Abstract Booklet

Symposium

Advanced Application of TEM in Materials Sciences

MPI Halle, January 31, 2019



Aim of the symposium

The symposium will bring together experts from different fields of transmission electron microscopy to discuss the application of advanced techniques in materials sciences and solid state physics.

The symposium will focus on:

- In-situ TEM of surface reactions
- Holography
- Ultra-fast electron microscopy
- Quantitative TEM
- In-situ Lorentz TEM

At the end of the afternoon sessions, a comprehensive discussion of the presented subjects is planned. Another occasion of this symposium concerns the farewell of our co-worker/colleague Dr. Peter Werner, who was working in the field of TEM here at the Institute - with several intermediate stations - for nearly 40 years.

We look forward to an exciting symposium at the Max Planck Institute in Halle Best regards,

Stuart Parkin

Off-axis electron holography: Basics, applications, and perspectives

Michael Lehmann

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In transmission electron microscopy (TEM), the phase of the electron wave is the most important information carrier of the specimen under investigation. This phase is modulated due to electric potentials from the mesoscopic scale (e.g. p-n junctions) down to the atomic scale as well as due to magnetic fields in form of domains or stray fields. In order to record the whole phase information, off-axis electron holography is utilized as an advanced TEM-method, which is most sensitive on tiny phase differences between the object-modulated electron wave and the unmodulated reference wave. In the meanwhile, electron holography has found a broader TEM community investigating e.g. p-n junctions, electrostatic polarization fields, charge distribution of grain boundaries and interfaces as well as domains in magnetic materials, correlation of stray fields of adjacent magnetic particles, and skyrmions. In-situ experiments by application of external electric potentials or magnetic fields mark the beginning of investigations of simple structures up to whole devices in operando. Furthermore, tomography and holography are combined for the three-dimensional characterization of magnetic fields and electrostatic potentials. Most recently, time-resolved electron holography using interference gating is currently opening the window into investigations of periodically changing magnetic fields and electric potentials with periods of less than 1 ns.



Michael Lehmann was born 1965 in Ulm

1986 – 1992: Study of Physics (diploma) at the Eberhard Karls Universität in Tübingen.

1997: Doctor in Physics at the Eberhard Karls Universität in Tübingen.

2004: Habilitation in Experimental Physics at the TU Dresden. Mentor: Prof. H. Lichte.

Since 2006: Full Professor (W3) for Experimental Physics/Electron- and Ion-Nanooptics at TU Berlin.

Since 2006: Scientific Head of the Center for Electron Microscopy (ZELMI) at TU Berlin.

2013: Otto Mønstedt Guest Professor at Denmark Technical University in Copenhagen.

2016 – 2017: Elected President of the German Society for Electron Microscopy (DGE e.V.).

2019: Host of the Microscopy Conference MC2019 (together with Prof. Ch. Koch/HU Berlin).

New methods in quantitative TEM

Wouter Van den Broek

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In ptychography, the phase of the object's exit wave is retrieved from a set of diffraction patterns recorded from overlapping beam positions.

If the beam is in-focus and condensed, good sampling in reciprocal space is achieved, but at the cost of the need to record many patterns, while with an out-of-focus condensed beam less recordings are needed, but the sampling in reciprocal space is worse. A diffuser is presented that combines both advantages by producing a wide and in-focus probe when inserted in the condenser plane. An experimental realization of the diffuser is shown, along with preliminary ptychographic reconstructions achieving super-resolution.

Compressed sensing (CS) exploits the signal's sparsity during the recording process so that it can be retrieved from surprisingly few measurements. CS is therefore often considered for dose (and associated beam damage) reduction in ADF-STEM. However, it is not a given that a reduction in number of measurements implies a reduction in dose. It is shown that the amount of Fisher information in CS measurements is equal to that in a conventional ADF-STEM image recorded with the same total dose, and that a CS reconstruction has the same mean squared error as an ADF-STEM image of equal dose and denoised with the same assumptions underpinning the CS reconstruction. Thus possible advantages of CS for beam damage must lie elsewhere, for instance with the rate or spatial distribution of the dose.



Wouter Van den Broek received his PhD in Science from the University of Antwerp in 2007. From 2007 until 2012 he was postdoc at the electron microscopy group (EMAT) of this university, and from 2012 until 2015 at the department of physics at Ulm University. Since 2016 he is a permanent staff scientist at the department of physics of the Humboldt University in Berlin.

His research interest lies in the development and application of numerical techniques in (scanning) transmission electron microscopy, with a focus on incorporating the physical processes and other prior knowledge. Applications are two- and threedimensional atomic resolution reconstructions from amplitude and phase contrast, compressed sensing, and ptychography.

