PICO 2022

25 Years of Aberration Correction

SEVENTH CONFERENCE ON FRONTIERS OF ABERRATION CORRECTED ELECTRON MICROSCOPY

Kasteel Vaalsbroek

May 08th – May 12th

2022

PICO2022 – KASTEEL VAALSBROEK

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celebrating

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Conference Organisers

Rafal Dunin-Borkowski (Jülich)

Joachim Mayer (Aachen)

Carsten Sachse (Jülich)

Knut Urban (Jülich)

Genevieve Wilbs (Jülich)

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25 Years of Aberration Correction

The organisers kindly acknowledge the support of PICO2022 from the following sponsors:

The cover displays a high-resolution micrograph of an interface between polar $LaVO_3$ (top) and nonpolar $SrTiO_3$ (bottom) recorded along the ‹110› zone axis of $SrTiO_3$ under NCSI conditions by Lei Jin. Upward shifts of every second oxygen column with respect to the adjacent vanadium columns (fourth row from the bottom) give evidence for an interface reconstruction. Local variations in the 90° elastic domain structures in $LaVO_3$ become evident when analysing the angular variation of -O-V-O-V-O- chains ranging from 164° on the left (verso) to about 180° on the right (front cover).

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Preface

PICO2022 – the seventh Conference on Frontiers of Aberration Corrected Electron Microscopy, celebrating 25 years of aberration correction – will be held in Kasteel Vaalsbroek from May 08th – May 12th, 2022. The event is hosted by the Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons (Forschungszentrum Jülich GmbH and RWTH Aachen University) and supported by FEI Electron Optics BV (Part of Thermo Fisher Scientific), JEOL (Germany) GmbH, AMETEK GmbH, Hitachi High-Tech Europe GmbH, CEOS GmbH, DECTRIS AG, Quantum Detectors Ltd, NanoMEGAS and EMSIS, condenZero GmbH, Quantifoil Micro Tools GmbH, DENSsolutions B.V., Protochips EMEA GmbH and EMS (European Microscopy Society).

As EMS Extension meeting, we are happy to announce that we will also host this year's EMS outstanding paper award.

The conference has attracted more than 190 participants from 25 countries throughout the world and the programme committee has put together an oral programme including 55 scientific keynote lectures. Further contributions are scheduled for poster presentations.

The organisers hope that you will have a wonderful time at Kasteel Vaalsbroek and the chance to discuss with new collaborators and meet old friends.

Rafal Lupinski-Borkowski

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PROGRAMME

EMS Extension

PICO 2022 Conference Programme 8 – 12 May 2022

Time	Sunday 8 May 2022	Time	Monday 9 May 2022	Time	Tuesday 10 May 2022		Time	Wednesday 11 May 2022	Time	Thursday 12 May 2022
		8:30	Krivanek	8:30	Fraser	Wright	8:30	Urban	8:30	Jian Min Zuo
		9:05	Hartel	9:05	Banerjee	Mattei	9:00	Allen	9:05	Koch
		9:40	Ropers	9:40	Holmestad	Stahlberg	9:45	Olsson	9:40	Barthel
		10:15	Coffee Break	10:15	Coffee Break		10:30	Coffee Break	10:15	Coffee Break
		10:45	Van Aken	10:45	Xiaodong Han	Sandblad	11:00	Stark	10:45	Haigh
		11:20	Ramasse	11:20	Kaplan	Jakobi	11:45	Ze Zhang	11:20	Basak
		11:55	Lubk	11:55	Raabe	Efremov			11:55	Rose
12:00	CONFERENCE REGISTRATION	12:30	Lunch	12:30	Lunch		12:30	Lunch	12:30	Closing Remarks
15:00	HOTEL CHECK-IN	14:00	Peijun Zhang	14:00	Stephan	Kukulski	14:00	Xiaoyan Zhong	12:45	Lunch
		14:35	Russo	14:35	Botton	Marlovits	14:45	Sinclair		
15:15	Welcome Reception	15:10	Ravelli	15:10	Suenaga	Mann	15:30	Muller	14:00	DEPARTURE
		15:45	**Commercial presentation** Thermo Fisher Scientific	15:45	**Commercial presentation** JEOL					
16:00	Opening Remarks	16:00	Coffee Break	16:00	Coffee Break		16.15	Coffee Break		
16:15	Nellist	16:30	Valpuesta	16:30	Verbeeck	Tacke	16:45	Lei Jin		
16:50	Van Aert	17:05	Plitzko	17:05	Zweck	Wolf	17:30	Kaiser		
17:25	Zaluzec	17:40	Peiyi Wang	17:40	Kirkland	Schröder				
18:00	EMS Outstanding Paper Award	18:15	**Trade Pitches** DENSsolutions Ametek JEOL	18:15	**TradeD Pitches** Protochips Quantifoil NanoMEGAS & EMSIS		18:15	**Trade Pitches** Hitachi DECTRIS Quantum Detectors		
		18:30	Dinner	18:30	Dinner		19:00	Wine Reception Dinner Speech Lambrecht		
19:00	Dinner	20:00	**Poster Session A & B**	20:00	**Poster Session C & D**		20:00	**Conference Dinner**		

SUNDAY MAY 8TH, 2022

12:00 – 16:00	CONFERENCE REGISTRATION "Woonkamer" castle ground floor
15:00 – 23:00	HOTEL CHECK-IN hotel reception desk
15:15 – 16:00	WELCOME RECEPTION "Binnenplaats" in front of the castle

SESSION A
"Morettipaviljoen" castle ground floor

16:00 – 16:15	**A0**	OPENING REMARKS Rafal Dunin-Borkowski, Forschungszentrum Jülich GmbH (Germany), Joachim Mayer, RWTH Aachen University (Germany), and Carsten Sachse, Forschungszentrum Jülich GmbH (Germany)
16:15 – 16:50	**A1**	2D AND 4D STEM IMAGING OF BEAM SENSITIVE MATERIALS Peter Nellist, University of Oxford (UK)
16:50 – 17:25	**A2**	NOVEL METHODS TO QUANTIFY THE ATOMIC STRUCTURE IN 2D AND 3D FROM STEM DATA THROUGH THE COMBINATION OF STATISTICAL PARAMETER ESTIMATION AND DEEP LEARNING Sandra Van Aert, University of Antwerp (Belgium)
17:25 – 18:00	**A3**	X-RAY SPECTROMETRY IN THE ERA OF ABERRATION CORRECTED ELECTRON-OPTICAL BEAM LINES Nestor Zaluzec, Argonne National Laboratory (USA)

SESSION EMS **OUTSTANDING PAPER AWARD CEREMONY**
"Morettipaviljoen" castle ground floor

18:00 – 18:10	**EMS0**	INTRODUCTION Randi Holmestad, Norwegian University of Science and Technology (Norway)
18:10 – 18:25	**EMS1**	CATEGORY: INSTRUMENTATION AND TECHNIQUE DEVELOPMENT Emma Silvester, University of Oxford (UK)
18:25 – 18:40	**EMS2**	CATEGORY: LIFE SCIENCE Rhian Jones, Aix-Marseille Université (France)
18:40 – 18:55	**EMS3**	CATEGORY: MATERIALS SCIENCE Daniel Wolf and Sebastian Schneider, Leibniz Institute for Solid State and Materials Research (Germany)

19:00 – 20:30 DINNER
"Kruidentuin" restaurant

MONDAY MAY 9TH, 2022

07:00 – 08:30 BREAKFAST
"Kruidentuin" restaurant

SESSION B
"Morettipaviljoen" castle ground floor

08:30 – 09:05 **B1** PROGRESS IN ULTRA-HIGH ENERGY RESOLUTION EELS

Ondrej Krivanek, Nion Co. (USA)

09:05 – 09:40 **B2** EXPLOITING THE FULL POTENTIAL OF STATE-OF-THE-ART ABERRATION CORRECTORS

Peter Hartel, CEOS GmbH (Germany)

09:40 – 10:15 **B3** INTERFACING PHOTONICS WITH ELECTRON MICROSCOPY

Claus Ropers, MPI for Multidisciplinary Sciences (Germany)

10:15 – 10:45 COFFEE BREAK

10:45 – 11:20 **B4** ATOMIC-SCALE INVESTIGATIONS OF ELECTRON-BEAM-SENSITIVE MOLECULES

Peter van Aken, Max Planck Institute for Solid State Research, (Germany)

11:20 – 11:55 **B5** ATOMIC AND ELECTRONIC STRUCTURE OF HETERO-INTERFACES USING DARK-FIELD EELS

Quentin Ramasse, University of Leeds (UK)

11:55 – 12:30 **B6** EXPERIMENTAL OBSERVATION OF CHIRAL MAGNETIC TEXTURES BY ELECTRON HOLOGRAPHY AND TOMOGRAPHY

Axel Lubk, Leibniz Institute for Solid State and Materials Research Dresden (Germany)

EMS Extension

12:30 – 14:00 LUNCH
"Kruidentuin" restaurant

SESSION C
"Morettipaviljoen" castle ground floor

14:00 – 14:35 **C1** IN SITU STRUCTURES OF NATIVE PROTEINS AT NEAR-ATOMIC RESOLUTION BY CRYO-ELECTRON TOMOGRAPHY

Peijun Zhang, University of Oxford (UK)

14:35 – 15:10 **C2** THE POTENTIAL OF ELECTRON CRYOMICROSCOPY IN SITU: IDENTIFYING MOLECULES IN CELLS

Christopher Russo, MRC Laboratory of Molecular Biology (UK)

15:10 – 15:45 **C3** VITRIFYING CELLS

Raimond Ravelli, University of Maastricht (NL)

15:45 – 16:00 **ST1** TOWARDS MAKING EVERY ELECTRON COUNT

Eric Van Cappellen, Thermo Fisher Scientific (USA)

16:00 – 16:30 COFFEE BREAK

16:30 – 17:05 **C4** STRUCTURAL MECHANISM FOR TYROSINE HYDROYLASE INHIBITION BY DOPAMINE AND REACTIVATION BY SER40 PHOSPHORYLATION

José Valpuesta, Centro Nacional de Biotecnología (Spain)

17:05 – 17:40 **C5** IS THE CRYOFIB LIFT-OUT PROCEDURE REALLY READY FOR LIFT-OFF?

Jürgen Plitzko, Max Planck Institute of Biochemistry (Germany)

17:40 – 18:15 **C6** APPLICATION OF CHROMATIC ABERRATION CORRECTOR FOR CRYO-EM IN THE STRUCTURAL BIOLOGY

Peiyi Wang, Southern University of Science & Technology (China)

18:15 – 18:20	**SP1**	STREAM - MEMS-BASED LIQUID PHASE ELECTRON MICROSCOPY: A PROMISING ROUTE FOR MATERIAL SCIENCE, ENERGY STORAGE & LIFE SCIENCE APPLICATIONS Eva Bladt, DENSsolutions BV (NL)
18:20 – 18:25	**SP2**	ENERGY-FILTERED 4D STEM FOR ALL KVs Saleh Gorji, Ametek GmbH (Germany)
18:25 – 18:30	**SP3**	EXCEPTIONAL CONTROL OVER THE DOSE ON YOUR SAMPLE – JEOL-IDES EDM IS THE KEY Manuel Reinhard, JEOL (Germany) GmbH (Germany)
18:30 – 20:00		DINNER "Kruidentuin" restaurant

PLEASE NOTE THAT POSTER SESSIONS A & B WILL RUN IN PARALLEL

POSTER SESSION A	**MATERIALS SCIENCE** **"Vaalsbroekerhof" castle ground floor**
20:00 – 22:00	See Poster Program
POSTER SESSION B	**ELECTRON OPTICS** **"Jachtkelder" castle ground floor**
20:00 – 22:00	See Poster Program

EMS Extension

TUESDAY MAY 10TH, 2022

please note

- ✓ sessions DA & DB will run in parallel
- ✓ sessions EA & EB will run in parallel

07:00 – 08:30 BREAKFAST
"Kruidentuin" restaurant

SESSION DA
"Morettipaviljoen" castle ground floor

08:30 – 09:05 **DA1** USE OF PICO-TYPE MICROSCOPY TECHNIQUES TO OPTIMIZING COMPOSITION AND MICROSTRUCTURE IN COMPOSITIONALLY COMPLEX ALLOYS

