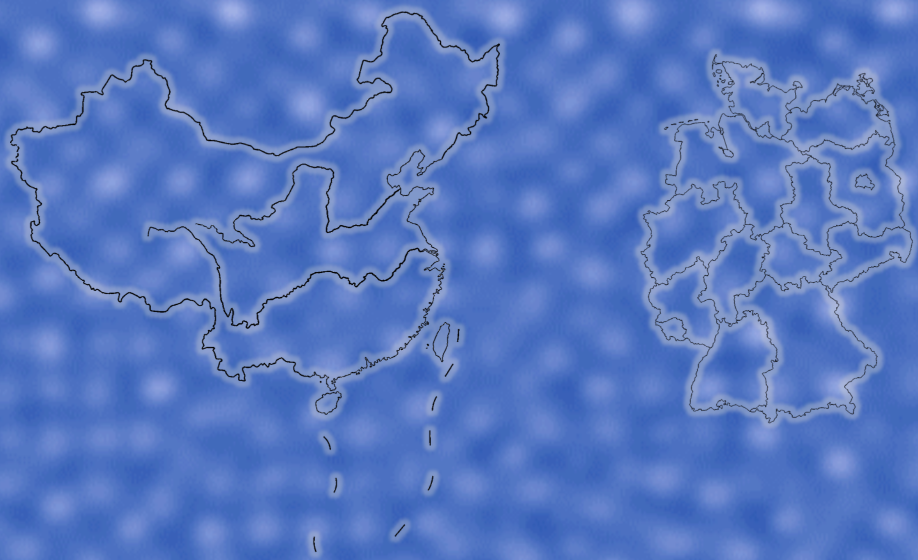

2nd Sino-German Symposium

on Advanced Electron Microscopy and Spectroscopy in Materials Science

Xi'an Jiaotong University, Xi'an, China

October 12 – 16, 2017

Program and Abstract

西安交通大學
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This symposium aims to provide a forum for researchers who are interested in advanced methods of electron microscopy and spectroscopy, including aberration-corrected electron microscopy, *in-situ* characterization of materials and applications to materials science. As these methods are of fundamental importance in virtually all technological fields, contributions are invited that address a broad spectrum of research, ranging from nanostructures to functional materials and from soft matter to bioscience. Novel methodological developments will be discussed alongside topical areas of research on thin films, bulk materials, surfaces, materials at the nanoscale and at the interface between the physical and life sciences, for understanding structure-property relationships of materials, as well as for metrology.

Symposium Organizers

西安交通大學 XI'AN JIAOTONG UNIVERSITY

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Chairs:

Prof. Rafal Dunin-Borkowski, Forschungszentrum Jülich, Germany

Prof. Wolfgang Jäger, Kiel University, Germany

Prof. Chunlin Jia, Xi'an Jiaotong University, China

Prof. Zhiwei Shan, Xi'an Jiaotong University, China

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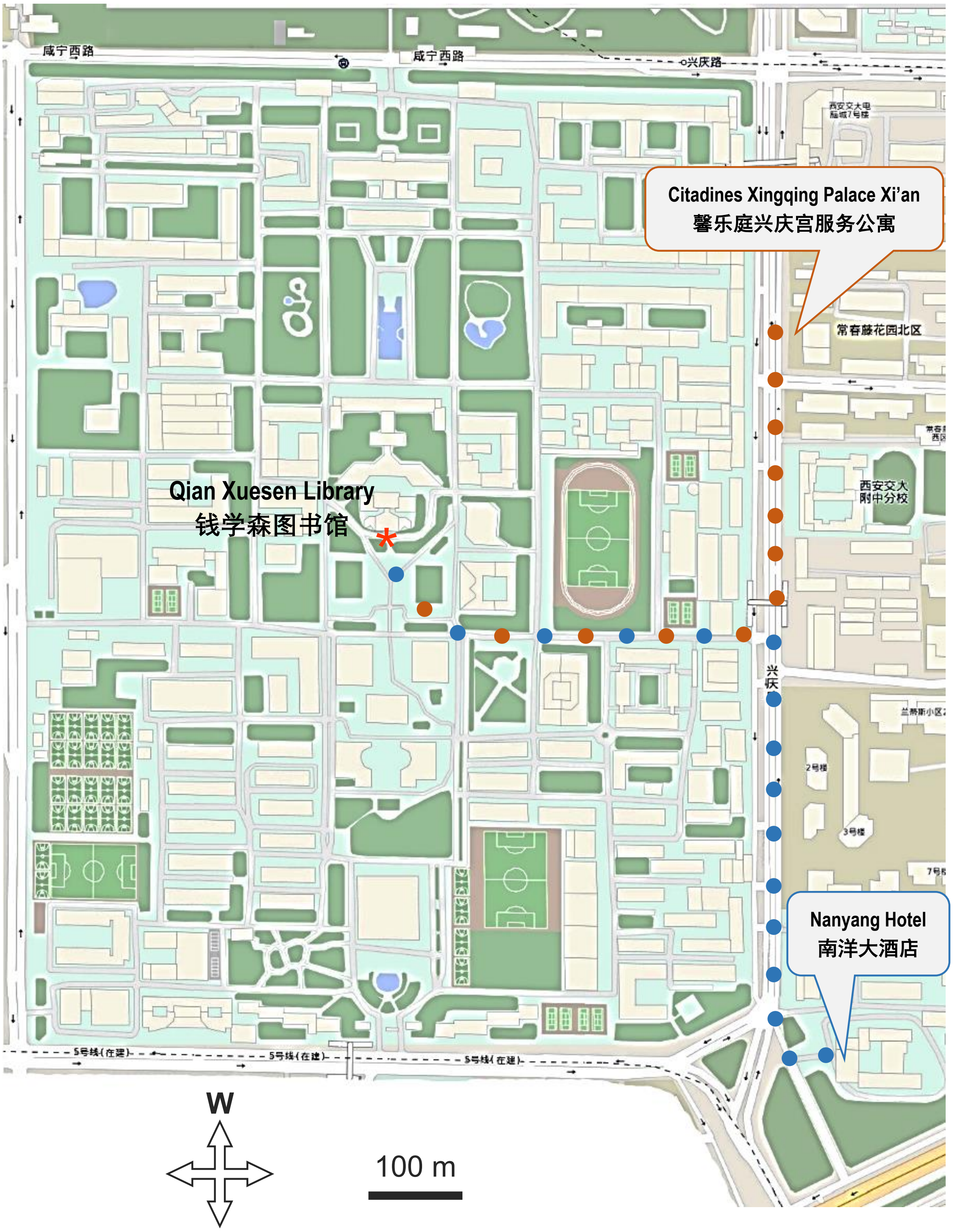
Symposium Venue:
Qian Xuesen Library in the campus of XJTU



Programme

Oct. 12, 2017 Thursday afternoon
14:00 - 19:00 Registration, Nanyang Hotel

Oct. 13 - 15, 2017, Qian Xuesen Library

Oct. 13, 2017 Friday morning			
8:15-8:30 Opening			
Session 1, Novel Instruments, Methods and Applications 1			**Chair: Prof. Chunlin Jia**
1	8:30 - 9:00	**Prof. Harald Rose** *Ulm University*	Holographic imaging and optical sectioning in the aberration-corrected STEM
2	9:00 - 9:30	**Prof. Max Haider** *CEOS GmbH*	Instrumentation for high-resolution TEM and STEM: Opportunities and limitations
3	9:30 - 10:00	**Prof. Jing Zhu** *Tsinghua University*	Application of advanced electron microscopy in material science
10:00 - 10:30 Coffee Break			

Session 2, Novel Instruments, Methods and Applications 2			Chair: Prof. Max Haider
4	10:30 - 11:00	**Prof. Dagmar Gerthsen** *Karlsruhe Institute of Technology*	On the benefits of STEM in a scanning electron microscope: basics, perspectives and applications in materials science and biology
5	11:00 - 11:30	**Prof. Peng Wang** *Nanjing University*	Electron ptychography: from 2D to 3D
6	11:30 - 12:00	**Mr. Penghan Lu** *Forschungszentrum Jülich*	Electron wavefront engineering: from spherical aberration correction to tunable orbital angular momenta
7	12:00 - 12:30	**Dr. Erwan Sourty** *Thermo Fisher Scientific*	Ultimate resolution available not only to microscopits but to any materials scientist on any material with the reproducibility and flexibility of the Themis Z platform
12:30 - 14:30 Lunch			

Oct. 13, 2017 Friday afternoon

Session 3, Advances in HRTEM and STEM			Chair: Prof. Ze Zhang
1	14:30 - 15:00	**Prof. Andreas Rosenauer** *University of Bremen*	ISTEM: Conventional TEM imaging beyond the diffraction and information limits
2	15:00 - 15:30	**Prof. Helmut Kohl** *Westfälische Wilhelms-Universität Münster*	Simulation of atomically resolved elemental maps with a multislice method for relativistic electrons
3	15:30 - 16:00	**Prof. Knut Urban** *Forschungszentrum Jülich*	Studying atoms in their cages - displacive disorder in bismuth zinc niobate pyrochlore