Next-Generation Ultrafast Transmission Electron Microscopy (UTEM) – Femtosecond resolution with a high coherence electron beam

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Electron microscopy is tremendously successful in unravelling material structures and compositions on the atomic scale, with a temporal resolution governed by detector response times. Utilizing a stroboscopic approach, ultrafast electron diffraction [1] and microscopy [2,3] techniques provide for a unique access to processes on ultrashort time scales. In ultrafast transmission electron microscopy (UTEM), combining nanoscale spatial resolution of electron microscopy with femtosecond temporal resolution of optical spectroscopy, a pulsed electron beam of sub-picosecond duration is able to probe rapid processes [2].

In this talk, I will introduce the UTEM methodology and discuss how the recent availability of coherent electron pulses enables us to combine it with the advanced capabilities of state-of-the-art TEMs.

The Göttingen UTEM instrument is based on a JEOL 2100F Schottky field emission TEM, which we modified to allow for optical sample excitation and the generation of highly coherent ultrashort electron pulses [4,5]. Single-photon photoemission from nanoscale tip-shaped photocathodes yields electron focal spot sizes down to 0.8 nm, an electron pulse width of 200 fs (full-width-at-half-maximum) and a spectral bandwidth of 0.6 eV [5].

I will give an overview of current experiments in ultrafast phase-contrast imaging and local probing, which include: i) femtosecond Lorentz microscopy [6], ii) phase-resolved RF-excited magnetic vortex dynamics, iii) the ultrafast imaging of structural phase transitions and vi) the local diffractive probing of GHz-THz strain dynamics [7].

In a further line of applications, the interaction of fast electrons with intense optical near-fields [4,8] enables the quantum coherent control of free electron pulses, tailoring the longitudinal and transverse electron beam properties by light. As a particular example, I will describe the optically-induced preparation of attosecond electron pulse trains, potentially pushing the limits of the temporal resolution in UTEM into the sub-optical-cycle regime [9].

References

- [1] R. J. D. Miller, Science 343, 1108 (2014).
- [2] A. H. Zewail, Science 328, 187 (2010).
- [3] J. S. Kim et al., Science 321, 1472 (2008).
- [4] A. Feist et al., Nature 521, 200 (2015).
- [5] A. Feist et al., Ultramicroscopy 176, 63 (2017).
- [6] N. Rubiano da Silva et al., Phys. Rev. X 8, 031052 (2018).
- [7] A. Feist et al., Struct. Dyn. 5, 14302 (2018).
- [8] B. Barwick et al., Nature 462, 902 (2009).
- [9] K. E. Priebe et al., Nat. Photonics. 11, 793 (2017).



Armin Feist is a postdoctoral researcher at the University of Göttingen in the group of Claus Ropers, where his Ph.D. work focused on the development and applications of Ultrafast TEM using coherent electron pulses. He studied in Leipzig, Leeds and Göttingen, where he worked in his B.Sc. and M.Sc. theses on angle-resolved fluorescence in photonic crystals and laser-triggered field ion microscopy, respectively. His current research interests include nanoscale structural dynamics, optically tailored free-electron beams and the exploration of new instrumental capabilities in UTEM, with the vision of combining methods from ultrafast science with state-of-the-art electron microscopy. For his Ph.D. work, he was awarded the '2015 EMS Outstanding Paper Award - Instrumentation and Technique Development' (European Microscopy Society) the 'Heinz-Bethge-Nachwuchspreis' (Heinz-Bethge-Foundation) and the 'Jan Peter Toennies Physik-Preis' (Faculty of Physics, Göttingen).

Dark-Field electron holography reveals the impact processing steps onto the strain distributions in FDSOI CMOS planar devices

Victor Boureau¹, ², Daniel Benoit³ and <u>Alain Claverie¹</u>

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Clever strategies have been set up and are today routinely used to generate strains of desired directions and amplitudes in the channels of CMOS transistors. Unfortunately, many of the processing steps which follow this strain engineering have a strong impact on the final strain state of these channels, sometimes even not foreseen. Here, we report on the use of Dark-Field Electron Holography to image strain in the channels of FD-SOI CMOS devices all along their fabrication route, from the fabrication of the co-integrated Si-SiGe layers to contact formation. We show that, in general, good background knowledge of the different materials characteristics and of the physics of the process, as well notions of structural mechanics, are enough to understand then eventually model the impact of these processing steps. However, our results evidence a particular mechanical weakness of the SiGe/BOX interface following the Ge condensation process which seriously challenges strain manipulation in the pMOS channel.