Hamish Fraser, Ohio State University (USA)

09:05 – 09:40 **DA2** CRYSTALLOGRAPHIC AND COMPOSITIONAL EVOLUTION DURING ISOTHERMAL ANNEALING OF REFRACTORY HIGH ENTROPY ALLOYS: INSIGHTS INTO HIGH TEMPERATURE PHASE STABILITY

Rajarshi Banerjee, University of North Texas (USA)

09:40 – 10:15 **DA3** ANALYSIS OF PRECIPITATES IN ALUMINIUM ALLOYS FROM DIRECT DETECTION SPED DATA

Randi Holmestad, Norwegian University of Science and Technology (Norway)

10:15 – 10:45 COFFEE BREAK

10:45 – 11:20 **DA4** MEASURING GRAIN/TWIN BOUNDARY PLASTICITY AT ATOMIC LEVEL

Xiaodong Han, Beijing University of Technology, (China)

11:20 – 11:55 **DA5** MICROSCOPIC DEGREES OF FREEDOM OF GRAIN BOUNDARIES AND MICROSTRUCTURAL EVOLUTION

Wayne Kaplan, Technion – Israel Institute of Technology (Israel)

11:55 – 12:30 **DA6** MECHANISMS BEHIND HYDROGEN-BASED GREEN STEEL MAKING

Dierk Raabe, Max-Planck Institut für Eisenforschung (Germany)

12:30 – 14:00 LUNCH
"Kruidentuin" restaurant

SESSION DB
"Vaalsbroekerhof" castle ground floor

08:30 – 09:05 **DB1** STRUCTURAL STUDIES OF VIRUSES BY CORRELATIVE CRYO-MICROSCOPY

Elizabeth Wright, University of Wisconsin-Madison (USA)

09:05 – 09:40 **DB2** EASY-GRID: ADVANCING SAMPLE PREPARATION FOR CRYO-EM

Simone Mattei, European Molecular Biology Laboratory (Germany)

09:40 – 10:15 **DB3** ULTRASTRUCTURAL INVESTIGATIONS OF SYNUCLEINOPATHIES: PD, MSA, HUMAN BRAIN, AND FIBRIL STRAINS

Henning Stahlberg, University of Basel (CH)

10:15 – 10:45 COFFEE BREAK

10:45 – 11:20 **DB4** BACTERIA CYTOSKELETON VISUALIZATION BY CRYO ELECTRON TOMOGRAPHY AND CORRELATIVE APPROACHES

Linda Sandblad, Umeå University, (Sweden)

11:20 – 11:55 **DB5** MEMS TECHNOLOGY FOR CRYO-EM SAMPLE PREPARATION

Arjen J. Jakobi, Delft University of Technology (NL)

11:55 – 12:30 **DB6** RESOLVING STRUCTURAL DYNAMICS OF PROTEINS USING DROPLET MICROFLUIDICS

Rouslan Efremov, Vlaams Instituut voor Biotechnologie (Belgium)

12:30 – 14:00 LUNCH
"Kruidentuin" restaurant

SESSION EA
"Morettipaviljoen" castle ground floor

14:00 – 14:35 **EA1** EVENT-BASED SPECTROSCOPY IN A STEM: FROM NANOSECOND RESOLUTION TO TIME-CORRELATED HYPERSPECTRAL IMAGING

Odile Stéphan, Université Paris-Saclay (France)

14:35 – 15:10 **EA2** ELECTRON ENERGY LOSS SPECTROSCOPY WITH HIGH SPATIAL RESOLUTION: APPLICATIONS TO CATHODE MATERIALS IN LI-ION BATTERIES

Gianluigi Botton, McMaster University (Canada)

15:10 – 15:45 **EA3** RECENT PROGRESS OF ELECTRON SPECTROSCOPY WITH THE SINGLE-ATOM LIMIT

Kazu Suenaga, Osaka University (Japan)

15:45 – 16:00 **ST2** OPTIMUM BF (OBF) STEM USING THE SAAF DETECTOR

Philipp Wachsmuth, JEOL (Germany) GmbH (Germany)

16:00 – 16:30 COFFEE BREAK

16:30 – 17:05 **EA4** WAVEFRONT SHAPING IN THE ELECTRON MICROSCOPY WITH A 48 PIXEL PROGRAMMABLE PHASE PLATE

Johan Verbeeck, University of Antwerp (Belgium)

17:05 – 17:40 **EA5** ADVANCED PROCESSING OF DIFFERENTIAL PHASE CONTRAST DATA

Josef Zweck, Universität Regensburg (Germany)

17:40 – 18:15 **EA6** PROSPECTS AND OPPORTUNITIES FOR ELECTRON PTYCHOGRPAPHY AT LOW DOSE

Angus Kirkland, Oxford University (UK)

18:15 – 18:20 **SP4** AXON: AN IN-SITU TEM SOFTWARE PLATFORM STREAMLINES IMAGE ACQUISITION, METADATA SYNCHRONIZATION AND DATA ANALYSIS, ENABLING DEEPER UNDERSTANDING, AND IMPROVED REPRODUCIBILITY OF IN-SITU EXPERIMENTAL RESULTS

David Nackashi, Protochips Inc. (USA)

18:20 – 18:25 **SP5** IMPROVING SAMPLE DISPERSION WITH AN ADDITIONAL ULTRATHIN CARBON LAYER

Claire Naylor, Quantifoil (Germany)

18:25 – 18:30 **SP6** DYNAMICAL STUDIES OF ADVANCED MATERIALS COMBINING IN SITU 4D-sPED MAPPING AND PIXELATED DETECTORS

Alan Robins, NanoMEGAS SPRL (Belgium)

18:30 – 20:00 DINNER
"Kruidentuin" restaurant

SESSION EB
"Vaalsbroekerhof" castle ground floor

14:00 – 14:35 **EB1** THE ARCHITECTURE OF ORGANELLE CONTACT SITES

Wanda Kukulski, University of Bern (CH)

14:35 – 15:10 **EB2** SECRETION SYSTEMS IN ACTION

Thomas Marlovits, Institute for Structural and Systems Biology (Germany)

15:10 – 15:45 **EB3** MACROMOLECULAR ORGANIZATION OF ATG18 OLIGOMERS

Daniel Mann, Forschungszentrum Jülich (Germany)

15:45 – 16:00 **ST2** PRESENTATION JEOL IN MORETTIPAVIJOEN

16:00 – 16:30 COFFEE BREAK

16:30 – 17:05 **EB4** KEEP IT CLEAN: AN IMPROVED CRYO-FIB WORKFLOW

Sebastian Tacke, Max Planck Institute of Molecular Physiology (Germany)

17:05 – 17:40 **EB5** TEMPORAL DYNAMICS OF CHARGE BUILD IN CRYO-ELECTRON MICROSCOPY

Matthias Wolf, Okinawa Institute of Science and Technology (Japan)

17:40 – 18:15 **EB6** AMYLOID FIBRILS AND THE INTERPRETATION OF COFACTOR DENSITIES

Gunnar Schröder, Forschungszentrum Jülich (Germany)

18:15 – 18:30 **SP** TRADE PITCHES IN MORETTIPAVIJOEN

18:30 – 20:00 DINNER
"Kruidentuin" restaurant

PLEASE NOTE THAT POSTER SESSIONS C & D WILL RUN IN PARALLEL

POSTER SESSION C **METHOD DEVELOPMENT & DATA SCIENCE / THEORY**
"Vaalsbroekerhof" castle ground floor

20:00 – 22:00 See Poster Program

POSTER SESSION D **LIFE SCIENCE**
"Jachtkelder" castle ground floor

20:00 – 22:00 See Poster Program

EMS Extension

WEDNESDAY MAY 11TH, 2022

'SPECIAL EVENT FOR CELEBRATING 25 YEARS OF ABERRATION CORRECTION

07:00 – 08:30 BREAKFAST
"Kruidentuin" restaurant

SESSION F
"Morettipaviljoen" castle ground floor

08:30 – 09:00 **F1** 25 YEARS OF ABERRATION CORRECTION IN ELECTRON MICROSCOPY

Knut Urban, Forschungszentrum Jülich (Germany)

09:00 – 09:45 **F2** ABERRATION CORRECTION IN TANDEM WITH MODELLING FOR ATOMIC RESOLUTION STRUCTURE DETERMINATION

Leslie J. Allen, University of Melbourne (Australia)

09:45 – 10:30 **F3** PROGRESS IN ABERRATION-CORRECTED ELECTRON MICROSCOPY IN MATERIALS SCIENCE

Eva Olsson, Chalmers University of Technology (Sweden)

10:30 – 11:00 COFFEE BREAK

11:00 – 11:45 **F4** SPHERICAL ABERRATION CORRECTION FOR HIGH-RESOLUTION PROTEIN STRUCTURE DETERMINATION: WHAT IS THE BENEFIT?

Holger Stark, Georg-August-Universität Göttingen, (Germany)

11:45 – 12:30 **F5** IN-SITU HREM STUDIES OF NANOCATALYSTS AND FERROELECTRIC DOMAINS UNDER ENVIRONMENTAL CONDITIONS

Ze Zhang, Zhejiang University (China)

12:30 – 14:00 LUNCH
"Kruidentuin" restaurant

EMS Extension

SESSION G
"Morettipaviljoen" castle ground floor

14:00 – 14:45	**G1**	MAGNETIC DICHROISM INVESTIGATED IN THE CS/CC CORRECTED ELECTRON MICROSCOPE Xiaoyan Zhong, City University of Hong Kong (China)
14:45 – 15:30	**G2**	IMPORTANCE OF ABERRATION CORRECTION FOR IN SITU HIGH RESOLUTION TEM Robert Sinclair, Stanford University (USA)
15:30 – 16:15	**G3**	IMAGING THE PROPERTIES OF ATOMS AND FIELDS AT THE PICOMETER SCALE INSIDE MATERIALS AND DEVICES David Muller, Cornell University (USA)
16:15 – 16:45		COFFEE BREAK
16:45 – 17:30	**G4**	QUANTITATIVE HIGH-RESOLUTION TRANSMISSION ELECTRON MICROSCOPY OF ENERGY CONVERSION MATERIALS Lei Jin, Forschungszentrum Jülich (Germany)
17:30 – 18:15	**G5**	UNDERSTANDING ELECTRON-BEAM-SPECIMEN INTERACTIONS AT LOW ACCELERATING VOLTAGES TO TAILOR PROPERTIES OF LOW-DIMENSIONAL INORGANIC AND ORGANIC MATERIALS Ute Kaiser, Ulm University (Germany)
18:15 – 18:20	**SP7**	LATEST DEVELOPMENTS FROM HITACHI Felix von Cube, Hitachi High-Tech Europe GmbH (Germany)
18:20 – 18:25	**SP8**	LET'S DESIGN TOGETHER YOUR NEXT 4D STEM EXPERIMENT Daniel Stroppa, DECTRIS Ltd. (CH)
18:25 – 18:30	**SP9**	PTYCHOGRAPHY WITH MERLIN EM DETECTOR Matus Krajnak, Quantum Detectors Ltd. (UK)

19:00 – 20:00	WINE RECEPTION & DINNER SPEECH ASTRID LAMBRECHT “Binnenplaats” in front of the castle
20:00 – 22:00	CONFERENCE DINNER castle – see seating plan

EMS Extension

THURSDAY MAY 12TH, 2022

07:00 – 08:30 BREAKFAST
"Kruidentuin" restaurant

SESSION H
"Morettipaviljoen" castle ground floor

08:30 – 09:05 **H1** CEPSTRAL SCANNING TRANSMISSION ELECTRON MICROSCOPY FOR ORDERED AND DISORDERED MATERIALS

Jian Min Zuo, University of Illinois (USA)

09:05 – 09:40 **H2** EXPLORING MATERIALS STRUCTURE AND PROPERTIES ON THE NANOSCALE BY COMPUTER-AUGMENTED ELECTRON MICROSCOPY

Christoph Koch, Humboldt-Universität zu Berlin (Germany)