16:00 - 16:30 Coffee Break			
Session 4, Advances in Spectroscopy			**Chair: Prof. Bernd Rellinghuas**
4	16:30 - 17:00	**Dr. Wilfried Sigle** *Max-Planck-Institut für Festkörperforschung*	Electron spectroscopy: Recent advances and applications
5	17:00 - 17:30	**Prof. Lin Gu** *Institute of Physics, CAS*	Probing structure and electronic structure of functional oxides at atomic scale by STEM
6	17:30 - 18:00	**Prof. Xiaoyan Zhong** *Tsinghua University*	Towards atomic-scale electron magnetic circular dichroism
7	18:00 - 18:30	**Dr. Hongchu Du** *Forschungszentrum Jülich*	Exploring defects and nanostructures by STEM and spectrum imaging
19:00 - 20:00 Dinner			

Oct. 14, 2017 Saturday morning			
Session 5, Imaging of Fields			**Chair: Prof. Xiaoyan Zhong**
1	8:30 - 9:00	**Prof. Josef Zweck** *Universität Regensburg*	Differential phase contrast - imaging of fields in matter on a mesoscopic and atomic scale
2	9:00 - 9:30	**Dr. Zi-An Li** *Institute of Physics, CAS*	Quantitative off-axis electron holography for magnetic skyrmions of nanostructures
3	9:30 - 10:00	**Prof. Bernd Rellinghaus** *IFW Dresden*	Magnetic characterization of nanoscopic materials in a TEM – Prospects and limitations

10:00 - 10:30 Coffee Break			
Session 6, In Situ TEM 1		**Chair: Prof. Wolfgang Jäger**	
4	10:30 - 11:00	**Prof. Ze Zhang** *Zhejiang University*	In situ high resolution TEM study on surface activity of some metallic nano-catalysts under chemical environment
5	11:00 - 11:30	**Prof. Kui Du** *Institute of Metal Research, CAS*	In situ electron microscopy investigation on plastic deformation in a metastable beta titanium alloy
6	11:30 - 12:00	**Prof. Xiaodong Han** *Beijing University of Technology*	In situ understanding the atomistic mechanisms of grain boundary plasticity in nanocrystalline FCC Pt
7	12:00 - 12:30	**Dr. Xiaofeng Zhang** *Hitachi High Technologies*	Hitachi state-of-the-art In situ environmental TEM technologies
12:30 - 14:30 Lunch			

Oct. 14, 2017 Saturday afternoon			
Session 7, In Situ TEM 2		**Chair: Prof. Xiaodong Han**	
1	14:30 - 15:00	**Prof. Christian Kübel** *Karlsruhe Institute of Technology*	Imaging the structural evolution of nanocrystalline metals during thermal annealing and mechanical deformation
2	15:00 - 15:30	**Prof. Richeng Yu** *Institute of Physics, CAS*	Tuning the magnetism of epitaxial cobalt oxide thin films by electron beam irradiation
3	15:30 - 16:00	**Prof. Litao Sun** *Southeast University*	In situ TEM study of sub-10-nm materials

16:00 - 16:30 Coffee Break			
Session 8, Oxides and Ferroics			**Chair: Prof. Knut Urban**
4	16:30 - 17:00	**Prof. Shaobo Mi** *Xi'an Jiaotong University*	Atomic-scale structure of heterointerface and planar defects in perovskite-type oxides
5	17:00 - 17:30	**Prof. Rong Yu** *Tsinghua University*	Perfect interfaces between corundum and rutile structures
6	17:30 - 18:00	**Prof. Xiuliang Ma** *Institute of Metal Research, CAS*	Hetero-interface and its performance to materials
7	18:00 - 18:30	**Prof. Yimei Zhu** *Brookhaven National Laboratory*	Topological vortices and charge-lattice interactions in multiferroic oxides
19:00 - 20:00 Dinner (sponsored by ThermoFisher Scientific)			

Oct. 15, 2017 Sunday morning			
Session 9, Nanomaterials and Defects 1			**Chair: Prof. Xiuliang Ma**
1	8:30 - 9:00	**Prof. Thomas Klassen** *Helmholtz-Zentrum Geesthacht*	Materials for hydrogen technology: Microscopic processes and mechanisms
2	9:00 - 9:30	**Dr. Martin Ritter** *Technische Universität Hamburg*	Preparation and analysis of nanoporous gold
3	9:30 - 10:00	**Prof. Wolfgang Jäger** *Christian-Albrechts-Univ ersity Kiel*	Atomic resolution TEM characterization of GaSb/GaInAs and GaSb/GaInP bond interfaces for high-efficiency solar cells

10:00 - 10:30 Coffee Break			
Session 10, Nanomaterials and Defects 2			**Chair: Prof. Gerhard Dehm**
4	10:30 - 11:00	**Prof. Xiaoqing Pan** *Nanjing University*	Understanding novel domain science in multiferroic materials by advanced electron microscopy
5	11:00 - 11:30	**Prof. Jianbo Wang** *Wuhan University*	Microstructures and dynamic evolutions in low-dimensional materials
6	11:30 - 12:00	**Prof. Jianfeng Nie** *Chongqing University*	Atomic-resolution HAADF-STEM and EDS-STEM characterization of metallic alloys
7	12:00 - 12:30	**Dr. Qiang Xu** *DENSsolutions*	Real time atomic scale imaging of catalysts during catalytic reaction
12:30 - 14:30 Lunch			

Oct. 15, 2017 Sunday afternoon			
Session 11, Metals and Alloys 1			**Chair: Prof. Jianfeng Nie**
1	14:30 - 15:00	**Prof. Gerhard Dehm** *Max-Planck-Institut für Eisenforschung*	Ag induced phase transformation of a ∑5 grain boundary in Cu
2	15:00 - 15:30	**Prof. Jianghua Chen** *Hunan University*	Atomic-scale structure evolution of growing precipitates in high-performance aluminum alloys

3	15:30 - 16:00	**Prof. Florian Pyczak** *Helmholtz-Zentrum Geesthacht*	Morphology and stability of orthorhombic and hexagonal phases in a lamellar γ-Ti-42Al-8.5Nb alloy - a transmission electron microscopy study
16:00 - 16:30 Coffee Break			
Session 12, Metals and Alloys 2			**Chair: Prof. Xiaoxu Huang**
4	16:30 - 17:00	**Prof. Christina Scheu** *Max-Planck-Institut für Eisenforschung GmbH*	Correlative Cs corrected STEM - atom probe tomography on Kappa-carbides in steel
5	17:00 - 17:30	**Prof. Xiaoxu Huang** *Chongqing University*	3D characterization of dislocation structures and solute distributions in light alloys
6	17:30 - 18:00	**Prof. Zhiwei Shan** *Xi'an Jiaotong University*	Temperature effect on hydrogen behavior in submicron-sized Al pillars
7	18:00 - 18:30	**Dr. Stavros Nicolopoulos** *NanoMEGAS SPRL*	New materials characterization with TEM orientation imaging/phase mapping in combination with e-PDF in situ analysis at nm scale
8	18:30 - 18:45	**End of the symposium - closing remarks**	
19:00 - 20:00 Dinner			

Oct. 16, 2017 Monday
09:00 - 16:00 Excursion and laboratory visits

Abstract

Holographic imaging and optical sectioning in the aberration-corrected STEM

Harald Rose

Ulm University, Center for Electron Microscopy, Albert-Einstein-Allee 11, 89069 Ulm, Germany

Scanning transmission electron microscopy (STEM) usually employs the high-angle annular dark-field (HAADF) mode for visualizing the atomic structure of objects. However, HAADF images show primarily heavy atoms because the contrast is approximately proportional to Z^2. Aberration correction offers the possibility to visualize low-Z atoms in the STEM by employing differential phase contrast (DPC) methods. Two representative procedures are the annular differential phase contrast (ADPC) mode and the integrated differential phase contrast (IDPC) mode. Both modes enable the use of the elastically scattered electrons located within the cone of the non-scattered electrons beneath the object. The annular ADPC has recently been realized by the correction of the third-order spherical aberration (C_3), by inserting a physical Fresnel phase plate in front of the objective lens and by using a detector geometry which matches that of the Fresnel phase plate. The additional correction of chromatic (C_c) and the fifth-order spherical aberration (C_5) improves significantly the performance of this method. The Fresnel phase plate for the DPC can be obtained more flexibly by adjusting the spherical aberration coefficients appropriately. On the condition that Johnson noise is eliminated or sufficiently suppressed, C_c/C_5-correction enables the optical sectioning by the ADPC mode in STEM with atomic resolution, as well as a depth of field shorter than 3Å even at a low accelerating voltage of 30kV. This mode allows the investigation of the inner structure of crystalline samples without slicing. Results of image simulations will be presented which demonstrate that the ADPC mode is also appropriate for weak and strong phase objects such as thin crystals.

Instrumentation for high-resolution electron microscopy: opportunities and limitations

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The development of new components for high-resolution Electron Microscopy has pushed the achievable resolution limit to new not anticipated areas over the last two decades. The emergence of correctors to compensate the unavoidable spherical aberration has improved the attainable information limit for TEM [1] as well as for STEM [2]. However, not only the spherical aberration limits the easily attainable resolution but also the chromatic aberration Cc. Hence, besides the corrector also monochromators have been developed with which the attainable information limit in TEM could be improved due to the reduced energy spread of the electron source. The other possibility to reduce the focus spread caused by Cc is the compensation of Cc by means of a Cc-corrector. However, after the development of a Cc-corrector the achieved resolution was not as high as anticipated. Detailed investigations of the source of this limited resolution at higher energies turned out that free electrons in any conductive piece next to the electron beam are causing a so-called Johnson noise [3].

