~30 keV) regime [6–9].

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# 4D electron microscopy and its applications in non-equilibrium dynamics

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Four-dimensional electron microscopy (4D-EM), which enables the direct observation of transient structures, morphologies and even carrier transport of materials in real time and space, has attracted increasing interest to the research community due to its powerful capability in the interdisciplines of physics, chemistry, material science, and biology [1-2]. In this presentation, I will firstly give a brief introduction of the development of 4D-EM and the state-of-the-art of 4D-EM and its applications in scientific research. Then, I will present some of our recent developments in situ 4D EM technologies and their applications, including ultrafast cathodoluminescence, liquid-phase 4D EM, laser-free 4D EM and two-color near field 4D EM etc. [3-5]. Following that, I will talk about our recent progress in the development of a new generation 4D-EM based on a 200kV field emission transmission electron microscope (Thermofisher Talos 200i) and its preliminary application in imaging surface plasmon dynamics [6]. The high versatility and sensitivity of our new generation 4D-EM would allow capturing the dynamics of a wide range of nanoscale materials with high spatiotemporal resolution.

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# Instrumental developments in ultrafast transmission electron microscopy

#### <u>Alexander Schröder<sup>1,2</sup>, Christopher Rathje<sup>2</sup>, Niklas Müller<sup>1,2</sup>, Jonathan Weber<sup>1,2</sup>,</u> <u>Sascha Schäfer<sup>1,2,3</sup></u>

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Time-resolved variants of transmission electron microscopy have started to provide an unparalleled view into the fast and ultrafast dynamics of solid-state nanostructures (see for example [1-4]). A crucial instrumental pre-requisite for constructing the next generation of time-resolved electron microscopes is the development of novel pulsed electron sources, fast detectors and versatile sample excitation schemes. In this talk, I will summarize our recent developments and characterization of a laser-driven cold-field emitter source (in collaboration with JEOL Ltd.), including the achieved electron pulse duration, spectral width, and electron beam brightness. A first application of these high-brightness electron pulses for the imaging of wavelength-dependent optical near-fields in transition metal dichalcogenides will be presented.

In the second part, I will focus on the application of event-based TimePix3 electron detectors in fast electron imaging. Using a neural network approach trained by experimental data with synchronized femtosecond electron pulses, we can improve the time-resolution of the TimePix3 detector utilizing the intrinsic correlations within event clusters [5]. Finally, as two applications for event-based electron imaging, I will discuss the excitation and phase-resolved mapping of nonlinear Duffing modes in a silicon membrane, demonstrating quality factors exceeding 10<sup>5</sup>, and the time-resolved Lorentz imaging of photoinduced charging in metal nanoparticles [6].

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| Biography   | <ul> <li>Prof. Dr. Sascha Schäfer holds a chair in Experimental Physics at University of Regensburg/Germany. His research group "Ultra: Nanoscale Dynamics" focusses on the development of novel ultra: transmission electron microscopy (UTEM) instrumentation and the resolved electron imaging approaches with applications in the prob of local structural and spin dynamics and free-electron/li interactions.</li> <li>Prof. Schäfer received his Ph.D. in Physical Chemistry at the Un of Darmstadt and worked as a PostDoc from 2009 – 2012 together w A. H. Zewail at Caltech. In 2012, he joined the group of C. Ropers the Univ. of Göttingen as the sub-group leader of the UTEM teat During this time, the Göttingen UTEM team pioneered development of highly coherent laser-driven electron-li interaction in optical near-fields. At the University of Oldenburg, established his own research group in 2017 funded by a Lichtenb professorship of the Volkswagen Foundation. In 2023, his resear group relocated to the University of Regensburg joining Regensburg Center for Ultrafast Nanoscopy (RUN).</li> </ul> |  |

#### **Contrast formation in Quantum Electron Microscopy**

#### Pieter Kruit

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For most biological specimen, the present resolution of transmission electron microscopes is more than sufficient. The problem is the sensitivity to radiation damage which results in "doselimited resolution". The approach called "Quantum Electron Microscopy (QEM)" has tried to reduce the damage by applying principles from "non-demolition", also called "interaction-free" techniques from quantum optics.