09:40 – 10:15 **H3** ROLE OF IONIZATION IN IMAGING AND SPECTROSCOPY UTILIZING FAST ELECTRONS THAT HAVE EXCITED PHONONS

Juri Barthel, Forschungszentrum Jülich (Germany)

10:15 – 10:45 COFFEE BREAK

10:45 – 11:20 **H4** 2D MATERIAL HETEROSTRUCTURE LIQUID CELLS: A PLATFORM FOR ATOMIC RESOLUTION STEM IMAGING WITH LIQUIDS

Sarah Haigh, University of Manchester, (UK)

11:20 – 11:55 **H5** THICKNESS DEPENDENT COATING BREAKING DURING BATTERY CYCLING BY IN SITU TEM

Shibabrata Basak, Forschungszentrum Jülich (Germany)

11:55 – 12:30 **H6** NOVEL THEORY OF THE STRUCTURE OF THE ELECTRON

Harald Rose, Ulm University (Germany)

12:30 – 12:45	CLOSING REMARKS Rafal Dunin-Borkowski, Forschungszentrum Jülich GmbH (Germany), Joachim Mayer, RWTH Aachen University (Germany), and Carsten Sachse, Forschungszentrum Jülich GmbH (Germany)
12:45 – 14:00	LUNCH “Kruidentuin” restaurant
14:00	DEPARTURE

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ABSTRACTS

EMS Extension

2D AND 4D STEM IMAGING OF BEAM SENSITIVE MATERIALS

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The development and widespread application of aberration-corrected electron microscopy over the past 25 years has made atomic resolution imaging and spectroscopy of materials routine. Many candidate materials for important applications in energy conversion and energy storage are highly susceptible to damage under the irradiating electron beam and so an important current challenge is to develop methods that can allow the high-spatial resolution advantages of aberration correction to be applied to these materials. Here the use of the aberration-corrected scanning transmission electron microscope (AC-STEM) for such applications will be described. It will be shown that the dose fractionation in both space and time enable by AC-STEM enables atomic resolution images to be formed for materials that hitherto have eluded such imaging. Rapidly scanned annular dark-field (ADF) will be used to analyse defects in the organic-inorganic hybrid perovskite material, formamidinium lead iodide. It is shown that the remnant lead iodide precursor material is stabilized in an uncommonly observed trigonal structure that forms highly coherent interfaces with the cubic perovskite [1]. Combining ADF imaging and ptychography provides a method that makes use of a very large fraction of the transmitted electrons. Careful choice of imaging conditions is required to enable the most dose-efficient imaging [2]. This approach will be demonstrated by application to defects in lithium-rich cathode materials for batteries and to the structure of crystalline domains in polymer materials. In both cases, the location of the light elemental species can be directly observed [3].

References:
[1] M.U. Rothmann, et al., *Science*, 370, eabb5940 (2020).
[2] C.M. O'Leary, et al., *Ultramicroscopy*, 221, 113189 (2021).
[3] The author acknowledges support from JEOL UK, PNDetector, and funding from the EPSRC (EP/K040375/1, EP/M010708/1, EP/P033229/1), The Henry Royce Institute (Grant reference EP/R010145/1), EU 823717 ESTEEM3.

NOVEL METHODS TO QUANTIFY THE ATOMIC STRUCTURE IN 2D AND 3D FROM STEM DATA THROUGH THE COMBINATION OF STATISTICAL PARAMETER ESTIMATION AND DEEP LEARNING

Sandra Van Aert[1*], Ece Arslan Irmak[1], Thomas Friedrich[1], Gizem Sentürk[1], Chu-Ping Yu[1], Annick De Backer[1], Annelies De wael[1], Ivan Lobato[1], Zezhong Zhang[1], Lewys Jones[2], Peter D. Nellist[3], Sara Bals[1]

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Determining the atomic structure of nanomaterials in 2D and 3D is critical to understand their unique properties. Therefore, a thorough quantitative characterization by TEM is of great importance. Recent progress in the development of quantitative methods allows us to extract reliable structural and chemical information from experimental STEM images. In quantitative STEM, images are treated as datasets from which structure parameters are determined by comparison with image simulations, by using parameter estimation-based methods, and more recently by applying deep learning. This contribution aims to explain recent developments using current state-of-the-art experimental examples. To overcome limitations related to conventional electron tomography, 3D characterization of beam-sensitive nanomaterials can be obtained from atom counts resulting from a single ADF STEM image. For this purpose, the scattering cross-section (SCS), corresponding to the total intensity of electrons scattered by a single atomic column, has been shown to be successful. When combining image simulations and statistical parameter estimation [1], the number of atoms in an atomic column can be counted. This method has recently been extended to analyze time series of images using a hidden Markov model [2] and is very promising for revealing dynamic structural changes resulting during *in situ* experiments [3]. Moreover, progress has been made to extend atom counting from homogeneous to heterogeneous materials by combining ADF STEM with EDX. This is facilitated by using a deep convolutional neural network and an atomic lensing model to efficiently compute SCSs of mixed atomic columns. Next, atom counts can be used to create an initial atomic model which serves as an input for energy minimization to obtain a relaxed 3D reconstruction of the nanostructure. New developments using a Bayesian genetic algorithm will be proposed as well as a methodology to characterize supported metallic nanoparticles at high temperature [4]. Finally, the information-richness and dose efficiency of 4D STEM is explored through the analysis of multiple virtual STEM images with freely chosen inner and outer angles and through phase reconstruction using deep learning.

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X-RAY SPECTROMETRY IN THE ERA OF ABERRATION CORRECTED ELECTRON-OPTICAL BEAM LINES

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Aberration Correction in the Analytical Transmission Electron Microscope (AEM) is most closely associated with improvements in high resolution imaging. In this paper the combination of that technology with new systems designs which optimize both electron optics and x-ray detection is discussed. Analytical performance has increased more than 10-fold since inception (Figure 1) and the sensitivity correspondingly increases with decreasing incident beam energy (Figure 2) both of which complement the advances in electron-optical performance achieved with modern aberration correctors over the last 25 years. [1]

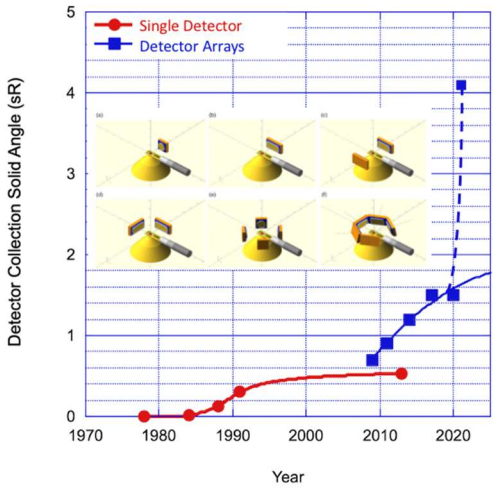

Fig. 1 Experimental X-ray detector collection solid angle in the AEM as a function of year.

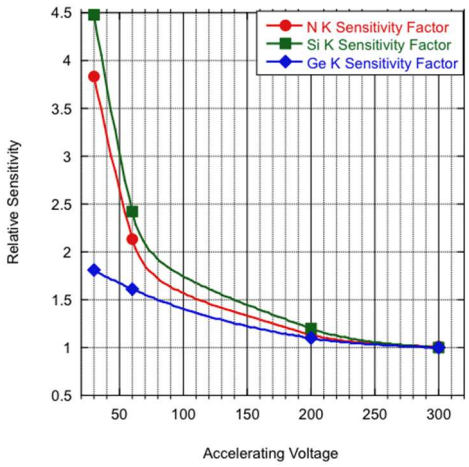

Fig. 2. Experimental relative sensitivity as a function of incident beam energy – normalized at 300 kV

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PROGRESS IN ULTRA-HIGH ENERGY RESOLUTION EELS

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Our ultra-**H**igh **E**nergy **R**esolution **M**onochromated **E**ELS–**S**TEM (HERMES™; EELS=Electron Energy Loss Spectroscopy, STEM=scanning transmission electron microscope) uses aberration-corrected optics operating at ground potential, and has achieved < 3 meV energy resolution (Fig. 1). It has opened up a wide range of applications, including: direct observation of atomic vibrations (phonons) in the electron microscope [1]; spatially-resolved studies of phonons and phonon-polaritons in 3D objects; separation of dipole and non-dipole vibrational signals, with the dipole signal able to provide damage-free analysis, especially useful in biological systems, and the non-dipole signal able to detect the vibrations of a single atom [2]; detection and spatial mapping of different isotopes such as those of carbon [3]; and angle-resolved mapping of phonon dispersion surfaces (Fig. 2). These and other applications will be shown at the meeting, and future directions discussed.

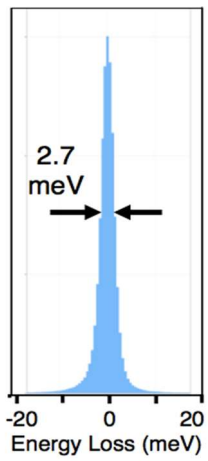

Fig.1. Zero Loss Peak (ZLP) recorded by Nion HERMES. 30 kV, 0.9 s acquisition (300 aligned spectra of 3 ms each).

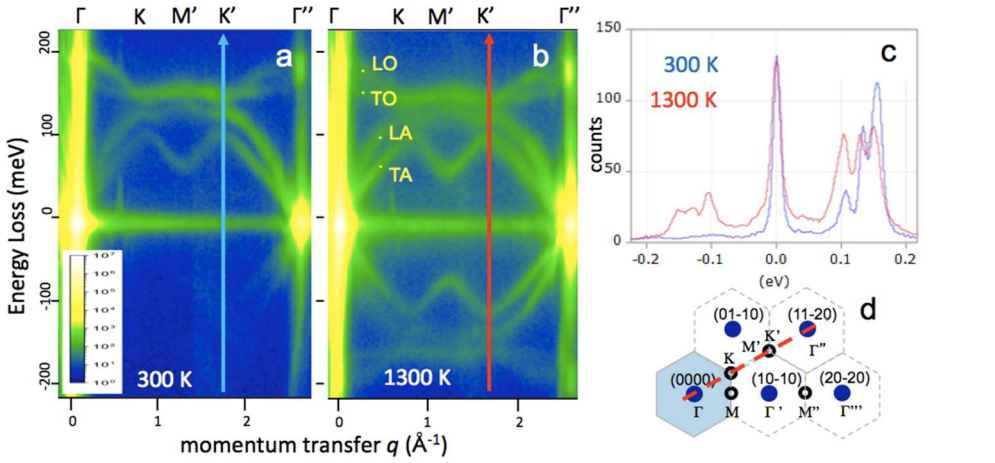

Fig.2. Experimental q-Ω (momentum-energy) patterns of hexagonal boron nitride (h-BN). Nion HERMES, 30 kV, 20 min acq. time, using a pre-EELS entrance slot. (a) q-Ω pattern at 300 K, (b) pattern at 1300 K, (c) energy profiles at K' point, (d) schematic showing the diffraction pattern, the principal Brillouin zone points, and the location of the EELS entrance slot aperture.

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EXPLOITING THE FULL POTENTIAL OF STATE-OF-THE-ART ABERRATION CORRECTORS

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The success story of hardware aberration correction in transmission electron microscopy started 25 years ago with the first resolution improvements achieved with a two-hexapole design in TEM [1] and a quadrupole-octupole design in STEM [2]. The two-hexapole design was sucessfully used as well in STEM later [3]. Within the TEAM project a careful redesign lead to the advanced two-hexapole corrector (DCOR/ASCOR) in STEM with full fourth-order correction and effectively vanishing fifth-order aberrations [4]. This design is still state-of-the-art – regardless whether additional A5 stigmators are used or not [5, 6]. For TEM, further development steps concentrated on off-axial aberration correction (BCOR) [7] and simultaneous chromatic aberration correction (CCOR, SALVE) [8, 9], while the introduction of the advanced two-hexapole corrector (ATCOR) [10] is still in progress. In order to fully exploit the potential of the aberration correctors an accurate optimization of the experimental imaging conditions is required. In STEM special care has to be taken for the choice of the optimum aperture semi-angle depending on the desired high tension and of the present chromatic aberration. Due to the availability of cold field-emission sources and monochromators, the chromatic limit becomes more relaxed. As a consequence, axial aberrations beyond fifth order have to be considered in measurement routines and in compensation schemes for aberration coefficients of different order but same multiplicity.