Besides the limitation of the attainable resolution by any sort of noise or by higher order or residual aberrations there are also opportunities to improve the resolution by dedicated aberration correctors, eventually in combination with a monochromator. The disturbing Johnson noise can be reduced at lower energies by an optimized Cc-corrector and ray path. As a consequence, new resolution limits could be achieved with Cc-correction for the SALVE project [4].

Due to these developments of correctors and monochromators there are new opportunities of high resolution electron microscopy for various applications.

U_A/kV	λ / pm	50 mrad limit		SALVE Achievements	SALVE with resp. λ
20	8.589	5.82 /nm	171.8 pm	128 pm / 139 pm	14.9 / 16.2
30	6.979	7.16 /nm	139.6 pm	108 pm / 115 pm	15.5 / 16.5
40	6.016	8.31 /nm	120.3 pm	88.5 pm / 90 pm	14.7 / 15.0
60	4.866	10.28 /nm	97.3 pm	80 pm / 83 pm	16.4 / 17.1
80	4.176	11.97 /nm	83.5 pm	69 pm / 76 pm	16.5 / 18.2

TAB. 1. The measured information limit of the SALVE instrument at energies between 20 – 80 keV

*after compensation of Cs and Cc. The best result with respect to the used energy was obtained at 40 keV with an information limit to be around 15 * λ. The 2 right columns are divided in two values showing the best and the weakest one.*

References

[1] M. Haider et al., Nature **392**, 768 (1998)

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[3] S. Uhlemann et al, PRL **111**, 046101 (2013)

[4] M. Linck et al, PRL **117**, 076101 (2016)

Application of advanced electron microscopy in material science

Jing Zhu

National Center for Electron Microscopy in Beijing
School of Materials Science and Engineering
Tsinghua University, Beijing, China

Three examples about the application of advanced electron microscopy in material science will be given in this talk:

1. Nano-mechanics and surface relaxation of single nanowire
   In-situ electron microscopy ⟶ Aberrative-corrected TEM
   Diameter dependence of modulus and core-shell model in ZnO nanowire
2. Application of EELS an EMCD
3. Edge dislocation and topological vortices domain in single phase multiferroic hexagonal manganites single crystal

Acknowledgements:

This work was financially supported by Chinese National Natural Science Foundation (11374174，51390471, 51527803)，National 973 Project of China (2015CB654902), and National key research and development program (2016YFB0700402). This work made use of the resources of the National Center for Electron Microscopy in Beijing, Forschungszentrum Jülich, Germany and Tsinghua National Laboratory for Information Science and Technology.

On the benefits of STEM in a scanning electron microscope: basics, perspectives and applications in materials science and biology

Dagmar Gerthsen

Laboratory for Electron Microscopy, Karlsruhe Institute of Technology, Germany

High-resolution (scanning) transmission electron microscopy ((S)TEM) at low electron energies has been tremendously brought forward during the past years to enhance the contrast of weakly scattering objects and to suppress knock-on damage in beam-sensitive materials. These efforts were up to now focused on transmission electron microscopes. However, STEM imaging can be also performed in scanning electron microscopes at electron energies of 30 keV and below if they are equipped with a STEM detector. The resolution for thin TEM specimens can approach the minimum electron-beam diameter, about 1 nm in state-of-the-art scanning electron microscopes, which is sufficient for solving many questions in materials science and biology. In addition, surface topography and material contrast information from a variety of different SEM detectors is inherently available in a scanning electron microscope and provides relevant additional data on the sample properties. Low-energy STEM and backscattered-electron intensities can be well modelled by Monte-Carlo simulations and facilitates, e.g., the determination of the composition and local TEM specimen thickness if some pre-information is available. Most recent technological advances in scanning electron microscopes allow bright-field STEM imaging with 0.34 nm resolution, which opens the door for high-resolution STEM imaging in scanning electron microscopes. I will present different application examples from materials science and biology, optimization strategies for low-energy STEM and SEM imaging and also limitations of the present instrumentation and image simulation approaches.

Electron ptychography

Peng Wang

Nanjing University, China

Transmission electron microscope (TEM) is a powerful tool for material science due to its high spatial resolution. Coherent diffraction imaging (CDI) is a "lensless" method that forms an image of an object by solving the phase problem from diffraction patterns with an iterative computer algorithm. This approach can, in principle, overcome the current image resolution limiting factors and ultimately achieve wavelength-limited resolution. However, conventional CDI requires an isolated sample and a priori knowledge about its shape or extent. Rodenburg *et al.* suggested an extended ptychographical iterative engine (ePIE) algorithm, which does not need this prior information and overcomes many of the other issues of CDI, such as non-unique solutions or limited field of view. In this talk, we will show recent two-dimensional (2D) ptychographical reconstructions with sub-ångström lateral resolution achieved using ePIE on CeO_2 and LaB_6 nanoparticles, in which light O or B atoms were clearly resolved together with the heavy Ce or La atoms in their reconstructed phases, respectively. As we know, when the sample becomes thicker, the multiplicative assumption of electron-sample interaction becomes invalid causing the ptychographical reconstruction to breakdown. Secondly, we will demonstrate a successful 3D reconstruction of nanostructured materials from an array of experimental electron diffraction data set using 3PIE based on multislice approach. Finally, we will show some primary work of ptychography on a fast detector.

Electron wavefront engineering: from spherical aberration correction to tunable orbital angular momenta

Peng-Han Lu[1], Roy Shiloh[2], Roei Remez[2], Lei Jin[1], Amir H. Tavabi[1], Martial Duchamp[1,†], Giulio Pozzi[1,3], Ady Arie[2] and Rafal E. Dunin-Borkowski[1]

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Seven years after the first reports of sculpting free electrons into vortex states [1, 2], structured electron waves have now been generated successfully using different approaches and have led to a wide variety of applications in electron microscopy and spectroscopy, as well as to studies of other exotic quantum effects [3]. Holographic diffractive optics [4] are currently most often used to generate shaped electron probes, but require the use of an additional filtering aperture and delicate beam alignment. Here, we present an alternative method, in which a refractive thickness-modulated thin film is used to reduce the spherical aberration of an electron lens [5], thereby providing an immediate low cost upgrade to existing electron microscopes. We also make use of the electrostatic Aharonov-Bohm effect to realize twisted electron beams with tunable orbital angular momentum, paving the way for highly-efficient dynamic electron wavecrafting [6, 7].

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[1] M. Uchida and A. Tonomura, *Nature* **464**, 737 (2010).

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[7] The authors are grateful to the European Research Council under the European Union's Seventh Framework Programme (FP7/2007-2013)/ERC grant agreement number 320832 and the Deutsche Forschungsgemeinschaft for a Deutsch-Israelische Projektkooperation (DIP) Grant for funding.

Ultimate resolution available not only to microscopists but to any materials scientist on any material with the reproducibility and flexibility of the Themis Z platform

Erwan Sourty

Thermo Fisher Scientific Electron Microscopy Solutions

The race for better resolution on the most powerful available microscope platform, the TEM, has culminated 10 years ago with the TEAM project, breaking the 50pm resolution mark on an optimized Titan in both conventional (spread illumination) and scanning (focused illumination) modes. The purpose then was to prove instrumental capability, making the material used a secondary concern, as long as it could help establish a proof of concept.

In more recent years, as resolution is now limited not so much by optics but rather physical dimensions of the atomic potential observed, no significant improvement were made in resolution, or maybe only incremental improvements at lower acceleration voltages. Instead, the focus has been more on increasing reproducibility and flexibility to take full benefit of the reached optical performance, with a deliberate effort to expand application space, in other words providing more meaningful information from more widely relevant materials.

Increasing reproducibility meant introducing remote, computer-assisted, software-enhanced, semi-automated operation, allowing scientists with minimal training to focus on their scientific challenges rather than on optimizing the microscope's optic, yet consistently getting uncompromised performance.

Increasing flexibility meant developing new experimental techniques from signal generation, to data collection, processing, and interpretation. New techniques allowing to discriminate information from target materials, which was previously either mixed up or simply not accessible. Examples are:

- iDCP: high signal-to-noise ratio (SNR) images with linear positive contrast on all materials light or heavy, no matter how beam-sensitive
- Super-X: high sensitivity chemical mapping able to detect all materials including trace elements
- 4D STEM / segmented / pixelated detectors: spatially-resolved electrostatic or magnetic fields, crystallographic phases, or enhanced resolution via ptychography
- Phonon or photon excitations revealed by monochromated electron beams combined with very fast detection speed
- Dynamic states under various stimuli, such as electrical, chemical, mechanical… observed in-situ with atomic resolution

In this presentation, we will discuss the above mentioned reproducibility and flexibility combined with the ultimate optical performance available to date available with the Themis Z platform.

ISTEM: Conventional TEM imaging beyond the diffraction and information limits

Andreas Rosenauer

University of Bremen, Institute of Solid State Physics - Electron Microscopy, Otto-Hahn-Allee NW1, 28359 Bremen, Germany

In this talk the ISTEM ("imaging STEM") imaging mode is introduced which combines STEM illumination with CTEM imaging. We will also reveal by image simulation that this new TEM mode is more robust against chromatic aberration, which allows overcoming the conventional information limit of a microscope. These calculations are confirmed by experimental data for GaN along the [1-100] and [11-20] directions taken on our TITAN 80/300 microscope with a conventional information limit of 80 pm, where we resolved Ga and N columns at a distance of 63 pm. Thus, ISTEM combines advantages of STEM imaging such as improved point resolution with advantages of the CTEM imaging mode while avoiding disadvantages of STEM. In STEM, the precision for determining atom column positions is limited by scan noise which is caused by errors in positioning the electron probe, and the resolution is influenced by the finite source size effect. In contrast, ISTEM images do neither suffer from scan noise nor is the image resolution influenced by the finite source size. Furthermore, we will show by theoretical considerations that ISTEM is independent of lens aberrations of the probe forming system, but only depends on the radius of the probe forming aperture. Due to the principle of reciprocity, ISTEM can be made equivalent to annular bright field STEM using a ring-shaped condenser aperture, promising ultra-high resolution imaging of light elements by avoiding scan noise and source size effect.