In QEM an electron is split into two states, a sample state  $|S\rangle$  and reference state. The sample state is used as a STEM focused probe while the reference state passes through empty space. The same electron is cycled through the sample several times. The  $|S\rangle$ -state experiences phase shift  $\delta\phi$  passing through the sample  $|S\rangle \rightarrow e^{i\cdot\delta\phi}|S\rangle$ . There is also probability for the electron to collapse onto the sample in an (inelastic) interaction. The energy lost by the electron is the damage dealt to the sample. Since the electron was split so that the amplitude of the sample state was small, the probability of damage by the electron is very low.

Previously, we have analysed situations where the amplitude contrast dominated [1], but more recently we have also analysed the effects in samples where phase contrast dominates. The results can be compared to TEM phase contrast with a Zernike phase plate. However, the imaging mode is as in scanning transmission electron microscopy and has features that look like STEM differential phase contrast with a biprism splitter.

To compare QEM with other EM methods we estimate the dose limited phase resolution. Intensity variations in an image occur either deterministically (reflecting the variations in the sample) or stochastically (shot noise). The minimal phase sensitivity  $\Delta \phi$  should be so great that

the change in intensity is comparable with the shot noise: 
$$\Delta \phi \left| \frac{dC}{d\phi} \right| I = \sqrt{C(\phi)I}$$
, where  $C(\phi)$  is

the contrast function and I is the average number of electrons per pixel. Hence for  $C(\phi)I$  electrons detected the phase shift of the sample is within interval  $\phi \pm \Delta \phi$  with probability of 63%. Note that  $\Delta \phi$  is proportional to  $\sqrt{I}$  just as SNR in the shot noise limited measurements. For different microscopy techniques phase resolution can be written as  $\Delta \phi = F \cdot \frac{1}{\sqrt{D}}$ , where F is a (technique dependent) factor and D is the illumination dose (which is not equal to I in case of QEM). For instance, for TEM with an ideal Zernike phase plate  $F_{TEM} = 0.5$ . If we use the pessimistic estimate for the dose in QEM  $D_{max} = I \cdot N/2$ , then  $F_{QEM} \approx \frac{1.6}{\sqrt{N}}$ , where N is the number of cycles.

I shall discuss the conclusions we can draw from our work on Quantum Electron Microscopy.

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| Biography   | Pieter Kruit is emeritus professor of physics at Delft University of<br>Technology in the Netherlands. He has had a chair in charged particle<br>optics from 1989 until November 2022.<br>His research has always been related to the development of electron-<br>and ion-optical instruments, "trying to improve our eyes to look at the<br>microscopic world and the hands that we use to create a new nano-<br>world". He has had research programs on nm-resolution electron<br>spectroscopy, developments of low energy-spread electron- and ion<br>sources and multi-beam optics for microscopy and lithography. Most<br>of his work was performed in cooperation with industry. For his work<br>in electron lithography he founded, with two of his graduates,<br>MAPPER Lithography. His involvement in trying to reduce magnetic<br>disturbances from a planned tramway through the university has led to<br>a novel current supply system implemented in tramlines in Lund and<br>Utrecht. Based on his ideas on combining electron microscopy with<br>light microscopy, he started. with some of his co-workers and students,<br>the company DELMIC. Among his organizational responsibilities was<br>the presidency of the Dutch Society for Microscopy, the editorship of<br>Ultramicroscopy and the directorship of the Delft physics education. In<br>acknowledgement for his contributions he received the national<br>physics "Valorization award" and the University's yearly teaching<br>award. In 2012 he was knighted in the order of the Dutch Lion. In<br>September 2021, he started a new career as Chief Technology Officer<br>of the electron column denartment of Applied Materials |  |

# **Entanglement-Enhanced Electron Microscopy and Its Generalizations**

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Quantum electron microscopy (QEM) is emerging at the boundary between the fields of electron microscopy and quantum information science, although it is still in its infancy. One of its main objectives is to address the problem of radiation damage. In QEM, we strive to use a minimal number of electrons to extract as much information as possible from a specimen before the specimen is destroyed, for example by approaching the Heisenberg limit. Examples of expected, simulated images of a biological molecule, without averaging over many molecules, are shown in Fig. 1 [1]. Mainly two schemes of QEM, namely multi-pass TEM [2] and entanglement-enhanced (qubit-assisted) TEM [3], have been put forward. We are currently working on the latter: A cryogenic electron optical testbed for a proof-of-principle experiment, along with relevant superconducting circuits, is under development [4].