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INTERFACING PHOTONICS WITH ELECTRON MICROSCOPY

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Providing the most detailed views of atomic-scale structure and composition, Transmission Electron Microscopy (TEM) serves as an indispensable tool for structural biology and materials science. Optical excitations in electron microscopy are accessible through spontaneous inelastic scattering of electrons, analysed in electron-energy loss and cathodoluminescence spectroscopy. The stimulated variants of the underlying scattering processes become accessible through optical illumination of the sample.

In this talk, I will introduce basic principles and selected applications of inelastic electron-light scattering in electron microscopy. These include the imaging of near-field intensity distributions [1] and Lorentz microscopy of optical fields [2], as well as the coherent control of the free-electron quantum state for spatial [3] and temporal [4-6] electron beam manipulation.

Moreover, recent progress in the coupling of electron beams to whispering gallery modes [7] and integrated photonic resonators [8] will be discussed, including the preparation and characterization of electron-photon pair states [9].

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ATOMIC-SCALE INVESTIGATIONS OF ELECTRON-BEAM-SENSITIVE MOLECULES

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Formation and characterization of low-dimensional nanostructures is crucial for controlling the properties of two-dimensional (2D) materials such as graphene. In this work, we have studied the structure of low-dimensional adsorbates of cesium iodide (CsI) on freestanding graphene at atomic resolution. CsI is deposited onto graphene as charged clusters by electrospray ion-beam deposition (ES-IBD). The interaction with the electron beam forms two-dimensional CsI crystals only on bilayer graphene (BLG), while CsI clusters consisting of 4, 6, 7, and 8 ions are exclusively observed on single-layer graphene (SLG). Chemical characterization by EELS mapping and precise structural measurements evidence the possible influence of charge transfer on the structure formation of the CsI clusters and layers[1].

Atomic design of graphene can be substantially influenced by etching, deliberately induced in a transmission electron microscope. It is achieved primarily by overcoming the threshold energy for defect formation by controlling the kinetic energy and current density of the fast electrons. Recent studies have demonstrated that the presence of certain atomic species can catalyze atomic bond dissociation processes under the electron beam by reducing their threshold energy. This work shows by experimentally comparing the interaction of alkali and halide species separately and conjointly with SLG that in the presence of electron irradiation, etching of SLG is drastically enhanced by the simultaneous presence of alkali and iodine atoms. Density functional theory and first-principles molecular dynamics calculations reveal that due to charge-transfer phenomena the C–C bonds weaken close to the alkali-iodide species, which increases the carbon displacement cross-section[2,3].

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ATOMIC AND ELECTRONIC STRUCTURE OF HETERO INTERFACES USING DARK-FIELD EELS

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Engineering the structural or chemical architecture of functional materials at the nano or even atomic level enables emergent properties that rely on the interplay between fundamental properties of matter such as charge, spin and local atomic-scale chemistry. A striking illustration of the relevance of this strategy is provided by placing Bi_2Se_3, a topological insulator (TI) with topologically-protected helical two-dimensional surface states and one-dimensional bulk states associated with crystal defects, in close proximity with graphene. The strong spin-orbit interaction and proximity effects result in subtle and controllable electronic band structure changes at and near the interface, with exciting potential for spintronic applications. Here we probe at high energy resolution the interfaces in a system consisting of Bi_2Se_3 films grown by chemical vapor deposition on epitaxial graphene/SiC(0001), where the number of carbon layers can be carefully controlled to tune possible proximity effects between the film and the substrate. All experiments were carried out on a monochromated Nion UltraSTEM100 MC operated at 60kV, with a probe convergence semi-angle of 31mrad and a beam current of approximately 4pA (after monochromation to ~12meV resolution). Chemical mapping confirms the atomic-level chemistry of the layers, while an analysis of the carbon *K* edge fine structure provides direct insights into the nature of bonding at the SiC/graphene and graphene/Bi_2Se_3 interfaces [1]. Strikingly, the use of a dark-field EELS geometry, either using a set of custom-developed annular apertures reveals the emergence of locally resolved fine structure in the ultra-low loss region of the spectrum. In addition to a further direct interrogation of the chemical bonds between the layers, and a demonstration of chemically and atomically sensitive phonon mapping at interfaces in a bulk sample, it is thought these observations could also be linked to the interplay between the various phonon modes and the Dirac plasmons in the TI layers.

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EXPERIMENTAL OBSERVATION OF CHIRAL MAGNETIC TEXTURES BY ELECTRON HOLOGRAPHY AND TOMOGRAPHY

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Chiral magnetic micro- and nanotextures, such as chiral domain walls or skyrmions, are patterns in magnetically ordered materials that arise from the (frustrated) interplay of magnetic stray field, symmetric exchange, antisymmetric Dzyaloshinskii-Moriya exchange, and magnetocrystalline anisotropy, and thus being strongly dependent on the geometry of a specimen and intrinsic material parameters. They are currently intensively studied for their fundamental properties such as relation between stability and topology as well as possible applications in magnetic memory and logic devices. Here we report on combination of off-axis electron holography (EH) and electron tomography (ET) applied in the TEM facilitating 3D characterization of these magnetic configurations down to nanometer length scales. We discuss the experimental details of vector field electron tomography (VFET) and the information content of the reconstructed 3D magnetic induction at the example of three topical chiral textures: 3D magnetic chiral domain walls in CoNi nanowires with large transversal magnetocrystalline anisotropy [1], Bloch skyrmion tube lattices in FeGe [2] (Fig. 1), and vortex strings in curvilinear nanomagnets.

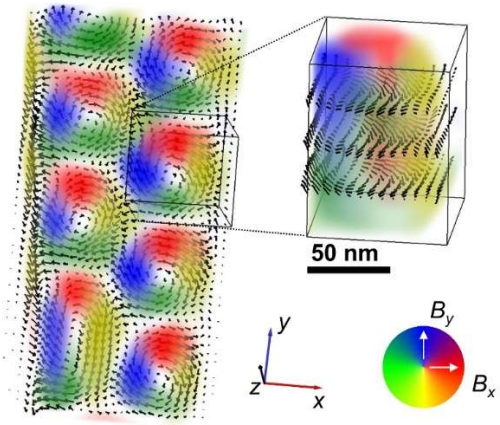

Fig. 1: Three-dimensional reconstruction of the magnetic induction B of a Bloch skyrmion tube lattice in a FeGe needle-shaped sample.

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[3] We acknowledge funding from European Research Council (ERC) (grant agreement number 715620).

IN SITU STRUCTURES OF NATIVE PROTEINS AT NEAR-ATOMIC RESOLUTION BY CRYO-ELECTRON TOMOGRAPHY

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Structural biology methods using cryoEM and cryo-electron tomography (cryoET) have become major tools for studying macromolecular complexes that are intrinsically flexible and dynamic, and often function in higher-order assemblies that are difficult to purify. The study of these complexes and assemblies *in situ* using cryoET and subtomogram averaging at sub-nanometer to near-atomic resolutions, coupled with cryoFIB and correlative and integrative imaging, opens a new frontier in structural cell biology [1], as exemplified in virus infection in human cells. I will present some of our recent studies on the SARS-Cov-2 virus infection processes [2], the structure and activity of particulate methane monooxygenase (pMMO) arrays in the intracytoplasmic membranes of methanotrophs, and the assembly and organization cargo Rubisco within native carboxysomes [3], to demonstrate the power of cryoET and subtomogram averaging for “in-cell” structural biology.

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THE POTENTIAL OF ELECTRON CRYOMICROSCOPY IN SITU: IDENTIFYING MOLECULES IN CELLS

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Electron cryomicroscopy (cryoEM) of biological specimens preserved by vitrification in water ice has made great strides in the last decade.The atomic structure of most biological macromolecules can, at least in principle, be determined by direct imaging using bright field phase contrast. Major technological advances – in electron imaging hardware, data analysis software, and cryogenic specimen preparation technology – continue at pace and contribute to the exponential growth in the number of atomic structures determined by cryoEM. It is now likely, that within a few years we will have structures for hundreds of thousands of unique protein and nucleic acid molecular complexes. But the answers to many important questions in biology would become obvious if we could identify these structures precisely inside cells with quantifiable error. In the context of an abundance of known structures, it is appropriate to now consider the current state of electron cryomicroscopy for frozen specimens prepared directly from cells, and try to understand what technology can be brought to bear on this goal, both now and in the foreseeable future.

VITRIFYING CELLS

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Cryogenic electron microscopy (cryo-EM) has become an indispensable tool for structural biology. Within less than a decade, the use of single particle analysis (SPA) techniques exploded. It has led to valuable insights in the structure and function of macromolecular complexes, crucial for the development of novel medicine and vaccines. Sample preparation is an essential step in cryo-EM. For SPA, macromolecules are purified, concentrated and trapped in a thin vitreous ice layer by a process called vitrification. Although such preparation method can result in near-atomic and even atomic resolution structures, inherent risks also became apparent. Isolated and sometimes detergent-solubilised molecules could alter their structure, and might lose their co-factors and function. Ideally, we could derive detailed 3D structures from macromolecular complexes within their native cellular context. Structural biology *in situ* is a term that applies to a scenario in which the cellular environment is entirely preserved. Cryo-FIB SEM allows for the production of thin lamella in such samples, which is followed by cryogenic electron tomography and sub-tomogram averaging. Such workflow critically depends on the availability of good, vitreous cellular samples. Traditionally, in order to achieve these, plunge freezing is used for 2-10 μm thick samples, slam freezing up to 20 μm, and high-pressure freezing for samples up to 200 μm. In here, we will present our novel jet-based cooling protocols, discuss its characteristics and potentials, and demonstrate some applications.

TOWARDS MAKING EVERY ELECTRON COUNT

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Ever since the invention of the electron microscope scientists have argued about how the electrons affect the sample. Life scientists have always looked at ways to increase contrast, and reduce beam damage, for improving productivity (throughput) and performance (resolution) when studying biological structures. Now materials science is commanding similar tactics. Many materials classes such as polymers, 2D materials, zeolites, MOF's and others require a frugal use of the electrons that have transited through the sample in order to minimize the damage they can cause. Here we'll list the recent developments but also the ongoing research and development projects at Thermo Fisher Scientific in the field of low-dose imaging and analysis. Figure 1 is a materials science example of very low-dose STEM imaging of a MOF and figure 2 shows a high-resolution structure of a sub 100kDa human hemoglobin protein. The images for this reconstruction were acquired with the latest generation Krios G4 equipped with a Selectris, an E-CFEG and a Falcon 4i direct electron detector camera.

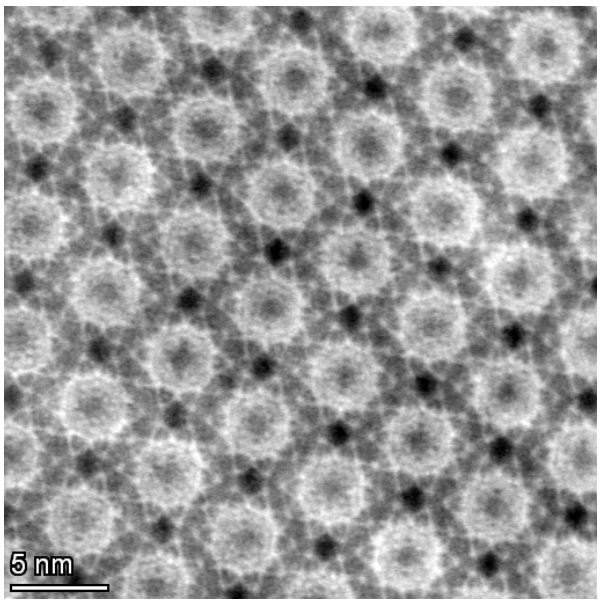

Fig. 1 200kV iDPC of a MOF (MIL 101). The total dose for this image is 42 $e^-/Å^2$
Sample courtesy: Prof. Han, KAUST.
Image by Anna Carlsson, Thermo Fisher Scientific.