Simulation of atomically resolved elemental maps with a multislice method for relativistic electrons

Stephan Majert and Helmut Kohl

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To determine the influence of relativistic effects on elemental maps obtained in an energy filtering transmission electron microscope (EFTEM), we have developed a multi-slice program describing the scattering processes in a fully relativistic manner. Our results show that relativistic effects can play a significant role for inelastic scattering, particularly for high energy losses, small objective aperture angles, and, evidently, high acceleration voltages.

Probing structure and electronic structure of functional oxides at atomic scale by STEM

Lin Gu

Institute of Physics, Chinese Academy of Sciences/Beijing National Laboratory for Condensed, Matter Physics, Beijing, 100190, P.R.China

Functional oxides, for example, lithium ion batteries (LIBs) and sodium ion batteries (SIBs), are promising energy storage devices due to their high energy density and power density, low cost and high safety, respectively. Performance optimization of them necessitates information about structural evolution of electrode materials at an atomic scale. However, limited understanding for the structural evolution of electrode materials at atomic resolution, especially the Li ions and Na ions distribution and arrangement at the atomic level, underlying electrochemical process substantially hinders our exploration of reaction mechanism and further performance optimization. The annular-bright-field (ABF) imaging in aberration-corrected scanning transmission electron microscopy (STEM) allows simultaneous imaging of light and heavy elements, providing an unprecedented opportunity to probe the nearly equilibrated local structure of electrode materials after electrochemical cycling at atomic resolution. In this report, we will present our recent efforts on revealing the atomic-scale structure of selected electrode materials with different charge and/or discharge state, e.g., the lattice distortion, phase interface structure, transition metal migration, surface reconstruction with (partial) intercalation and de-intercalation; information of electronic structure was also be obtained by atomic resolved electron energy loss spectroscopy, extending the current understanding of electrochemical reaction mechanism. At last, our in-situ results on the $LiCoO_2$ will be presented.

Towards atomic-scale electron magnetic circular dichroism

X.Y. Zhong[1,*], Z.C. Wang,[1] A. H. Tavabi,[2] L. Jin,[2] J. Rusz,[3] D. Tyutyunnikov,[3] H.B. Jiang,[1] Y. Moritomo,[4] J. Mayer,[2,5] R.E. Dunin-Borkowski[2], R. Yu[1], J. Zhu[1]

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An atomic-level knowledge of local spin configuration of the magnetic materials is of great importance to predict and control their physical properties, in order to meet the challenges of ever-increasing demands on performance of functional materials. One of the most powerful techniques for imaging magnetic microstructure in the transmission electron microscope is electron energy-loss magnetic chiral dichroism (EMCD) [1]. The technique is similar to XMCD, in that it permits quantitative element-selective determination of spin and orbital magnetic moments in crystalline materials from spectra measured at core loss edges [2-4]. When compared to XMCD, EMCD offers higher spatial resolution and greater depth sensitivity due to the short de Broglie wavelength and penetration of high-energy electrons.

In this talk, we would like to develop the method to access the electron magnetic circular dichroism atomic plane by atomic plane by using spatially resolved electron energy loss spectroscopy in a chromatic/spherical aberration corrected transmission electron microscope and provide direct insight into magnetic spins in materials at the atomic scale. These atomic scale information are not only contribute to a fundamental understanding of the local interplay between charge, spin, orbital and lattice degrees of freedom in magnetic functional materials, but also pave the way for new designs of magnetic materials for future applications with improved device functionality.

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Exploring defects and nanostructures by STEM and spectrum imaging

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Much of our understanding of the properties of bulk crystalline materials comes from their ideal periodic structures. With the reduction of the size down to nanometer scale, ubiquitous defects in the materials often dramatically alter the properties. In order to clarify the role of defects in the properties of the materials, local structure, composition, and bonding need to be studied the atomic scale. Here, we use aberration-corrected scanning transmission electron microscopy (STEM) and EDX/EELS spectrum imaging techniques to explore the local atomic structures, compositions and electrical properties of $SrTiO_3$ based bicrystals, thin films, nanostructures and devices. We determine atomic structures and compositions of dislocation cores at small tilt angle grain boundaries in $SrTiO_3$ bicrystals [1] and antiphase nanodomains in Fe-doped $SrTiO_3$ thin films [2]. We characterize surface atomic structure of $Sr(Ti,Zr)O_3$ nanocubes [3], and we quantitatively study conducting filaments in Fe-doped $SrTiO_3$ thin film resistive switching memories [4]. Our findings may have profound implications for the applications of local defects and interfaces in future devices.

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Differential phase contrast - imaging of fields in matter on a mesoscopic and atomic scale

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Differential phase contrast is known since the 1970s to be able to image mesoscopic magnetic induction distribution within magnetic specimens. In contrast to other imaging techniques which are also capable of magnetic structure imaging - such as Lorentz or Fresnel microscopy - the images obtained are in focus and can be quantified rather straightforward. Meanwhile, imaging of electrostatic potential gradients, both on a nm and pm scale, have been published and gained quite some attention. While measurements taken with the traditional annular four-quadrant detector suffer from deficiencies for quantification, the advent of pixelated detectors allows to close this gap. Measurements of magnetic structures and on structural defects (dislocations) will be presented, taken with the traditional and also with a pixelated detector.

Quantitative off-axis electron holography for magnetic skyrmions of nanostructures

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The particle-like swirling spin textures, termed as magnetic skyrmions, exhibit novel properties including topological stability, easy motion by small electric currents, and nanoscale compact size make them promising magnetic elements for data storage and spintronics applications. Recently, intense research interest is directed to the confinment effects of skyrmions in reduced dimensions, e.g., thin films and nanostructures. Direct imaging of skyrmion in nanostructures provides valuable insights into the skyrmion physics at the nanoscale. However, resolving skyrmionic spin textures in nanostructures is highly challenging because of the required spatial resolution and magnetic sensitivity. The technique of off-axis electron holography (EH) in the TEM has nanometer spatial resolution, high phase sensitivity and permits reliable quantification of magnetic structures in nanostructures. In this contribution, we will discuss our recent EH investigations of (i) highly confined skyrmion chain in wedge FeGe nanostripes, (ii) crystallographic grain-boundary-mediated skyrmion formations, and (iii) formation of chiral antiskyrmion lattice in periodically modulated micro-nano twinned Heusler Mn-Pt-Sn alloys.

In situ electron microscopy investigation on plastic deformation in a metastable beta titanium alloy

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Metastable beta titanium alloys with body-centred cubic lattice have attracted extensive research because they exhibit a combination of high strength and toughness. Ti–24Nb–4Zr-8Sn (Ti2448) alloy, one of recently developed metastable beta titanium alloys, combines low Young's modulus, superior biocompatibility and good workability. In addition, its nonlinear pseudo-elasticity and absence of double yielding also generate strong research interest. The present study investigates the evolution of deformation mechanisms in single crystal Ti2448 alloy with in situ tensile tests in scanning and transmission electron microscopes. The results reveal an evolution from martensitic transformation to deformation twinning to dislocation slip during the deformation, which are responsible for the pseudo-elasticity and high damping effect of the alloy. Quantitative strain analysis on aberration-corrected electron microscopy images resolves nanometer scaled local stress around deflected crack paths of fatigue tests and indicates the origin of crack deflection and bifurcation, which introduce excellent fracture toughness for the alloy subject to high cyclic loading.

Imaging the structural evolution of nanocrystalline metals during thermal annealing and mechanical deformation

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In situ TEM techniques allow to directly image the structural changes in response to an external stimulus thus enabling a detailed analysis of the corresponding processes and mechanisms. We have developed a combination of *in situ* studies with 4D-STEM imaging, in particular using ACOM-STEM, to not only follow morphological changes of a statistically meaningful sample volume but to retrieve crystallographic information of the structural changes during post processing using both local and statistical analysis of the resulting crystal orientation maps. However, critical attention has to be paid to artifacts for the data interpretation, in particular due to bending and projection effects in nanocrystalline materials as well as the so called 180° ambiguity. Using this approach, we have extensively investigated strain induced grain structure changes (twinning, Σ9 boundary formation, grain rotation) and the Bauschinger effect in nanocrystalline PdAu alloys, which was further supported by 'classical' BF/DF *in situ* TEM to also acquire high-quality mechanical data. As another example, the thermally induced grain growth of these alloys will be presented.