Looking ahead, we envision *universal* QEM [5], which is a generalized version of singlequbit-assisted QEM. Universal QEM may be programmed to do anything quantum mechanically possible and can, in particular, *query* the specimen in the sense relevant to the "query model" of computational complexity theory. For example, one might imagine Grover-like search for a known molecule in a crowded cellular environment in cryoelectron tomography. In the absence of nearterm "killer apps" of quantum computing, QEM could be a much-desired small-scale application of quantum computing with provable quantum advantage.



Fig. 1. Simulated images of the molecule VP35 [1]. (a) A band-pass filtered phase map. (b) An ideal conventional method. (c) Quantum-enhanced image. The horizontal length of each image is 10 nm.

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#### Acknowledgement

We thank our students that include Yuki Okuda, Shota Uchita and Takumi Higuchi. H. O. enjoyed relevant discussions with Marek Malac, Nobuo Tanaka and Fu-Rong Chen in recent years. This research was supported in part by the JSPS "Kakenhi" Grant (Grant No. 19K05285).

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# **Quantum Resonator in A Time-resolved Electron Microscope**

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Advancements in electron optical devices, such as aberration correctors and data collection technologies, have enabled transmission electron microscopes to provide images with single-atom sensitivity at sub-angstrom spatial resolution. Observations at the atomic level necessitate intense electron irradiation, which often results in changes to the nanostructure of materials. This makes radiation damage a significant bottleneck issue. The primary method currently is to use cryo-electron microscopy to reduce electron radiation damage in a low-temperature environment. However, the rapid freezing process can potentially alter the sample's morphology, and dynamic information about the reaction process becomes unobservable post-freezing. It has been established that the use of pulsed electron sources can minimize radiation damage to materials. However, even a very low electron dose rate can cause radiation damage to soft materials and those sensitive to the beam. The Quantum Zeno effect has been employed to achieve interaction-free measurement, thereby avoiding the interaction between detection particles and materials. This concept has been experimentally confirmed in optics.

The development of a quantum electron microscope, capable of achieving interaction-free measurements between detection electrons and materials, can help overcome the bottleneck of radiation damage. With the support of the Futian District of Shenzhen, the City University of Hong Kong Futian Research Institute has developed key components of a compact electron microscope equipped with a pulsed electron source. Based on this, the team is working on a quantum resonator for use with a pulsed electron source as a key component to achieve a quantum electron microscope. To enhance the quantum effect of interaction-free measurement, it is necessary to increase the number of electron cycles in the resonator. Unlike optical systems, current electron optical components often experience significant losses when the electron beam is split and re-coupled. We have designed an electron resonator based on a multipole field arrangement that can precisely split pulsed electron source can not only reduce radiation damage and provide time resonator.

To effectively enhance quantum efficiency and reduce the volume of the resonator, we have also designed other key components to match the quantum resonator, including afocal lenses for deceleration and acceleration, a Kepler multipole lens for correcting the electron beam, prism arrays for circulating the electron beam, and a fast kicker for controlling the pulsed electron beam entering and leaving the resonator. The goal is to effectively enhance quantum efficiency and experimentally verify interaction-free measurement based on the Quantum Zeno effect.

Many vital energy and optoelectronic materials are based on soft materials, and many properties of materials, especially the dynamic responses of nanomaterials under external field stimulation, still contain many unknown elements. The development of a quantum electron microscope based on a pulsed electron source is expected to address the key issue of radiation damage and become a new generation tool for studying soft materials at the atomic scale.
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| Distinction | Media/Press: CityU becomes world's first university to manufacture<br>next-generation self-designed electron microscopes (2023)<br>Meissner Fellowship and Research Assistantship, Purdue (2012 -<br>2018)<br>Government Scholarships for study abroad, Taiwan (2012)<br>Honorary Member of the Phi Tau Phi Scholastic Honor Society at<br>National Taiwan University (2009)  |  |
| Biography   | Dr. Yu-Chun Hsueh received his PhD degree in Electrical and<br>Computer Engineering from Purdue University in 2018.<br>His recent research interests focus on the development and<br>manufacturing of ultrafast and quantum technology with scanning and<br>transmission electron microscopes. He also has many years of<br>experience in laser optical design, optical measurement system, optical<br>and THz waveguide design, optical force theory and aperiodic<br>nanostructures design. |  |