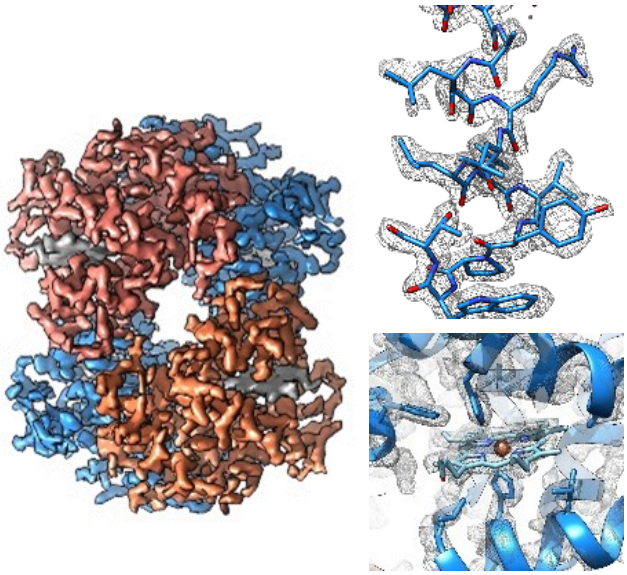

Fig. 2. 64kDa Human Hemoglobin protein @ 2.0Å resolution.
Image by Abhay Kotecha, Thermo Fisher Scientific.

STRUCTURAL MECHANISM FOR TYROSINE HYDROYLASE INHIBITION BY DOPAMINE AND REACTIVATION BY SER40 PHOSPHORYLATION

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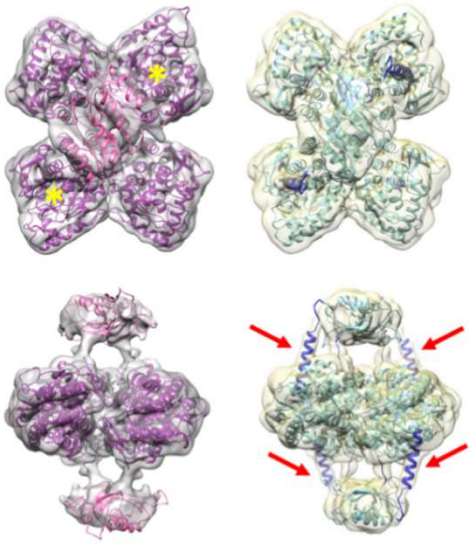
Tyrosine hydroxylase (TH) catalyzes the hydroxylation of L-tyrosine (L-Tyr) to L-3,4-dihydroxyphenylalanine (L-Dopa), the first and rate-limiting step in the synthesis of the catecholamines (CAs) dopamine (DA), noradrenaline and adrenaline [1]. In the brain, CAs are essential neurotransmitters and neuromodulators involved in processes such as motor control, emotion, reward, biorhythms and learning. Mutations in the TH gene are associated with congenital TH deficiency (THD), and a deficiency in striatal TH is a hallmark of Parkinson's disease [2]. Inhibition by CAs and reactivation by S40 phosphorylation are key regulatory mechanisms of TH activity and conformational stability. We have used cryoEM to determine the structures of full-length human TH without and with DA, and the structure of S40 phosphorylated TH, complemented with biophysical and biochemical characterizations and molecular dynamics simulations. TH presents a tetrameric structure with dimerized regulatory domains that are separated 15 Å from the catalytic domains (Fig. 1, left). Upon DA binding, a 20-residue □-helix in the flexible N-terminal tail of the regulatory domain is fixed in the active site, blocking it (red arrows in Fig. 1, right), while S40-phosphorylation forces its egress. The structures reveal the molecular basis of the inhibitory and stabilizing effects of DA and its counteraction by S40-phosphorylation, key regulatory mechanisms for homeostasis of DA and TH [3].



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IS THE CRYOFIB LIFT-OUT PROCEDURE REALLY READY FOR LIFT-OFF?

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Lift-out lamellar preparation is an established method in materials science, but since its introduction for cryo-ET [1] it has rather rarely been used, quite contrary to lamellar preparation on the grid, which has now become part of the cryoET-routine. So, one must ask: Why is this so? and provocatively - When will the cryoFIB lift-out method be ready for take-off?

Is that due to the sample? Well, larger samples, such as small organisms and tissues that need to be frozen under high pressure, are indeed the most exciting. This means that many are interested, and the number of adopters should be higher. So, we can't really blame it on the sample. Well, maybe partly, because high pressure freezing (HPF) and the resulting ice quality have their difficulties and peculiarities. And there is no way to assess the ice quality before further preparation steps are performed: Only after the first TEM image does one know whether the ice is "good" or "bad".

Is that due to the FIB/SEM-instrument? Now that lift-out routinely works in materials science, there should be nothing stopping it from being used for cryo-samples. However, extraction of lamellae is time-consuming, targeted FIB-SEM volume imaging or integrated CLEM are not yet widely available and unloading and loading of fragile samples is still manual and therefore largely user-dependent. The transfer success rate was originally less than 20% and is now 50% [2-4]. So, we can't really blame the instrument. Well, maybe partially, since material removal rates with gallium ions are low and using higher currents is compromised due to spherical aberration and the resulting larger spot sizes. Getting a grip on this would require novel hardware, e.g., plasma ion sources with higher ablation rates and currents to make even 200 μm HPF samples readily accessible. In any case, the whole procedure for the fabrication of lamellae from HPF samples, whether with gallium or plasma ions, needs to be streamlined and automated to become more efficient and user-friendly.

In our presentation, we report on the latest developments on the way to full automation of the lift-out process. We also are showcasing the integration of fluorescence light microscopes into the FIB chamber for fully automated correlative lamella preparation workflows [5]. Finally, perspectives and preliminary results are presented on improving (and possibly bypassing) lift-out to increase transfer efficiency and make cryoFIB lift-out of multicellular specimens ready for take-off.

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APPLICATION OF CHROMATIC ABERRATION CORRECTOR FOR CRYO-EM IN THE STRUCTURAL BIOLOGY

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Chromatic aberration (Cc) is presented as image blur on the Gaussian imaging plane of TEM caused by energy spread of the incident electron beam whilst the electron pass through a specimen. It is the main limiting factor for resolution once the spherical aberration (Cs) has been corrected in TEM. Cc-corrected TEM has been proven very useful in physics and materials science research as it can extend the contrast transfer function (CTF) of TEM to higher spatial frequency and improve the ability of TEM to image thick specimens. Both advantages are essential for cryo-EM in the field of structural biology, especially for cryo-electron tomography (cryo-ET). Cryo-electron tomography can resolve the structures of biological specimens in their native states, and combined with sub-tomogram averaging, it allows structural and cellular biologists to obtain a sub-nanometer resolution of specimens in “in-situ”. However, the Cc corrector has not been applied in cryo-EM yet until recently. The first Cc/Cs corrected cryo-EM is currently being installed at the Southern University of Science and Technology (SUSTech) in China. This talk will present first time some background, installation progress and user experiences of this microscope, and particularly the talk will focus on the coherent imaging of the energy-loss electrons by using Cc/Cs corrected cryo-EM and whether these inelastic electrons can be beneficial for cryo-EM methods such as single particle analysis and cryo-ET. Preliminary results will be presented and discussed.

STREAM - MEMS-BASED LIQUID PHASE ELECTRON MICROSCOPY: A PROMISING ROUTE FOR MATERIAL SCIENCE, ENERGY STORAGE & LIFE SCIENCE APPLICATIONS

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Having the capability to enable electron microscopy imaging in liquid environments has been attracting much interest from the scientific community. The possibility to visualize, in real time, the dynamic mechanisms of different samples in their native liquid nature, as a function of stimuli such as heating or biasing, opens up a wide variety of opportunities in fields like energy storage, electrocatalysis, corrosion, materials synthesis and life science applications.

Here, we introduce a system for in situ liquid phase studies inside the Transmission Electron Microscope (LPTEM), referred to as the “Stream” System (Figure 1). This system uses a Micro Electro-Mechanical System (MEMS)-based device as a smart sample carrier (Figure 2), which contains an integrated set of biasing electrodes to enable in situ electrochemistry, or an integrated microheater to allow liquid heating experiments.[1,2]

Historically, the LPTEM community has been facing several challenges, including uncontrolled liquid layer thickness that affect the imaging resolution and hinder analytical techniques such as electron diffraction, EDS or EELS. Furthermore, controlling the microfluidic environment around the sample (i.e. pressure, flow rate) has proven to be extremely challenging. We have designed the MEMS-based sample carriers to address these key challenges in order to provide meaningful results. During this talk, we will highlight and discuss the key features and benefits of the DENSsolutions Stream System.

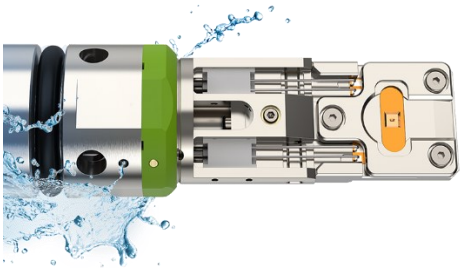

Fig. 1. The tip of the environmental holder of the Stream System.

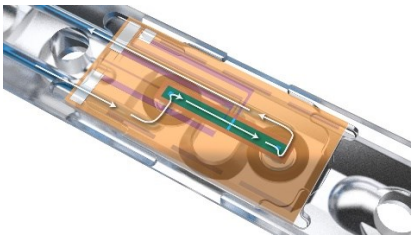

Fig. 2. The MEMS-based sample carrier highlighting a well-defined microfluidic channel.

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ENERGY-FILTERED 4D STEM FOR ALL KVs

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Four-dimensional scanning transmission electron microscopy (4D STEM) is becoming more popular in correlative and comprehensive electron microscopy research, as commercial electron detection cameras are made more sensitive and faster. In a 4D STEM experiment the region of interest is scanned in a two-dimensional (2D) array using a scanning probe, while a 2D diffraction image is captured at each probe position on a high-speed pixelated electron detector (camera). Such pixelated detector can be installed pre or post an imaging filter on the microscope and in the latter case higher signal to noise ratio diffraction images are captured by filtering inelastically scattered electrons using an energy slit. We have previously demonstrated the benefits of energy filtered 4D STEM using fast detection and read-out capabilities of a K3 direct detection camera, in a GIF Continuum K3 system. K3 camera has a large array monolithic transmission detector with a thin low-Z material that gives an excellent point spread function (PSF) at high kVs. The sharp PSF ensures a highly localized single electron detection, thus high detection quantum efficiency (DQE). At lower kVs (<80 kV), however, the PSF in monolithic detectors starts to spread, resulting in lower DQE. To address this we have recently integrated a hybrid pixel electron detector (Gatan Stela), in the GIF Continuum K3. Stela camera uses Dectris hybrid pixel electron technology, with excellent PSF at low kVs (<80 kV), high speed electron counting (>16000 fps), and high dynamic range (essential for 4D STEM diffraction applications). The GIF Continuum system with both the K3 and Stela cameras, covers the entire range of 30-300 kV (Fig. 1), allowing for energy filtered 4D STEM studies on a large variety of specimens (batteries, 2D materials, polymers and biomaterials to ceramics, metals and semiconductors). Additionally, the integrated hardware system with DigitalMicrograph software, and pre-optimized 4D STEM tools, allows for high quality data acquisition and processing irrespective of the user level of expertise or complexity of the experiments in a number of different applications such as strain mapping, virtual imaging, and differential phase contrast Imaging.

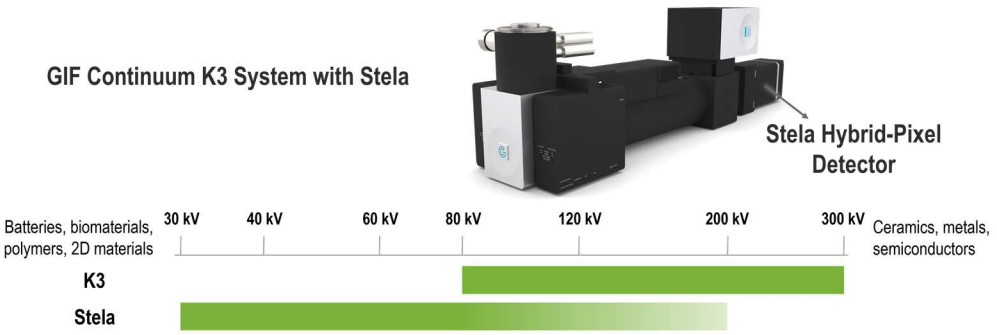

Fig. 1 Integrated GIF Continuum K3 system with Stela Camera. Energy-filtered imaging and diffraction for the entire range of 30-300 kV.