Materials Scientist Research Director at CNRS Former CEMES Director (2011-2015) Alain obtained the Dipl.-Ing. degree in Solid State Physics in 1981 from the National Institute for Applied Science (INSA) of Toulouse then his PhD in 1984 from the University Paul Sabatier of Toulouse. From 1985,

he was Staff Scientist (permanent position) in the semiconductor group at CEMES, an autonomous laboratory of the National Center for Scientific Research, where he performed experimental work on TEM characterization of ion implanted materials. From 1988 to 1993, he has been visiting or staff scientist in national laboratories abroad (about 3 years), in India (Chandigarh) and California (Berkeley), notably working on MBE grown semiconductors. For ten years, he has been leading a group of 10 in the field of ion implantation and dopant diffusion in semiconductors. From 2000 to 2004, he was the coordinator of the EC supported NEON (nanoparticles for electronics) a GROWTH Project aimed at engineering nanocrystals for memory applications. Appointed as "Directeur de Recherches" in 2002, he founded and led the "nanoMaterials Group" at CEMES where about 30 permanent scientists and about the same number of PhDs and PostDocs worked on the synthesis, the physics, the characterization and the integration of nano-crystals and ultrathin films in systems for applications in electronics, magnetism and optics. From January 2011 to December 2015, Alain was the Director of CEMES, a leading laboratory of 170 people owned by the Centre National de la Recherche Scientfique (CNRS) in the field of nanosciences and nanomaterials (www.cemes.fr).

Alain's interest ranges from the nucleation and growth of extended defects and nanoprecipitates in solids, diffusion anomalies in semiconductors, strain transfer in nanostructures and very low energy ion implantation. He is the author or co-author of more than 300 publications in international journals (h factor=40), the editor of the book "Transmission electron microscopy in micro/nanoelectronics (Ed. ISTE Wiley London) and gave more than 70 invited talks in international conferences. He has been the organizer of several international conferences (MRS (3), E-MRS (2), IEEE, GADEST...) and is a member of the scientific/technical boards of several others. In 2013, he was the general chairman of the Spring E-MRS Conference which hosted 25 Symposia and where more than 3000 attendees participated. Today, Alain is engaged in a number of projects in collaboration with semiconductors companies (STMicroelectronics, Soitec...) and also works as a private scientific consultant.

The past and future of TEM at Weinberg Campus

Ralf Boris Wehrspohn

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Transmission electron microscopy has an almost 60 years lasting tradition at Weinberg Campus in Halle.

Starting in 1960, enthusiasts linked with the Institute for Solid State Physics and Electron Microscopy, the International Center for Electron Microscopy, the Max Planck Institute of Microstructure Physics, the Fraunhofer Institut, the Heinz Bethge Foundation, as well as a number of Halle University groups, all located at Weinberg, have made significant contributions to the advancement of electron microscopy and spectroscopy.

Covering sample preparation, various modes of imaging and analytics, modelling and the establishment of microstructure-properties relationships, a vivid community is nowadays covering topics from materials to life sciences.

This review takes a look down the memory road but also sketches dawning future developments.



MAIN RESEARCH FIELDS

Nanostructured materials and components as used in microelectronics, sensor technology, photonics and photovoltaics

EDUCATION & ACADEMIC POSITIONS

since 06/2006: W3 professor for experimental physics at the Martin-Luther-University of Halle-Wittenberg, chair of Microstructure-based Materials Design, held concurrently with the office of the institute director of the Fraunhofer Institute for Mechanics of Materials, Halle

04/2003–05/2006: C4 professor for experimental physics at the University of Paderborn, chair of Nanophotonic Materials

10/1999–03/2003: Leader of a junior research group at the Max Planck Institute of Microstructure Physics in Halle, department of Prof. Gösele in the field of the application of porous materials and assistant professor at the Martin-Luther-University of Halle-Wittenberg

09/2003: Post-doctoral thesis at the Martin-Luther-University of Halle-Wittenberg (Prof. Gösele); Geordnete poröse Nanostrukturen – ein Modellsystem für die Photonik (Ordered porous nanostructures – a model system for photonics)

09/1995–06/1997: European PhD thesis, written at the University of Oldenburg (Prof. G.H. Bauer) and at the École Polytechnique, Palaiseau, France. Porous amorphous silicon – pore formation and photoluminscent properties;

10/1990–08/1995: Studies of physics at the University of Oldenburg

Multi-scale observation of catalyst dynamics under reactive conditions

Marc-Georg Willinger

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Electron microscopy plays an important role in the characterization of catalysts and their precursors.