#### A few "quantum" and spooky ideas and experiments in electron microscopy

<u>Vincenzo Grillo</u><sup>1</sup>, Amir Tavabi<sup>2</sup>, Enzo Rotunno<sup>1</sup> Lorenzo Viani<sup>1,3</sup>, Paolo Rosi<sup>1</sup>, Alessio Derrico<sup>4</sup>, Alberto Roncaglia<sup>5</sup>, Luca Belsito<sup>5</sup>, Gian Carlo Gazzadi<sup>1</sup>, Marco Beleggia<sup>3</sup>, Stefano Frabboni<sup>3</sup>, Ebrahim Karimi<sup>4</sup>, Giovanni Maria Vanacore<sup>6</sup>, Rafal E Dunin-Borkowski<sup>2</sup>

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The use of quantum methodologies and ideas in electron microscopy is promising and already producing a complete paradigm shift in electron microscopy. In this contribution I will discuss a few interesting ideas and experimental results for my personal approach on the topic of quantum experiments.

There is a common denominator connecting from the new idea of ghost imaging, computational ghost imaging (CGI), coherence determination and inelastic interferometry and I will try to highlight how the present and future TEM technologies can produce a new way of considering elastic and inelastic scattering. The tool for this is the beam shaping technology and I will explain present advances in both the MEMS technology to produce innovative electron optics and the Ultrafast TEM-based near field light-electron beam modulation approach.



Fig. 1 Left: density matrix models in OAM representation describing incoherence in plasmon scattering. Right: metalenses for electron–optics interaction that allow for a better resolution in controlling the e-beam



Fig. 2 Example of simulations for CGI reconstruction and experimental image of one of the caustics used to illuminate the sample. CGI can increase the microscope resolution beyond aberrations

#### Probing Dynamic Responses of Nano-Materials at the Boundary between Classical and Quantum Mechanics detecting Coherent-Inelastic Electron Selfinterferences

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The Copenhagen convention of quantum mechanics describes a boundary between the quantum world, where wave functions are indefinite in space and time, and the classical world of particles that we perceive as definite. Heisenberg's Uncertainty Principle defines this boundary, but details about its relationship to electron-sample interactions remain incomplete. Specifically, it seems unfortunate that matters concerning wave-particle duality, time-dependent measurements, or the entanglement of wave functions and their collapse at a detector still require detailed investigations to enable progress. Certainly, this is no academic matter because such aspects become the bottleneck for the deployment of microscopes with high spatiotemporal resolution that already reaches towards ~ 1 Å spatial resolution with ~1 ps time resolution and enable investigations of dynamic processes in solids (1,2). With the development of aberration-correction, the characterization of static nano-materials by electron microscopy has made tremendous progress. The point is reached where single atoms can be chemically identified and detected in 3D that reveal beam-induced, dynamic behavior (3). Further, their operation in ultra-low dose conditions guaranties that only single electrons participate in scattering events (4), which allows maintaining the pristine structure of radiation sensitive matter. Here, we explore new principles by experimentally analyzing the relation between particle and wave descriptions of electron-matter interactions measuring the delocalization of an evanescent field in energy-filtered real-space images of sample/vacuum interfaces recorded. Its spatial extension coincides with the energydependent self-coherence length of propagating wave packets that obey the time-dependent Schrödinger equation and undergo a Goos-Hänchen shift. The wave packets are created by selfinterferences during coherent-inelastic Coulomb interactions and exhibit a decoherence phase  $\Delta \phi$ = 0.5 rad. Due to a reciprocal dependence on energy, they shrink below atomic dimensions for electron energy losses beyond 1000 eV when the wave packets appear particle-like. Consequently, atomic resolution observations inevitably include pulse-like wave propagations that stimulate structural dynamics at any electron energy loss.

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Acknowledgment:

Electron microscopy at the Molecular Foundry is supported by the Office of Science, the Office of Basic Energy Sciences, the U.S. Department of Energy, Contract No. DE-AC02-05CH11231.