EXCEPTIONAL CONTROL OVER THE DOSE ON YOUR SAMPLE – JEOL-IDES EDM IS THE KEY

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JEOL-IDES beam blanking and deflection technologies are powerful tools which can upgrade existing TEMs with time resolution enabling new applications and explorations of dynamics of specimens across a range of very fast time scales. The optional Electrostatic Dose Modulator (EDM), a fast beam blanking system the beam ca be switched on or off in less than 50 ns. This 100,000x improvement in blanking speed immediately improves the clarity of data taken at fast exposure times. EDM can also very easily attenuate the electron beam intensity without affecting imaging conditions, giving TEM and STEM users exceptional control over the dose on their samples. Cutting-edge electronics and software add-ons unlock advanced applications such as temporal dose structuring and STEM synchronization.

USE OF PICO-TYPE MICROSCOPY TECHNIQUES TO OPTIMIZING COMPOSITION AND MICROSTRUCTURE IN COMPOSITIONALLY COMPLEX ALLOYS

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A recently developed refractory MPEA, AlMo0.5NbTa0.5TiZr, exhibits an interesting microstructure with an ordered B2 phase being the matrix and a disordered bcc phase being the precipitate, unlike the conventional Ni-based superalloys where the ordered phase (γ') is the precipitate and the disordered phase is the matrix (γ). Specific heat-treatments have been employed which have been designed to yield mechanistic details of the phase transformation pathway leading to the final microstructure. Based on these experiments, it appears that the pathway includes conditional spinodal decomposition, phase separation, and congruent ordering and disordering transformations. A critical parameter that influences the final microstructure is the interfacial energy between the ordered and disordered phases, and so the properties of the interfaces, for example, the misfit between the bcc and B2 phases, have been studied in detail. These experimental determinations are being used to develop a phase field model of the phase transformation pathway of this alloy, and this model is being employed to provide predictions of heat-treatments that will result in optimized microstructures. Of interest is the ordering scheme for the B2 compound, and attempts have been made to determine the sublattice occupancy using spatially-resolved XEDS spectroscopy in an aberration-corrected (S)TEM. Finally, the deformation mechanisms that are activated in this alloy have been studied and been shown to involve dislocation glide of closely coupled superlattice pairs.

CRYSTALLOGRAPHIC AND COMPOSITIONAL EVOLUTION DURING ISOTHERMAL ANNEALING OF REFRACTORY HIGH ENTROPY ALLOYS: INSIGHTS INTO HIGH TEMPERATURE PHASE STABILITY

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Refractory high entropy alloys (RHEAs) are promising candidates for next generation high temperature materials. The microstructure similar to Ni-base superalloys can be achieved in RHEAs containing Al, albeit with a combination of disordered BCC and ordered B2 phases. Since the ordered B2 phase is based on BCC parent matrix, distinguishing these two phases can be rather challenging. Advanced characterization techniques are necessary for a reliable qualitative and quantitative analysis of BCC and B2 phases in RHEAs. Additionally, there is a tendency for transformation of ordered B2 phase into more complex ordered-omega type phases that are usually deleterious to mechanical properties. The current study focuses on the phase stability of a candidate RHEA, $Al_{0.5}Mo_{0.5}NbTa_{0.5}TiZr$. The use of correlative transmission electron microscopy (TEM) and atom probe tomography (APT) reveals an interesting phase transformation pathway when this RHEA is isothermally annealed at 800°C. The results show that a metastable two-phase BCC+B2 microstructure formed in early stages of decomposition, later transforms into a three-phase BCC+B2+hp18 microstructure. The hP18 phase is an ordered omega-type phase derived from the ordered B2 phase. The correlative TEM and APT results reveal very interesting structural and compositional characteristics of these different phases and how they evolve during 800°C annealing.

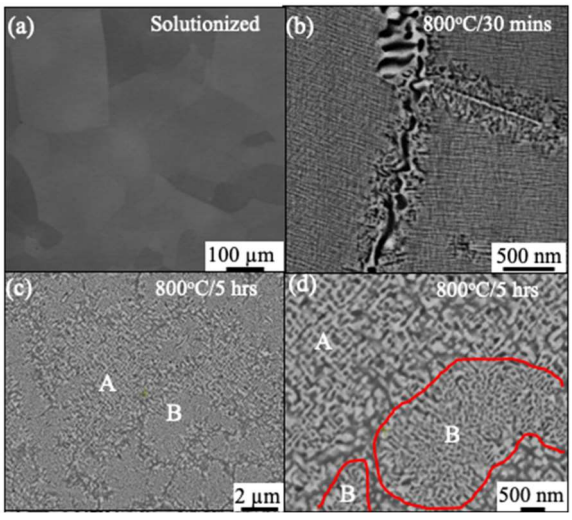

Fig. 1 Backscattered SEM micrographs of the microstructural evolution at 800°C. Microstructure in (a) solutionized condition; after annealing for (b) 30 minutes (c-d) 5 hrs

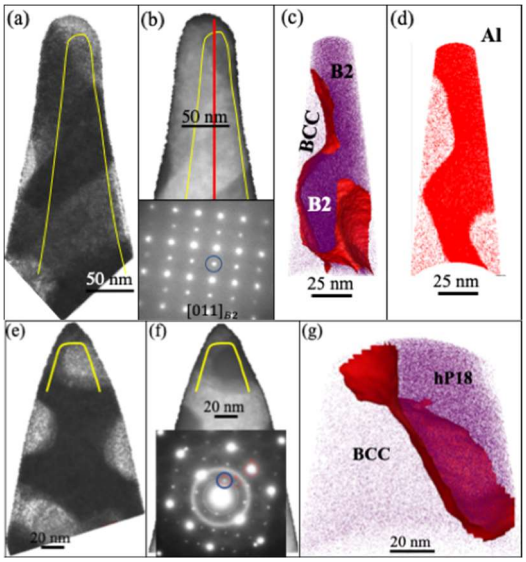

Fig. 2. Correlative TEM-APT of the three-phase microstructure in 800°C/5hrs condition. (a-d) BCC+B2 region (e-g) BCC+hP18 region

ANALYSIS OF PRECIPITATES IN ALUMINIUM ALLOYS FROM DIRECT DETECTION SPED DATA

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In scanning precession electron diffraction (SPED) a convergent probe scans a 2D region of the specimen while a precessed diffraction pattern is recorded for every probe position, creating a 4D dataset from a relatively large area. Analyzing SPED data from age-hardenable aluminium (Al) alloys can provide good statistics of the relative phase occurrence of nanoscale precipitates [1]. To evaluate various data analysis methods, the methods were applied to model data collected from an Al-Cu-Li alloy containing precipitate phases distinguishable by shape. The model data were acquired in a [001]Al zone axis, using a JEOL 2100F equipped with a Nanomegas system and a QD Merlin detector. Since the precipitates have certain orientation relationships (ORs) to Al, the data only contain five unique precipitate diffraction patterns. This follows from two precipitate phases, T1 and θ', with two and three possible ORs, respectively. Figure 1 shows the virtual dark-field (VDF) image from a typical dataset along with examples of the five unique diffraction patterns. The different data analysis methods were tested on the dataset to assess their performance in terms of speed and accuracy. The methods include non-negative matrix factorization, template matching, a vector-based approach, and neural networks. The goal is to establish a robust and unbiased way of obtaining phase maps of precipitate distributions in Al alloys. All data analyzes are done in the open-source python library for multi-dimensional diffraction microscopy PyXem [2].

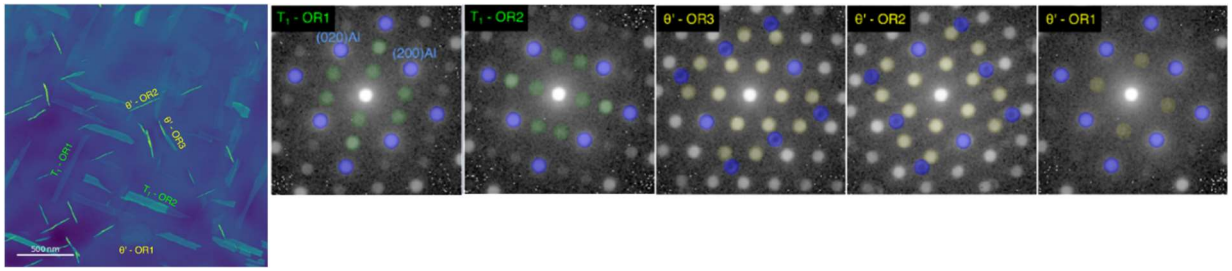

Fig. 1. VDF image of a typical dataset from an Al-Cu-Li alloy acquired in [001]Al zone axis, along with examples of the five unique precipitate diffraction patterns identified.

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MEASURING GRAIN/TWIN BOUNDARY PLASTICITY AT ATOMIC LEVEL

L.H. Wang, Z.P. Li, J.F. Zhang, Y. Zhang, Z. Zeng, H. Zhou, J. He, P. Liu, J. Han, D.J. Srolovitz, J. Teng, Y. Z. Guo, G. Yang, D.L. Kong, Y.L. Hu, B.C. Yin, X.X. Huang, S.C. Mao, M.W. Chen, E. Ma, T. Zhu, Z. Zhang[2] and Xiaodong Han[1]

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How to characterize and measure the interface phenomena on the microscopic level is one of the most fundamental questions [1]. Grain boundary (GB) and twin boundary (TB) are two type important and fundamental interfaces in materials and solids. How to characterize and measure grain/twin boundary plasticity at high spatial resolution, particularly at atomic scale, remain challenging. Cs-corrected transmission electron microscope brought the characterization of materials structures towards sub-A scale which enable to characterize the arrangement of individual atoms, atomic columns and planes and their chemistry, even electronic states at atomic scale [2]. 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 dislocations creates dislocation locks. The unlock and re-lock processes of the interacted 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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MICROSCOPIC DEGREES OF FREEDOM OF GRAIN BOUNDARIES AND MICROSTRUCTURAL EVOLUTION

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The fundamentals of the five *macroscopic* degrees of freedom of grain boundaries (GBs) has been a topic of research leading to concepts such as the O-lattice, the coincident site lattice (CSL) and general aspects of microstructural evolution (grain growth) [1]. Studies of the *microscopic* degrees of freedom of GBs have been dependent on advanced electron microscopy and spectroscopy, often combined with atomistic simulations, but usually limited to special grain boundaries which do not necessarily play a role in microstructural evolution or the properties of polycrystalline materials. Nevertheless, these experiments have provided important information on the atomistic aspects of grain boundaries and interfaces in general [2].

In recent years it has become clear that 2-D transitions in the “state” of a GB can lead to significant changes in GB kinetics and may be connected to changes in properties such as embrittlement [3]. Formally, these transitions include changes in both the 2-D structural symmetry and chemistry of an interface, and have been described via diffuse interface theory [4,5]. Due to limitations on the ability to characterize the atomistic structure of general high-angle GBs, most transitions have been identified by changes in chemical excess, or even erroneous measurements of GB “thickness” [6]. More recently it has been hypothesized and experimentally confirmed that disconnection motion is the mechanism for grain boundary motion [7,8]. This presentation will discuss the role of solute adsorption as it interacts with disconnection formation and motion, and the possible role of GB transitions on disconnection formation and motion. Examples demonstrating the challenges for advanced electron microscopy and spectroscopy will be discussed.

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MECHANISMS BEHIND HYDROGEN-BASED GREEN STEEL MAKING

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More than 1.8 billion tons of steel are produced every year, making it by far the most important alloy in terms of volume, impact and commercial turnover. While steel is a sustainability enabler, through lightweight car parts, wind farms, railway infrastructure and magnets, its primary production is the opposite. Iron is reduced since more than 3500 years from oxidic ores using carbon. This produces about 2t CO_2 per ton of steel, standing for >30% of the global CO_2 emissions in manufacturing. Drastically reducing these enormous primary greenhouse gas emissions is the biggest materials science challenge of our generation.