Tuning the magnetism of epitaxial cobalt oxide thin films by electron beam irradiation

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Tuning magnetic properties of perovskite thin films is a central topic of recent studies because of its fundamental significance. In this work, we demonstrated the modification of the magnetism of $La_{0.9}Ca_{0.1}CoO_3$ (LCCO) thin films by introducing stripe-like superstructure in a controllable manner using the electron beam irradiation (EBI) in a transmission electron microscope. The microstructure, electronic structure, strain change and origin of magnetism of the LCCO thin films were studied in details using aberration-corrected scanning transmission electron microscopy, electron energy loss spectroscopy and *ab initio* calculations based on density functional theory [1]. The results indicate that the EBI induced unit cell volume expansion accompanies the formation of oxygen vacancies and leads to the spin state transition of Co ions. The low spin state of Co^{4+} ions depress the stripe-like superstructure, while higher spin states of Co ions with lower valences are conductive to the formation of "dark stripes" [2]. Our work clarifies the origin of magnetism of epitaxial LCCO thin films, benefiting a comprehensive understanding of correlated physics in cobalt oxide thin films.

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In situ TEM study on sub-10-nm materials

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With the development of semiconductor technology, the 10 nm feature size of fabrication is approaching. It is thus quite essential to explore more precise nanofabrication and characterization method to evaluate the shape/structure stability and possible new properties of sub-10-nm material components, especially under external field such as strain, electric, or thermal fields. Here we review our recent progress in atomic resolution nanofabrication and dynamic characterization of individual nanostructures and nanodevices based on the idea of “setting up a nanolab inside a transmission electron microscope”. The electron beam can be used as a tool to induce nanofabrication on the atomic scale. Additional probes from a special-designed holder provide the possibility to further manipulate and measure the electric/mechanical properties of the nanostructures in the small specimen chamber of a TEM. Recently, the optical signal also was introduced into the electron microscope to enrich the coverage of investigation inside the “multifunctional nanolab”. All phenomena from the in-situ experiments can be recorded in real time with atomic resolution.

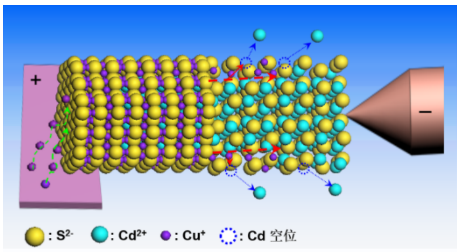

Fig 1 Schematic diagram of electrically driven cation exchange for in-situ fabrication of individual nanostructures.

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Atomic-scale structure of heterointerface and planar defects in perovskite-type oxides

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Nowadays, advanced imaging techniques based on aberration-corrected electron microscopy, e.g. negative C_s imaging (NCSI), high-angle annular dark field (HAADF) and annular bright field (ABF) imaging have been applied for not only acquiring atomic-resolution structure images of materials, but also determining the relative shifts of atomic columns with a precision of a few picometres, which provide us with unrivaled capability to understand the behavior of materials at the atomic level.

Here we provide the atomic-scale structure details of the interface and domain wall in the $Pb(Zr,Ti)O_3/SrRuO_3$ heterostructures, and planar defects in lead-free piezoelectric (1-*x*)($Na_{0.5}Bi_{0.5}TiO_3$)-*x*($BaTiO_3$)(NBT-BT) films by aberration-corrected high-resolution electron microscopy. In the $Pb(Zr,Ti)O_3/SrRuO_3$ heterostructure, the formation of an interfacial depolarization layer and 180° domains was observed in the ferroelectric films, which compensates the polarization bound charges induced by the ferroelectric film at the interface. In the NBT-BT films, two types of planar defects, zigzag-like planar defects and Aurivillius-type planar defects have been characterized. The complex propagation of planar defects results in kinks of the same type of planar defects and junctions between different types of planar defects in the NBT-BT films. The formation of the planar defects is due to Na deficient and Bi excess in the films, which is responsible for the difference in the physical properties between the bulk materials and the films.

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Perfect interfaces between corundum and rutile structures

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Corundum and rutile are two common structure types of oxides. In addition to alpha alumina itself, corundum structure is also found in Fe_2O_3, Cr_2O_3, V_2O_3, and other transition metal oxides. In addition to the rutile mineral itself, the structure is also found in CrO_2, VO_2, MnO_2, and RuO_2, etc. We found that these two seemingly distinct structures can be transformed to each other through topotactic reaction by in-situ reduction in transmission electron microscope and form a perfect coherent interface. The heterojunctions of various corundum oxides and rutile oxides were prepared by thin film growth. Combining aberration-corrected TEM and the first-principles calculations, the atomic configuration, electronic structure and magnetism, stable configurations of cations near the interfaces, and the behavior of cations in topotactic reaction have been revealed.

Hetero-interface and its performance to materials

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Although elastic strains, particularly inhomogeneous strains, are known to be able to tune, enhance, or create novel properties of some nanoscale functional materials[1], potential devices with exotic properties dominated by inhomogeneous strains have not been achieved so far. Such stagnations lie in the general predetermination that the elastic energy resultant from the disclination strains therein would be so pronounced that a large scale assembling of inhomogeneous strains could be rather difficult. We fabricate inhomogeneous strains with a linear gradient as giant as 10^6 per meter, featuring an extremely lower elastic energy cost compared with a uniformly strained state[2]. The present strain gradient, resulting from the disclinations in the perovskite $BiFeO_3$ nanostructures array grown on a $LaAlO_3$ substrate via a high deposition flux, induces a polarization of several microcoulomb per square centimeter through flexoelectric coupling, which is derived from the strain mapping on the basis of aberration-corrected scanning transmission electron microscopy. It leads to a large built-in electric field of several megavoltage per meter which is comparable to that of the conventional p-n junctions and Schottky diodes. It also gives rise to a large enhancement of solar absorption as confirmed by the UV-visible absorption measurements. Our results indicate that by engineering strains with giant linear gradient it is possible to build up a large-scale strain-dominated nanostructure array with exotic properties, which in turn could be useful in the development of novel devices for electromechanical and photoelectric applications.

In addition, we will also present our recent studies on giant enhancement of polarization in ultrathin ferroelectric films mediated by charge transfer[3,4].

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Topological vortices and charge-lattice interactions in multiferroic oxides

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Topological structures emerging near spontaneous symmetry-breaking transitions are ubiquitously observed in wide branches of science. Topological defects are invariant under continuous deformations or perturbations, and thus considered to be protected by topology. Here, we report structural transformation of sixfold vortex domains into two-, four-, and eightfold vortices via a different type of topological defect in hexagonal manganites. Combining high-resolution electron microscopy and Landau-theory-based numerical simulations, we investigate the remarkable atomic arrangement and the intertwined relationship between the vortex structures and the topological defects. The roles of their displacement field, formation temperature, and nucleation sites are revealed. All conceivable vortices in the system are topologically classified using homotopy group theory, and their origins are identified.

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Preparation and analysis of nanoporous gold

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Nanoporous metals are of great interest for current research in materials science due to a variety of potential applications, i.e. in catalysis, as actuators or as lightweight material with distinct mechanical properties. The local crystal structure, elemental distributions as well as the three dimensional morphology of this comparatively new class of materials is of relevance because it correlates with important properties, e.g. deformation behavior, mechanical stability or surface structure.

Electron microscopy and related techniques are some of the preferred tools to analyze such materials: large-area, high-resolution structural and orientation information can be obtained by Scanning Electron Microscopy (SEM) and Electron Backscatter Diffraction (EBSD). To study the morphology of larger volumes, Focused Ion Beam (FIB) in combination with SEM (FIB-SEM tomography) is applied. Energy dispersive X-ray spectroscopy (EDS) or tomography (3D EDS) using Scanning Transmission Electron Microscopy (STEM) yields the high-resolution, three-dimensional morphology and elemental distribution in smaller volumes.

However, due to their high surface area and small structure sizes, most nanoporous metals are particularly susceptible to the introduction of preparation defects, especially when using mechanical sample preparation methods. Also, the three-dimensional analysis of its tomographic data is not straight forward and free of artifacts. Hence, one must exercise particular care when interpreting the information on nanoporous metals provided by electron microscopic data.

This study investigates the impact of relevant sample preparation techniques like Ultramicrotomy (UM) or FIB-SEM for crystallographic investigations as well as data analysis techniques like decomposition and clustering for chemical or three-dimensional structural analysis on one of the model systems for nanoporous metals: nanoporous gold (npg).

Atomic resolution TEM characterization of GaSb/GaInAs and GaSb/GaInP bond interfaces for high-efficiency solar cells

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Multi-junction solar cells based on III-V semiconductors reach the highest conversion efficiencies and are currently used primarily in concentrator photovoltaic systems and for power generation on satellites or spacecraft. Several cells of different III-V compound semiconductor materials are generally combined to absorb a different wavelength range of the solar spectrum and to convert it into electric power. Fabrication of these cells by wafer bonding is of interest since efficiencies of up to 46% have been obtained [1], and efficiencies of up to 50% are within reach. Fast atom beam activation is generally used as a pre-treatment to remove oxides and contamination before bonding [2]. Activation treatment and bond processing often result in the formation of amorphous interface layers with inadvertent impurities [3]. We have applied high-resolution imaging and spectroscopic transmission electron microscopy (TEM) techniques to investigate interface regions in as-bonded and thermally annealed (at temperatures of $T \leq 500^{o}C$) GaSb/GaInAs and GaSb/GaInP layer systems. We used aberration-corrected high-resolution TEM, high-angle annular dark-field scanning (S)TEM, and energy-dispersive X-ray spectroscopy (XEDS) with an aberration-corrected probe to monitor elemental distributions with high precision and sub-nanometer spatial resolution.