Atomically resolved images of catalyst particles serve as reference for theoretical modelling and have influenced the way in which we depict active sites. However, since high-resolution imaging and local compositional analysis is generally performed under vacuum and close to room temperature, the obtained atomistic details concern an equilibrium state that is of limited relevance if the active state of a catalyst is in the focus of the investigation. Indeed, heterogeneous catalytic reactions are highly non-linear chemical processes that are operated far from thermodynamic equilibrium. In situ electron microscopy has clearly demonstrated that the interaction with the gas phase induces changes of shape, composition and chemical dynamics.

During the last years, the availability of MEMS-based TEM holders for in situ experiments with controlled heating & biasing under liquid- and gas- environment has strongly enhanced our abilities to study materials under the influence of a physical or chemical stimulus.

Using a combination of in situ TEM and in situ SEM, we have studied the dynamics of active catalysts under simple redox conditions as well as under industrially relevant reactions, such as methanol oxidation. By bridging the scale from the Å to the mm range and pressures from 10⁻⁵ to 10⁺³ Pa, we are able to reveal the dynamic nature of active catalysts and to bridge the materials and pressure gap between simplified model systems and real-world catalysis. It will be shown that simultaneous detection of reaction products by RGA enables correlating structural dynamics with catalytic activity. We observe rate oscillations and oscillatory behaviour that is inherent to the action of a catalyst, which has to break bonds and facilitate the formation of new ones over and over again.



Marc Willinger studied physics at the Technical University in Vienna, Austria and did his master in the field of electron energy loss spectroscopy and DFT simulation of the electronic structure under the supervision of Prof. Peter Schattschneider and Prof. Robert Schlögl, Director of the Department of Inorganic Chemistry at the Fritz-Haber-Institute (FHI) of the Max Planck Society in Berlin.

Marc Willinger obtained his PhD from the Technical University in Berlin for the investigation of the electronic structure of vanadium phosphorous oxides. After a 1.5 years post-doc at the Fritz-Haber-Institute, he moved to the University of Aveiro in Portugal, where he worked as an independent researcher for 4 years. In 2011 he went back to the Fritz-Haber-Institute as group leader for electron microscopy. Since 01.02.2018 he has been at the "Eidgenössische Technische Hochschule" (ETH) in Zürich, where he is focusing on the development and implementation of in-situ electron microscopy techniques.

In situ investigations of non-collinear spin textures by Lorentz transmission electron microscopy

Rana Saha

Max Planck Institute of Microstructure Physics, Halle, Germany | <u>rana.saha@mpi-halle.mpg.de</u>

One of the major topics in spintronics today is the study of the steady state and dynamical properties of non-collinear spin textures with various topologies. One of the most important of these are skyrmions that are magnetic nano-objects with chiral magnetic boundaries. Most recently, anti-skyrmions were discovered in tetragonal inverse Heusler material experimentally, using in situ Lorentz transmission electron microscopy (LTEM). Anti-skyrmions that have more complex boundaries than skyrmions, are of great current interest both for their topological characteristics and potential spintronic applications. Therefore, it is of significant importance to understand the physical properties of these nanoscopic spin textures. In this talk I will describe some recent in situ investigations on the nucleation, stability and manipulation of anti-skyrmions using LTEM. It is a powerful technique for performing real-space in situ imaging of such nanoscopic magnets under external magnetic fields at different temperatures. LTEM can follow phase transitions from helical magnetic phases to the anti-skyrmion phases over a wide range of temperature and magnetic field in situ. LTEM study shows that anti-skyrmions are stable for a larger window of magnetic fields and temperature as well as the size of these spin textures are tunable, which is important for practical applications. This finding is an important step towards understanding such complex spin textures for chiraltronic applications.



Rana Saha is a currently a post-doctoral researcher in the NISE department of Max Planck Institute of Microstructure Physics in Halle (Saale), Germany.

He obtained his PhD in Materials Science from JNCASR in Bangalore, India, where he carried out his PhD research on the magneto-electric properties of strongly correlated transition metal oxides. He then moved to MPI-Halle, where he has been pursuing his work post-doctoral on in situ nucleation, manipulation and detection of non-collinear topological spin textures such as magnetic antiskyrmions using Lorentz TEM, for applications in spintronic memory and logic devices. His research includes interest magnetic microscopy of nanoscopic chiral spin textures as well as their in situ and ex situ magneto-transport properties to understand the structure-property correlations.

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