While current transient strategies by industry often lie in CO_2 emission trading and massive underground CO_2 storage, materials science can help to identify more sustainable and commercially viable solutions for direct CO_2 avoidance [1].

More specific, emissions could be radically reduced when replacing carbon by hydrogen-containing radicals, protons or electrons and their carriers as reductants and heat sources for melting scraps [1,2].

The lecture presents some recent progress in understanding the key mechanisms behind two of these approaches, namely, hydrogen-based direct reduction and hydrogen-based plasma reduction [3]. The kinetics of the reactions strongly depend on mass transport kinetics in the oxides, nucleation during the multiple phase transformations, the oxide's chemistry and microstructure, and on damage and fracture associated with the phase transformation and mass transport phenomena occurring during reduction. Understanding these effects is key to make hydrogen-based reduction of iron ores towards high metallization at fast kinetics commercially viable, enabling massive CO_2 reductions.

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STRUCTURAL STUDIES OF VIRUSES BY CORRELATIVE CRYO-MICROSCOPY

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Respiratory Syncytial Virus (RSV) is a pleomorphic, enveloped, negative-sense, single-stranded RNA virus. The RSV genome contains 10 open reading frames, encoding nine structural proteins and two non-structural proteins. The surface exposed attachment glycoprotein (G), fusion glycoprotein (F), and small hydrophobic protein (SH) are embedded in the viral membrane. The matrix protein (M), which drives virion formation and elongation, lines the interior of the viral membrane. The genomic RNA is encapsidated in the ribonucleoprotein complex (RNP) that is present in the interior of virions. Cryo-ET studies have shown that the morphology of individual virions does vary, with the majority of virions having an average diameter of ~130 nm and a length that ranges from ~500 nm to over 10 μm [1, 2]. The general arrangement of structural proteins within the virion is known, however, the molecular organization of M and other structural proteins has remained elusive. We will discuss developments with correlative microscopy, whole-cell cryo-electron tomography (cryo-ET), multi-shot montage tomography [3], and sub-tomogram averaging and their application to structural studies of RSV (Fig. 1). We will show that RSV M is arranged in a packed helical-like lattice of M-dimers ordered preferentially to the viral long axis (Fig. 2). Sub-tomogram averages including F and M suggest that the position of F is correlated with the underlying M lattice.

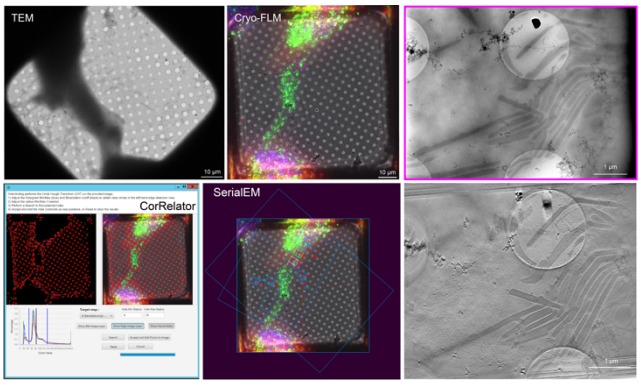

Fig. 1. Correlative cryo-microscopy strategy for studying RSV assembly.

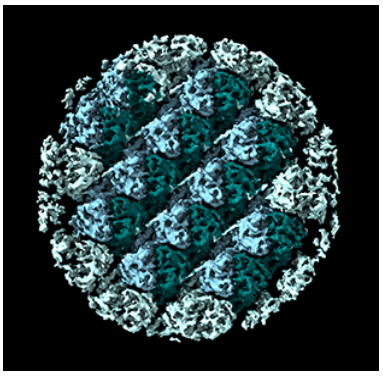

Fig. 2. Structure of the RSV matrix (M) protein lattice at 4.6 Å.

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EASY-GRID: ADVANCING SAMPLE PREPARATION FOR CRYO-EM

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Cryogenic electron microscopy (cryo-EM) allows to investigate the structure of biological specimens in a close to physiological condition by imaging the macromolecules of interest vitrified within biochemically functional buffers. Despite the impressive developments of microscopy hardware and image processing methods that contributed to the so called 'resolution revolution', the preparation of high-quality specimens is often the limiting step in the determination of molecular structures by cryo-EM [1]. In the last decade, an effort has begun to develop more sophisticated vitrification methods enabling deposition of minimal sample volumes, blotless sample spreading, and rapid vitrification [2-5]. Despite these exciting recent advances, although theoretically possible, at present the high-throughput screening of a large array of conditions is practically unattainable. We aim at establishing a high-throughput cryo-EM laboratory by developing automated pipelines for large-scale cryo-EM sample preparation and screening. To this end, we are developing the EasyGrid, a next-generation modular robotic platform that provides controlled and efficient preparation of cryo-EM specimens. The EasyGrid will enable fully automated handling of samples for cryo-grid production, "on-grid" light-induced activation of caged compounds for the investigation of dynamic processes by time-resolved cryo-EM, and rapid vitrification of the sample by liquid ethane jet-freezing improving vitrification of thick specimens for cellular structural biology studies. Our developments will be implemented as scientific service provided to the community through the EMBL Imaging Centre, EMBL's new service unit enabling access to cutting-edge electron and light microscopy technologies.

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ULTRASTRUCTURAL INVESTIGATIONS OF SYNUCLEINOPATHIES: PD, MSA, HUMAN BRAIN, AND FIBRIL STRAINS

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Parkinson's disease (PD) is a neurodegenerative disease of multifactorial origins. It's molecular causes or cellular mechanisms are not clear. Available treatments only ease symptoms, but cannot stop or delay the disease. The hallmark of Parkinson's disease is the presence of intraneuronal aggregates in the brain, termed Lewy bodies, which can be found after death of the patients. These Lewy bodies contain a high concentration of the protein alpha-synuclein (aSyn), making it a prime suspect for the cause of Parkinson's disease and a drug target in ongoing clinical trials. aSyn is capable of aggregating into prionoid fibrils. aSyn misbehavior is also suspected to be involved in a larger number of other diseases, such as Multiple System Atrophy (MSA) or Dementia with Lewy Bodies (DLB). The structural analysis of these fibril by cryo-electron microscopy (cryo-EM) will be presented, and high-resolution structures of different aSyn fibril strains will be discissed, which reveal a structural polymorphism that might explain the variability of observed phenotypes of synucleinopathies [1]. In parallel, EM studies of post-mortem human brain will be presented, and the ultrastructure of plaques in the brain tissue of synucleinopathies such as PD and multiple system atrophy (MSA) will be discussed [2].

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BACTERIA CYTOSKELETON VISUALIZATION BY CRYO ELECTRON TOMOGRAPHY AND CORRELATIVE APPROACHES

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Cryo electron tomography (cryo-ET) provides us with possibilities to visualize the architecture and molecular machines driving bacterial growth and development [1]. The bacterial cytoplasm and plasma membrane is a crowded environment, filled with biomolecules, with unique spatial organization and dynamic properties, we aim to envision. However, bacteria dimensions and the electron density of biomolecules under native conditions, makes the transmission electron microscopy signal to noise ratio less favorable for cryo-ET of bacterial specimen. Recent developments of cryo focused ion beam (cryo-FIB) preparation, correlative fluorescence and electron microscopy as well as spatial proteomics in combination with cryo-ET has shown great importance for visualization of cell volumes at near native state. At Umeå Centre for Electron Microscopy (UCEM), we prepare electron-transparent lamella from vitrified bacteria culture by cryo-FIB milling. Lamellas are subsequently analyzed by cryo-ET and 3D image reconstruction using the IMOD software package, yielding low contrast but high-resolution information. We use correlative approaches including cryo-ET to study the filamentous bacterium *Streptomyces coelicolor* intermediate filament like protein FilP. Bacterial intermediate filament like cytoskeleton proteins can be identified by their secondary and tertiary structural characteristics and chemical properties. We found that purified FilP polymerize *in vitro* into thick, branched, repeatedly segmented filaments and networks with a 60 nm repetitive unit (Fig. 1). We further studied the *in vitro* assembly of FilP and were able to build a 3D-model of the filament bundle structure [2]. The cellular localization of FilP is guided by proteins of the *Streptomyces* polarisome complex. One of the polarisome proteins DivIVa can be fluorescently tagged with GFP to guide cryo-ET data collection to the region of interest. The heteromorph appearance of FilP filaments, in analogy to intermediate filaments, makes ET and 3D reconstruction methods crucial to characterize the intracellular spatial organization.

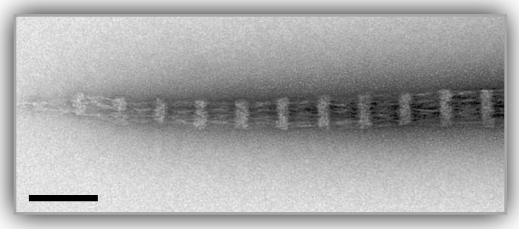

Fig. 1: FilP, bacteria intermediate like cytoskeleton, scale bar 100 nm

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MEMS TECHNOLOGY FOR CRYO-EM SAMPLE PREPARATION

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Cryogenic electron microscopy has become an essential tool for structure determination of biological macromolecules. Despite many technical advances, the difficulty to reliably prepare samples with uniform ice thickness and the excessive sample loss during grid preparation still present a major barrier for routine high-resolution imaging and limit the current throughput of the technique. Nanofabrication techniques employed for Micro-/Nanoelectromechanical Systems (M/NEMS) provide opportunities to miniaturise and automate cryo-EM sample preparation. We have shown that MEMS-based nanofluidic sample supports with well-defined geometry can be used to prepare cryo-EM specimens with reproducible ice thickness from picoliter sample volumes [1]. The sample solution is contained in electron-transparent nanochannels that provide uniform thickness gradients without further optimisation and eliminate the potentially destructive air-water interface. We demonstrate the possibility to perform high-resolution structure determination with various protein specimens. Besides the possibility to develop our method into a viable alternative to holey support films, nanofabricated sample supports bear potential to completely automate the cryo-EM workflow, and to explore new frontiers for cryo-EM applications such as time-resolved imaging and high-throughput screening.

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RESOLVING STRUCTURAL DYNAMICS OF PROTEINS USING DROPLET MICROFLUIDICS

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Single particle cryogenic electron microscopy (cryo-EM) permits determining high-resolution structures of biological macromolecules vitrified in a thin layer of ice. Using cryo-EM multiple conformational states present in the ensemble of proteins can be resolved and conformational flexibility, as well as compositional heterogeneity of the proteins and protein complexes, can be inferred providing insight into the function and mechanisms of the studied proteins. Cryo-EM can be used to study proteins as they are performing their function; however, established sample preparation techniques probe the distribution of functional states equilibrated on a timescale of seconds. As a result, the conformations with short lifetimes are not resolved. Due to its rapid vitrification speed, cryo-EM permits studying proteins out of equilibrium on milliseconds or even microsecond timescales which can be achieved by rapid mixing of reagents followed by their application onto a cryo-EM grid and consequent plunge freezing after a defined delay time. While this approach has already enabled trapping short-living and functionally important protein conformations, combining rapid sample mixing with rapid preparation of cryo-EM grids remains technically challenging limiting the application of this method. We have developed an apparatus for rapid mixing of protein solutions and delivering them on cryo-EM grids with a millisecond time scale using droplet microfluidics. The new instrument and examples of applications to molecular machines will be presented.