For GaSb/GaInAs, we find that the crystal lattices are interconnected. The bond interfaces exhibit terraces, misfit dislocations, and nanometer-sized pores, as well as compositional fluctuations in the near-interface regions. For GaSb/GaInP, the interface regions are characterized by an amorphous interlayer that has a thickness of approximately 1.5 nm, with a minor enrichment of Ga. These phenomena are attributed to the wafer pre-treatment before bonding. Thermal annealing, which is often used as a method for improving the interface conductance, results in changes in structure and composition. The amorphous interlayers are reduced in thickness by recrystallization, resulting in a largely epitaxial interface after annealing at 500 ^{o}C. XEDS mapping reveals detectable amounts of In and P after annealing at temperatures $T \geq 225°C$, as well as small pores and In-rich crystalline precipitates for

T ≥ 400°C in the GaSb near-interface regions. These observations provide an understanding of the electrical properties of the interfaces [4] and can be compared with results obtained from GaAs/Si wafer-bond interfaces [3]. From a methodological point of view, our results show how aberration-corrected TEM can be used to contribute to the monitoring, control, and optimization of concepts for the fabrication of high-efficiency solar cells. An optimized 4-junction solar cell based on GaInP/AlGaAs//GaInAs/Ge currently has an efficiency of 38.5% under a concentration of 188 suns, while a GaInP/GaAs/GaInAs//GaSb cell has a first efficiency of 29.1 % under 194 suns.

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Understanding novel domain science in multiferroic materials by advanced electron microscopy

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As advances in transmission electron microscopy (TEM) have enabled the determination of the three-dimensional structure and local properties of materials with the sub-angstrom resolution, the recent development of *in situ* TEM techniques allows one to follow the dynamic response of nanostructured materials to applied fields. In this talk, I will present our TEM studies of the polarization ordering and dynamic domain switching behaviors of ferroelectric/multiferroic thin films. It was found that the charged domain walls can be created or erased by applying a bias, and the resistance of the local film strongly depends on the characteristics of the charged domain walls. It will also be show that the surface monolayer of conducting oxide can induce a giant spontaneous polarization in ultrathin multiferroelectric films and that a peculiar rumpled nanodomain structure, which is in analog to morphotropic phase boundaries (MPB), is formed. Finally, it will be demonstrated that small defects in ferroelectric thin films can act as nano-building-blocks for the emergence of novel topological states of polarization ordering, namely, hedgehog/antihedgehog nanodomain arrays.

Microstructures and dynamic evolutions in low-dimensional materials

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Low-dimensional materials spark tremendous interest owing to their intriguing mechanical, physical and chemical features. Here, applying the advanced electron microscopy and X-ray powder diffraction techniques, we present detailed atomic-scale structural investigations in several low-dimensional materials, including semiconductor InAs nanowires (NWs), and some functional oxides (e.g. $Li_{1+\alpha}Mn_{2-\alpha}O_{4-\delta}$, CuO, etc)[1-6]. The atomic-scale structural characterizations reveal the existance of different domain structures, such as twinning variants, which inevitably affect the related properties.

In addition, in-situ transmission electron microscopy has been employed to study the real-time structural evolution of nanomaterials subjected to external stimuli, such as mechanical stress and electric field, which are frequently encountered during the applications[7-11]. For example, during compression, the CuO NWs exhibited high bending capabilities associated with high mechanical stress[10]. Interestingly, anelasticity was consistently observed after stress release. A mechanism based on the cooperative motion of twin-associated atoms is proposed to account for this phenomenon. Meanwhile, by constructing the nano-scale solid-state sodium ion battery, real-time microstructural evolution during the sodiation/desodiation of CuO nanowires was directly recorded[11]. We show that the surface coating with carbon (C), gold (Au) could effectively constrain the nanowire (NW) elongation rate along the <110> growth direction as well as increase the electro-chemical reaction speed, which result in the improved cycling performance.

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Atomic-resolution HAADF-STEM and EDS-STEM characterization of metallic alloys

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Lightweight metallic alloys (such as aluminium alloys, magnesium alloys, and intermetallics) are a major group of structural materials. Mechanical properties of this group of materials depend critically on their microstructures which are strongly influenced by alloying elements. Several models have been proposed on the precise roles of alloying elements in these alloys, but the validity of these models has not been rigorously examined due to a lack of direct experimental observations at atomic scale. While the advent of high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) provides much more information at the atomic-scale, there is still a need for performing atomic-resolution chemical mapping using advanced STEM techniques, such as energy-dispersive X-ray spectroscopy (EDS-STEM) and/or electron energy-loss spectroscopy (EELS-STEM), if the precise roles of the alloying elements are to be revealed.

Real time atomic scale imaging of catalysts during catalytic reaction

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Chemistry, especially heterogeneous catalysis, has a pivotal role in solving the global challenges of energy and environment. This is not only because catalysts are involved in 85% of the daily synthesis of materials, but also because they contribute to the effective energy storage [1]. Revolutionary advances in catalysis research, instead of evolutionary developments, is required by the society as the risk of disturbing the thermal equilibrium system of the planet getting more and more serious [2].

Conventional transmission electron microscopy (CTEM) has been widely used for characterizing the microstructure of the catalyst particles before and after the reaction. Knowledge of particle size distribution, chemical composition, crystal, atom and electronic structure, greatly helps us to understand the structure information of the catalyst at its stable states [3]. However, as the working structure of a catalyst is often a transient, metastable form, and highly dependent to the reaction environment, to fully understand the catalytic mechanism catalysts are needed to be studied during the catalytic reaction. This is not possible in CTEM, due to the high vacuum environment and room temperature limitation of CTEM.

Using the latest nano-reactor technology [4], we have realized the high-pressure gas and high-temperature environment (10^5 Pa, 1000 C) required for chemical synthesis and catalyst reaction inside the electron microscope maintaining the characterization power of TEM. The entire workflow of catalysis, starting from preparation/synthesis of the catalyst, evaluation of the reaction activity and characterization of the real-time structure dynamics can be observed directly in TEM at atomic level.

This report will show in situ characterization of a series of Cu, Ni, Ir-CeO_2 catalyst. Through these results, we would like to demonstrate that in situ TEM provides a revolutionary approach to catalysis research.

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Ag induced phase transformation of a ∑5 grain boundary in Cu

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Recently, it has been revealed that grain boundaries can undergo phase transformations leading to changes in material properties such as diffusion and grain growth. In this study, we explore the differences in atomic structure of a pure and Ag alloyed ∑5 36.9° {310} Cu tilt grain boundary. The grain boundary is fabricated by growing a bulk Cu bicrystal using the Bridgeman method. Ag alloying of the ∑5 grain boundary is performed by sputter deposition of a Ag film on the bicrystal followed by annealing at 800°C for 120 h under high vacuum conditions. Scanning transmission electron microscopy (STEM) in a FEI Titan Themis 60-300 is used to acquire STEM micrographs at 300 kV under high angle annular dark field (HAADF) conditions resolving the atomic and chemical structure of the grain boundary. Constraints of beam induced Ag migration effects and changes in grain boundary structure of the pure and alloyed grain boundary are discussed in the talk.

Atomic-scale structure evolution of growing precipitates in high-performance aluminum alloys

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Developments of high-strength aluminum alloys have always faced a difficult problem: owing to their small size, the early-stage strengthening precipitates are difficult to characterize in terms of composition, structure and evolution. Here we employ atomic-resolution transmission electron microscopy (TEM) imaging and first-principles energy calculations to address these problems. Recent years, we have investigated tens of typical high strength aluminum alloys, such as 2xxx (AlCu, AlCuMg and AlCuLiMg), 6xxx (AlMgSi and AlMgSiCu) and 7xxx (AlZnMg and AlZnMgCu) alloys, with different compositions and with varying thermal processes for understanding their property-structure-process correlations. Using aberration-corrected high-resolution TEM (HRTEM) and aberration-corrected scanning TEM (STEM), much of our attention has been paid to revisit the strengthening precipitates in these important alloys and to clarify the controversies left in the past about their precipitation behaviors. Our study demonstrates the followings:

(1) Atomic-resolution imaging in STEM can provide straightforward structure models at the atomic-scale, whereas atomic-resolution imaging in HRTEM with rapid quantitative image simulation analysis can provide the refined structures with high precision beyond the resolution limitation of the microscope. The combination of the two techniques can be more powerful in solving difficult structure problems in materials science.

(2) Most of the early-stage precipitates in aluminum alloys are highly dynamic in both composition and structure. Typically, having their characteristic genetic skeletons to guide their evolution, these dynamic precipitates initiate, mature and grow with thermal aging following characteristic evolution paths. The fine precipitation scenarios revealed in our studies are rather different from previous understandings in the textbooks and literatures published thus far.

Morphology and stability of orthorhombic and hexagonal phases in a lamellar γ-Ti-42Al-8.5Nb alloy – a transmission electron microscopy study

Florian Pyzcak

Heike Gabrisch and Florian Pyczak, Helmholtz-Zentrum Geesthacht*

In intermetallic titanium aluminides alloyed with niobium depending on the overall alloy composition new phases unknown from the binary Ti-Al system may be introduced in the alloys' microstructure. Here we describe the microstructure of the alloy Ti-42Al-8.5Nb. We identify the orthorhombic O-phase within α_2 laths of lamellar (α_2 / γ) colonies by single crystal electron diffraction and high-resolution imaging. Domains of O-phase variants and α_2 phase form columnar crystallites in α_2 (0001) planes having low indexed α_2 {11-20} and {1-100} interface planes. The nm-sized domains are rotated with respect to each other resulting in elastic strains across the domain boundaries. No elemental segregation of Nb was detected in α_2/O-phase lamellae.