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| Affiliation | Dr., Lawrence Berkeley National Laboratory, USA  |  |
| Distinction | Affiliated staff scientist at LBNL (retired), Consultant   |  |
| Biography   | Affiliated staff scientist at LBNL (retired), Consultant<br>Dr. Christian Kisielowski was Principle Investigator and Staff<br>Scientist at the Molecular Foundry, Lawrence Berkeley National<br>Laboratory, CA. He was awarded his PhD in natural sciences (physics,<br>mathematics, chemistry) and his Habilitation in experimental physics at<br>the University of Cologne / Germany in 1985 and 1990, respectively, for<br>performing spectroscopic studies of defects in semiconductors.<br>Thereafter, he joint AT&T Bell Laboratories (1991 – 1994) where he<br>invented new quantitative tools for image analyses in High Resolution<br>Electron Microscopy. Since 1997, Dr. Kisielowski serves as Staff<br>Scientist at the NCEM where he developed and applied atomic resolution<br>electron microscopy, sample preparation, and computational tools. He<br>was the first to demonstrate sub-Ångstrom resolution in 1999 by mid-<br>voltage phase contrast microscopy and reached record resolution below<br>0.5 Å, which touches the physical limits of obtainable resolution and was<br>established in 2009 within the TEAM Project (Transmission Electron<br>Aberration-corrected Microscopy).<br>Recognizing that beam-sample interactions and time resolution are<br>most challenging aspects of atomic resolution electron microscopy, he<br>developed a low dose rate technique, which enables time-resolved<br>investigations of functionality on a single atom level in three dimensions<br>and environmental meaningful conditions (elevated p, T).<br>Instrumentation providing spatiotemporal resolution of 1 Å and 1 ps is<br>within reach. Such capabilities touch quantum mechanical limits and are<br>of significant interest in the context heterogeneous systems for<br>sustainable energy research that commonly contain soft and hard matter<br>components including single-digit nanoparticles, two-dimensional<br>materials, interfaces, surfaces or point and extended defects. Dr.<br>Kisielowski has published over 200 peer reviewed articles including<br>multiple publications in respected journals such as Science, Nature<br>Materials, Nano Letters, Angewandte Chemie, Phys. R |  |

## **Participated Companies**

# **ThermoFisher** S C I E N T I F I C



## **Co-Chairs**



**Professor Rafal E. Dunin-Borkowski** Professor Dr., Forschungszentrum Jülich, Germany



**Professor Fu-Rong Chen** Chair Professor, Department of Materials Science and Engineering, City University of Hong Kong, China



**Professor Wolfgang Jäger** Professor Dr., Institute for Materials Science, Christian-Albrechts-Universität zu Kiel, Germany



**Professor Xiaoyan Zhong** Associate Professor, Department of Materials Science and Engineering, City University of Hong Kong, China

### **Local Organizing Committee**

#### **City University of Hong Kong**

Prof. Fu-Rong CHEN Prof. Xiaoyan ZHONG Dr. Feng ZHU Prof. Yu-Chun Hsueh Prof. Xun-Li WANG Prof. Xiaocheng ZENG Ms. Fiona Hung Ms. Tiffany CHOY Miss Pui Kei PONG

#### HKIAS

Prof. Shuk Han Cheng Ms. Jane Li Ms. Sophie Xie Miss Prudence Lau Miss Eunice Ng Miss Sara Ho Ms. Candy Wong Ms. June Ho Mr. Tony Tsang Mr. Tsz-him Chan

City University of Hong Kong Matter Science Research Institute (Futian, Shenzhen) Miss Manyu CHEN Miss Simin CAO Mr. Sen GUAN Mr. Yan LI

## Volunteers

| Mr. Bin LIN        | Miss Yalin GAO    |  |
|--------------------|-------------------|--|
| Dr. Qi WANG        | Mr. Qi LIU        |  |
| Mr. Zhihao HUANG   | Dr. Goshu TAMURA  |  |
| Dr. Feng ZHU       | Dr. Xiaocui LI    |  |
| Dr. Min DU         | Mr. Yuxuan ZHANG  |  |
| Miss Jie REN       | Mr. Ji ZHOU       |  |
| Mr. Qingxiang WANG | Mr. Xiaojun ZHANG |  |
| Miss Zhuo LI       | Miss Miao ZHANG   |  |
| Mr. Xiaofeng GUO   | Mr. Guoliang JIN  |  |
| Mr. Tianyi ZHOU    | Mr. Zhaohua WANG  |  |
| Mr. Yanwei ZHAO    | Miss Yini ZHENG   |  |
| Mr. Yuchi CHEN     | Miss Jing REN     |  |
| Miss Jinyuan DENG  | Miss Manyu CHEN   |  |
| Miss Simin CAO     | Dr. Yan CHEN      |  |
| Mr. Lingqi KONG    | Mr. Shubin HUANG  |  |
| Mr. Hanbo GENG     | Mr. Li YONG       |  |
| Ms. Liying WANG    |                   |  |