Correlative Cs corrected STEM-atom probe tomography on Kappa-carbides in steel

C. H. Liebscher[1], M. Lipinska-Chwalek[2,3], M. Yao[1], M. Herbig[1], J. Mayer[2,3], G. Dehm[1,] D. Raabe[1] and C. Scheu[1]

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High-Mn steels have a high strength, which is due to a regularly arranged network of nano-sized $(Fe,Mn)_3AlC$ kappa carbides embedded in an austenitic matrix. In the present work we correlate aberration-corrected scanning transmission electron microscopy (STEM) with atom probe tomography (APT) to determine the atomic arrangement within the kappa carbides, their chemical composition as well as the interface structure to the matrix. The measurements are performed at identical samples which are first investigated in STEM and then field-evaporated in the APT. The analysis reveals that two types of kappa carbides with cuboidal and plate shaped geometry have formed in a Fe-29.8Mn-7.7Al-1.3C (wt%) alloy annealed at 600°C for 24h, both having a non-stoichiometric composition. The interface between the kappa carbides and the austenitic matrix is fully coherent and no misfit dislocations occur. Interesting the STEM images indicate that the matrix between two closely spaced carbides is heavily strained and tetragonal distorted. While the APT data reveal a rather broad diffusion profile across the interface, the STEM images show chemical fluctuations at the interface only in the order of few atomic distances.

3D characterization of dislocation structures and solute distributions in light alloys

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*xiaoxuhuang@cqu.edu.cn

Mechanical properties of metals and alloys are related to their chemical, microstructural, and crystallographic parameters. Recent development of electron tomography, 3D orientation mapping and 3D atom probe has enabled that a diversity of 3D microstructural, crystallographic and chemical information can be obtained at nanoscale for a variety of materials, offering new opportunities for an enhanced understanding of structure-property relationships. In this presentation focus is made on 3D characterization of dislocation structures and solute distributions in light alloys, and examples will be demonstrated and discussed with respect to their effect on materials properties.

Temperature effect on hydrogen behavior in submicron-sized Al pillars

Zhiwei Shan

Xi'an Jiaotong University, Xi'an, China

Hydrogen can facilitate the detachment of protective oxide layer off metals and alloys. The degradation is usually exacerbated at elevated temperatures in many industrial applications; however, its origin remains poorly understood. In order to solve this puzzle, we have developed a series of MEMS based heating devices (China patents: 201410542592.X[P], 201510437346.2[P], 201510710901.4[P], 201621190157.6[P]) which enable us to solve the major problems of all the existing heating devices, such as the incapability of getting high resolution image of the micro-structural evolution during temperature change, ununiformed temperature distribution, inaccurate temperature measurement and control. Further, we demonstrate that by heating hydrogenated aluminum inside an environmental transmission electron microscope, we show that hydrogen exposure of just a few minutes can greatly degrade the high temperature integrity of metal–oxide interface. Moreover, there exists a critical temperature of 150 degree, above which the growth of cavities at the metal–oxide interface reverses to shrinkage, followed by the formation of a few giant cavities. Vacancy supersaturation, activation of a long-range diffusion pathway along the detached interface and the dissociation of hydrogen-vacancy complexes are critical factors affecting this behavior. These results advance the understanding of hydrogen induced interfacial failure at elevated temperatures. [1]

Reference:

1. Li, M., et al., *Effect of hydrogen on the integrity of aluminium-oxide interface at elevated temperatures.* Nature Communications, 2017. **8**.

New materials characterization with TEM orientation imaging/phase mapping in combination with e-PDF in situ analysis at nm scale

Stavros Nicolopoulos

NanoMEGAS SPRL Blvd Edmond Machtens 79 B-1080 Brussels Belgium

The development of TEM based- automated crystallographic mapping techniques (ASTAR) in combination with precession electron diffraction (PED) has pushed the resolution limit at 1 nm scale. Combination of ASTAR 4D scanning orientation/phase map technique with advanced TEM Cs corrected /FEG instruments over the last 10 years allowed study of various materials such as metals alloys,minerals , semiconductors and even organic materials. The combination of PED with scanning nanodiffraction enables to measure accurately strain (precision up to 0.01%) with 1-3 nm resolution at semiconductor devices.

The recent application of e-PDF (electron Pair Distribution Function) techniques allows to analyse at local scale Electron Diffraction (ED) patterns even from amorphous materials. e-PDF technique allows to analyse interatomic distances, bonding and possible short/large scale order of nanocrystalline /amorphous materials at nm scale, enabling to monitor in situ solid state reactions, structure of glassy materials, layered thin films quality and amorphous/ re-crystallization studies in semiconductor devices.

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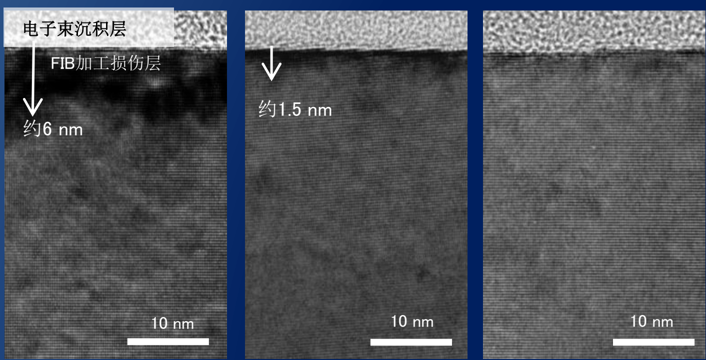
  High-grade TEM sample preparation possible with Micro Sampling* and Triple Beam system*.
  *optional

三束系统应用/ Triple-Beam effect



(a) 5kV-Ga 离子加工
(a) 5kV Gallium ion milling

(b) 1kV-Ga离子加工
(b) 1kV Gallium ion milling

(c) 1kV-Ar离子加工
(c) 1kV Argon ion milling

样品(GaN)： [1-100]方向的高分辨TEM像
Ga离子加工后可以观察到有损伤层（如箭头所示）
Ar离子加工后还可看到样品自表面开始的结晶特性保持完好
High-resolution TEM images of GaN crystal in <1-100> plane
1kV Argon ion milling preserve crystallinity to the surface

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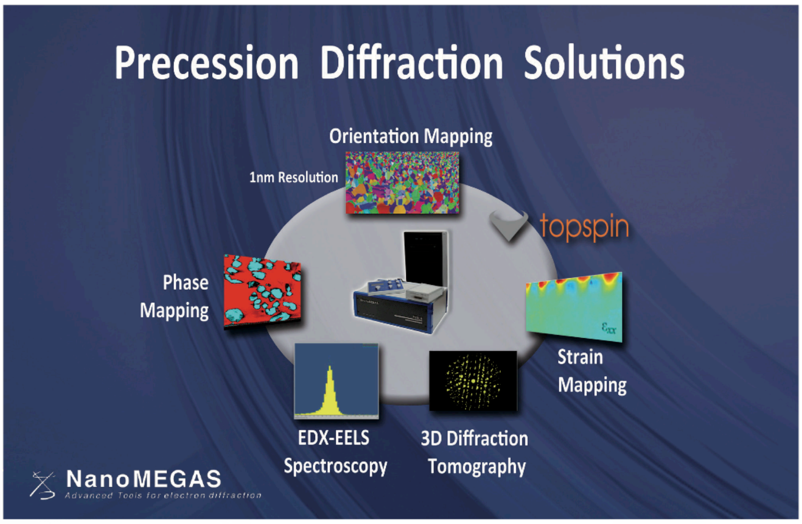
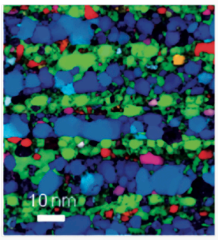
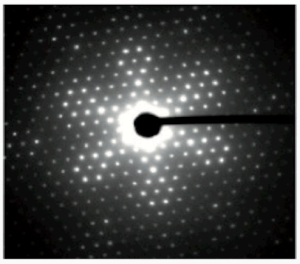
TEM orientation / phase imaging (ASTAR), Presession enabled electron diffraction (PED), with Strain mapping at 1-4 nm scale (AutoSTRAIN), Automated PED tomography (ADT-3D) for ab-initi solution of nanostructures.

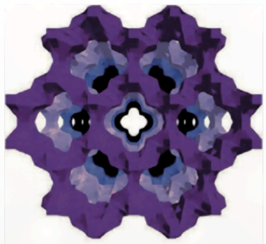
TOPSPIN platform has been developed as an analytical experimental framework that offers a suite of PED enabled advanced solutions including ASTAR, Autostrain and other custom-tailored experiments.

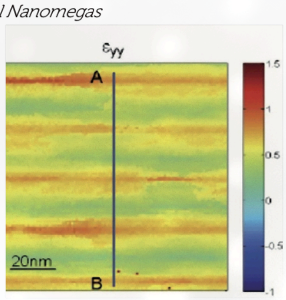




ASTAR-TOPSPIN TEM orientation imaging of Cu interconnects (Libra 200 F, PED 0.4°, step size 3 nm)
courtesy Dr. Darbal Nanomegas USA


PED patern of a mayenite cubic mineral


Structure of ITQ-43 zeolite revealed by ADT-3D analysis
courtesy Prof. Kolb Mainz Univ



TOPSPIN-AutoStrain map with Si / SiGe calibration sample (step size 1 nm, ARM 200 F, PED angle 0.7°
courtesy Dr. A.Darbal Nanomegas USA

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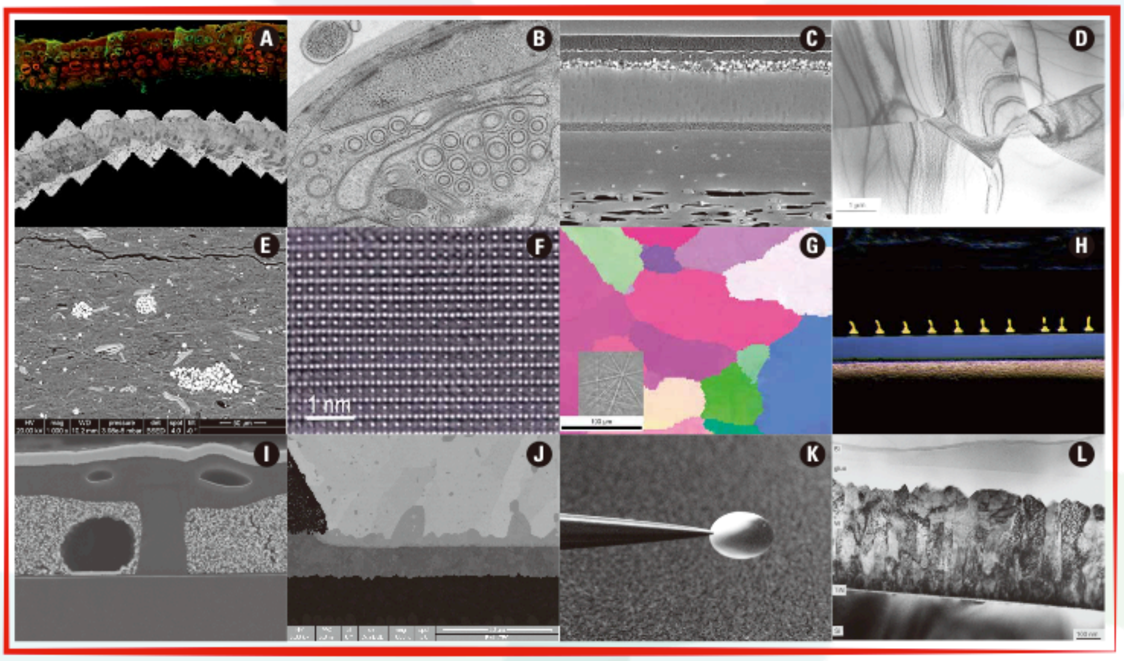


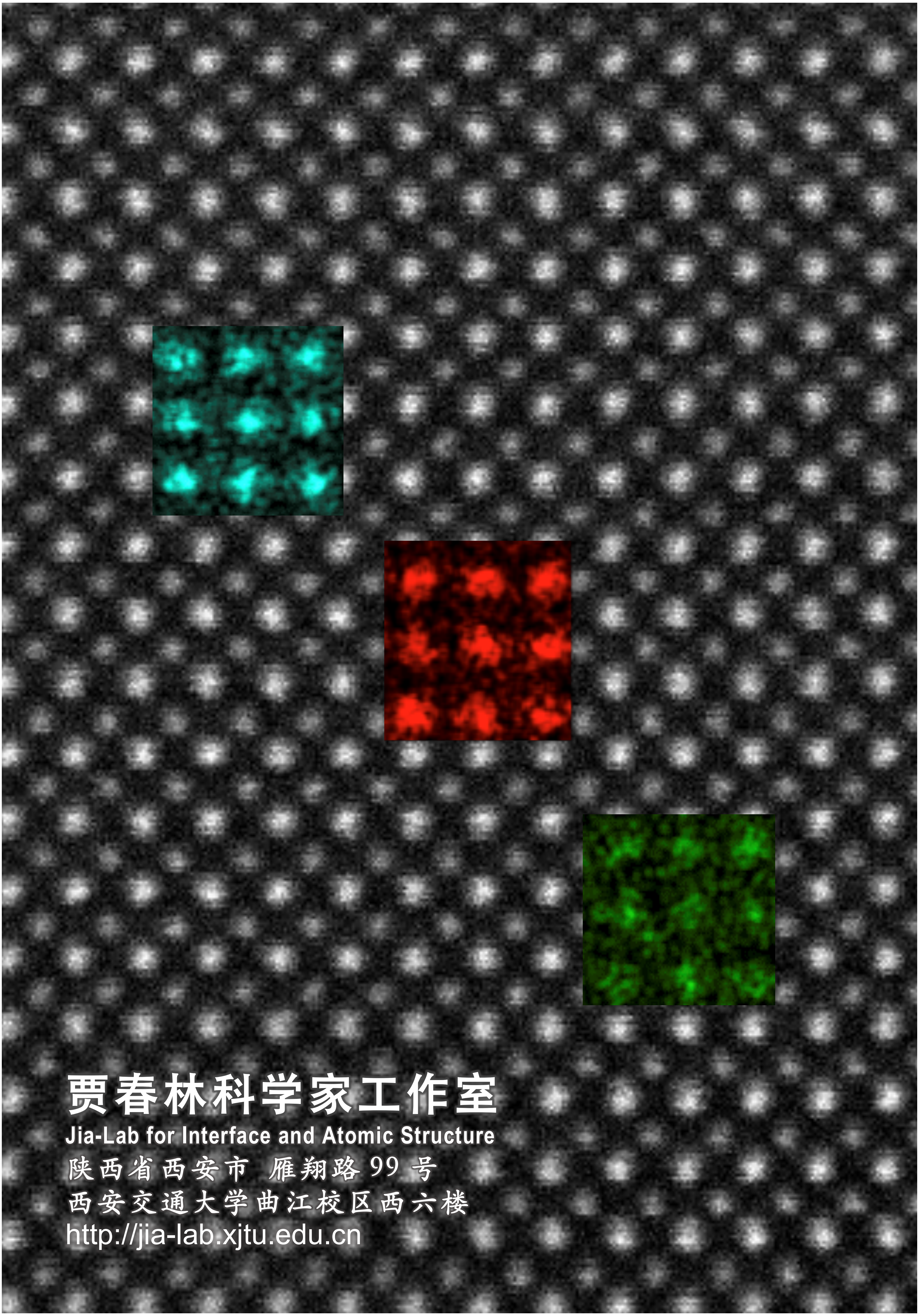
A: 小鼠表皮细胞 B: 线虫头部截面 C: 啤酒瓶标签纸截面 D: Ti薄膜 E: 泥页岩样品 F: $SrTiO_3$ G: 铝合金样品 H: 金线焊接点截面 I: 电子书
J: 焊球截面 K: 直径3mm碳化钨样品薄片（9μm厚度） L: 多层膜样品

工业材料制样流程

	1	2	3	4	5	6	应用实例
高分子材料/TEM	修块 TXP	超薄切片 UC7/FC7(KMR3)	TEM				
高分子材料/SEM	修块 TXP	钻石刀切割 UC7/FC7(KMR3)	镀膜 ACE200/600	SEM			C
固体材料/TEM	机械抛光减薄 TXP	离子束减薄 RES102	TEM				D F L
固体材料/SEM	样品目标定位 TXP	离子束切割/TIC 3X	镀膜 ACE200/600	SEM/EBSD			E G I J
易氧化样品/SEM	手套箱中预处理 TXP	离子束切割/抛光TIC 3X	环境传输 VCT	镀膜 ACE600	环境传输 VCT	SEM	
固体材料内部/SEM	机械研磨抛光 TXP	SEM/LM					H K
不导电材料形貌/SEM	镀膜 ACE200/600	SEM/EDX					
流体材料/Cryo-SEM	高压冷冻 ICE	真空冷冻传输 VCT	镀膜 ACE600	真空冷冻传输 VCT	Cryo-SEM		

生命科学制样流程

	1	2	3	4	5	6	7	应用实例
常温TEM	组织处理 TP/AMW	修块 TRIM2/RAPID/TXP	超薄切片 UC7 (KMR3)	染色 AC20	TEM			
常温TEM	高压冷冻 ICE	冷冻替代 AFS2	修块 TRIM2	超薄切片 UC7 (KMR3)	染色 AC20	TEM		B
光镜电镜联用CLEM	光学成像(带定位网格)	高压冷冻 ICE	冷冻替代 AFS2	修块 TRIM2	超薄切片 UC7 (KMR3)	染色 AC20	TEM/CLEM	A
冷冻CLEM	高压冷冻/冷冻切片 或投入冷冻 GP	冷冻工作站 和真空冷冻传输 VCT	冷冻光学成像 (New Cryo LM, Only Leica!)	CLEM 软件位置定位	Cryo-TEM	Cryo-CLEM		
冷冻TEM	投入冷冻 GP	Cryo-TEM						
冷冻TEM	高压冷冻 ICE	冷冻超薄切片 UC7FC7	Cryo-TEM (CEMOVIS)					
常温SEM	组织处理 TP	临界点干燥 CPD300	镀膜 ACE200/600	SEM				
冷冻SEM	高压冷冻 ICE	真空冷冻传输 VCT	镀膜 ACE600	真空冷冻传输 VCT	Cryo-SEM			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