EVENT-BASED SPECTROSCOPY IN A STEM: FROM NANOSECOND RESOLUTION TO TIME-CORRELATED HYPERSPECTRAL IMAGING

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The Pico2022 conference celebrates 25 years of aberration correction and the many advances that these developments have brought to the electron microscopy community. In scanning transmission electron microscopy (STEM), a more recent breakthrough has been the development of new-generation monochromators, preserving the brightness of electron sources, and thus opening the way to new applications in electron energy-loss spectroscopy (EELS). More than just improving spectral resolution (now available with atomic resolution), monochromation in EELS has given access to a whole new range of low-energy elementary excitations (down to the infrared range) with nanoscale resolution. The field of nano-optics using fast electron beams has been booming in recent years, boosted by recent developments in experiments combining photons and electrons in the microscope: cathodoluminescence, electron energy-gain spectroscopy, photon-induced near-field electron microscopy, etc. Further advances are still to be expected by pushing the limits of time resolution, both as instrumentation advances and for accessing new physical information. We have developed new acquisition schemes making use of a Timepix3 direct electron detector providing sub 10 ns time resolution over arbitrary EELS energy ranges. Examples for event-based hyperspectral acquisition will be given, showing that entire spectral images with arbitrary scanning pixel dwell time can be reconstructed and thus that hyperspectral images can be acquired at the same rate as that of a single channel detector, such as annular dark-field [1]. In parallel, we are developing at Orsay new schemes aiming at synchronizing a continuous electron beam with (in some cases pulsed [2]) photon beams, as a complement to approaches using a pulsed electron gun. I will present a new type of time-correlated experiments based on coincidence measurements between inelastic electron scattering and photon emission events: cathodoluminescence excitation (CLE) spectra (so called as the counterpart to photoluminescence excitation (PLE) spectroscopy) are constructed with EELS events that are time-correlated with a photon emission. I will show how CLE permits us to recover the phase-locked nature of plasmons in Au nanoparticles and to unveil all excitation pathways towards light emission from individual point defects in h-BN, across the visible-UV and soft X-ray ranges [3]. More generally, I will show how this newly developed CLE spectroscopy can image energy transfer pathways at the nanometer scale.

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ELECTRON ENERGY LOSS SPECTROSCOPY WITH HIGH SPATIAL RESOLUTION: APPLICATIONS TO CATHODE MATERIALS IN LI-ION BATTERIES

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Electron energy loss spectroscopy (EELS) is an invaluable technique to study the detailed structure and the chemical state of materials at unprecedented spatial resolution. Today, this technique is used to characterize nanoscale materials used in a myriad of applications from energy storage and conversion, to solid-state devices. This technique also provides complementary information to broad-beam photon-based synchrotron techniques that can probe materials in dynamic operating conditions. In this presentation, I describe recent developments in electron energy loss spectroscopy (both at very low energies and for core-loss spectroscopy). I will briefly describe some latest results on the investigations of plasmonic activity in metallic nanostructures that that show promise in photo-electrochemical activity to understand how strong plasmonic and broadband response originates in these nanostructures [1-3]. Then I will focus on the combination of EELS and synchrotron-based methods to elucidate the structure and chemical composition of novel engineered high-performance cathode materials and phase transformations arising in these materials when materials are electrochemically cycled [4-5]. A comparison of the information obtained from electron beam methods with photon-based methods will be shown.

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RECENT PROGRESS OF ELECTRON SPECTROSCOPY WITH THE SINGLE-ATOM LIMIT

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Electron energy-loss spectroscopy (EELS) has been widely used for elemental identification at the single-atom limit in transmission electron microscopes (TEM) by using core-level excitations. Recent developments of monochromators after the e-beam guns have enabled us to access optical and vibrational information from the valence EELS ranges of nanometric materials with high-spatial/energy resolution. Here we show our latest studies to demonstrate the possibilities of EELS applied for low-dimensional materials. Examples for atomic defects in in-plane hybrid TMDCs [1], monolayer structures of metal chlorides intercalated in bi-layer graphene [2, 3], surface adatoms for catalysis [4, 5], two-dimensional iodine-monofluoride epitaxy [6], and isotopically heterogeneous graphene [7] will be shown.

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OPTIMUM BF (OBF) STEM USING THE SAAF DETECTOR

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Imaging beam-sensitive materials at low-dose conditions remains a very challenging topic in transmission electron microscopy (TEM), especially if the goal is the direct observation of the atomic structure of these materials. In particular, electron beam induced damage during image tuning can make visualizing the sample at atomic resolution an extremely difficult task. In this presentation we will discuss a new ultra-high contrast Scanning transmission electron microscopy (STEM) technique that has recently become available on JEOL TEMs called optimum bright-field (OBF) STEM [1]. This method works with both segmented as well as pixelated STEM detectors and has been shown to be highly dose efficient while at the same time allowing for real-time imaging under low-dose conditions. The capability to image beam sensitive materials in real time under low-dose conditions promises to make TEM work on these materials significantly easier. Here we will give a brief overview of the OBF STEM Method and present several application examples on different materials highlighting the performance and capabilities of this new imaging technique.

References:

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WAVEFRONT SHAPING IN THE ELECTRON MICROSCOPY WITH A 48 PIXEL PROGRAMMABLE PHASE PLATE

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We report on advances in the development of a versatile programmable phase plate for use in electron microscopy. The current design provides up to 48 individually addressable electrostatic phase shifting elements that can be laid out in multiple design patterns depending on the target application. The phase plate state can be synchronously updated at up to 100 kHz rate providing an attractive route for high speed adaptive algorithms shaping the electron probe while optimizing a goal function. We discuss different phase plate implementations and their usefulness towards applications. We show the realization of dipolar, quadrupolar and vortex phase symmetries in sub nm electron probes when placing the phase plate in the condenser plane of a modern STEM microscope. We demonstrate adaptive algorithms maximizing for contrast in HAADF STEM images as well as discussing their prospects for focusing inside thick samples.

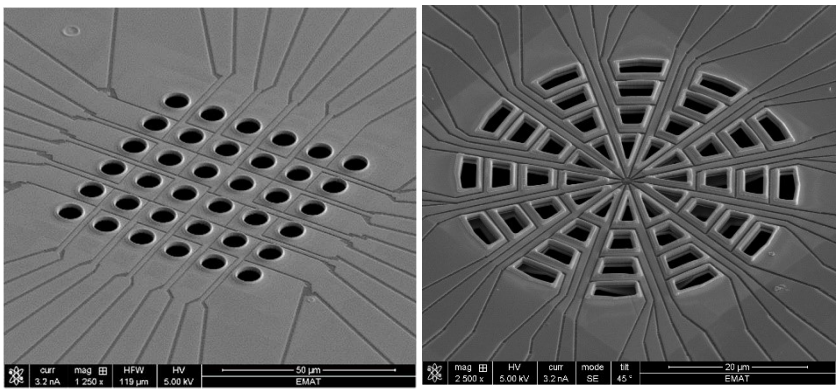

Fig. 1. Example of two prototype phase plate realisations showing a 36 pixels cartesian design (left) and a polar segmented 48 pixel design.

References:

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ADVANCED PROCESSING OF DIFFERENTIAL PHASE CONTRAST DATA

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Differential phase contrast (DPC) or first moment microscopy measures the gradient of the local phase experienced by the electron probe. In general, one retrieves a vector field with components in x and y direction, perpendicular to the optical axis. This vector field can be interpreted as the average lateral momentum transferred to the probe electrons at large. For the common sources of beam-specimen interaction, electric and/or magnetic fields, it can be shown that the resulting vector fields should be rotation-free. This is used, when the divergence of the vector field is taken to derive the charge which is responsible for the electrostatic interaction. However, general vector fields can be considered to be a superposition of a rotation-free and a divergence-free vector field (plus a constant). Helmholtz showed a way to decompose a vector field into these two components [1]. From the above said, one would expect any vector field measured by DPC to be rotation-free, but for real measured data they are not. One possible cause is a misalignment between the specimen's and the detector's coordinate systems, but this artefact can be easily avoided or detected. This opens a door to effects which cause rotational contributions to DPC data, and those will be discussed. From simulations it is obvious that geometric crystal distortions, such as screw dislocations (Fig. 1), will create non-zero rotational components in the measured vector fields (Fig. 2) and therefore allow structural characterization with first moment measurements.

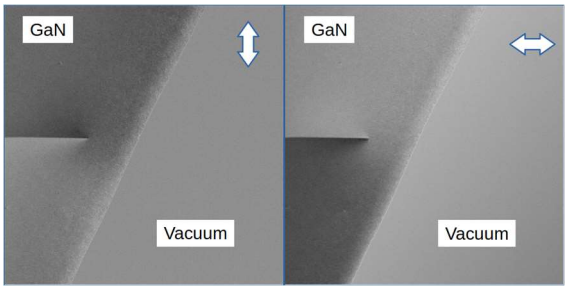

Fig. 1: First moment image pair of a dislocation in GaN. Double-headed arrows indicate the sensing direction. Data acquired at the ERC Jülich.

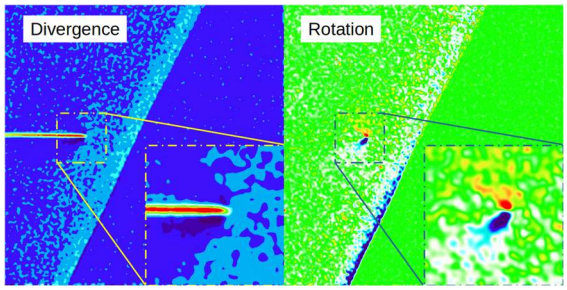

Fig. 2: Divergence (left) and rotation (right) reconstructed from data shown in Fig. 1.

References:

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PROSPECTS AND OPPORTUNITIES FOR ELECTRON PTYCHOGRPAPHY AT LOW DOSE

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This lecture will describe firstly recent developments in the use of Electron Ptychography as applied to studies of materials, including biological structures at low electron dose.

I will highlight the importance of fast detectors operating in a counting mode for electron ptychography at low dose. Acquisition of a ptychographic dataset requires the collection of a series of far field diffraction patters as a function of probe position at the specimen plane. This dataset can then be used to recover the complex specimen object function using either an iterative or non-iterative algorithm. Importantly, ptychography is a dose efficient technique, enabling effective phase reconstruction of radiation sensitive samples. At low dose the sampling of the diffraction pattern in the far field is sparse and a counting direct electron detector can be operated in a binary mode to provide an effective speed increase. I will illustrate this particular application using examples of radiation sensitive mesoporous materials.

In the life sciences Cryo-electron ptychography (Cryo-EPt) [1] is a promising new method which uses a defocused probe to scan across a specimen with highly overlapped probe positions. This can also be extended in a development of single particle analysis to provide 3D structures (Fig.1) and taking advantage of the known resolution variation of the effective ptychographic transfer function with convergence angle (Fig. 1) to provide wide spatial frequency bandwidth transfer.

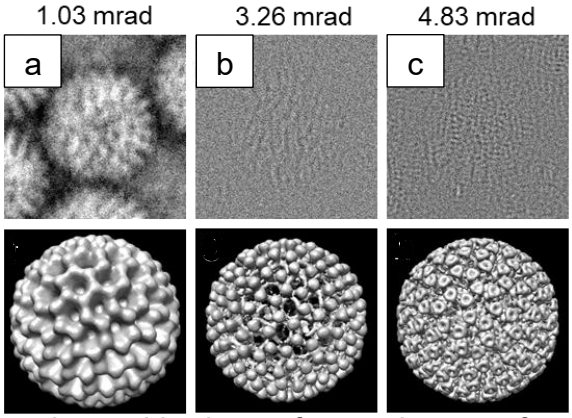

Fig.1. (a-c) Recovered ptychographic phase of a rotavirus as a function of convergence angle with corresponding 3 D density maps.

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AXON: AN IN-SITU TEM SOFTWARE PLATFORM STREAMLINES IMAGE ACQUISITION, METADATA SYNCHRONIZATION AND DATA ANALYSIS, ENABLING DEEPER UNDERSTANDING, AND IMPROVED REPRODUCIBILITY OF IN-SITU EXPERIMENTAL RESULTS

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A recurring refrain within the in-situ community is the need to develop minimum requirements for reporting experiment conditions and parameters to improve analysis and enable the reproducibility of results by other researchers [1]. The need to standardize reporting and enable other researchers to access metadata created during microscopy experiments was recently highlighted by Sarkans et al. as a general area for improvement within the wider biological imaging community [2,3]. Protochips recently introduced AXON, a software solution designed to address needs in image stabilization and metadata consolidation. In this talk, scientific examples of in situ applications will be presented and the role metadata analysis had on deriving scientific conclusions (Figure 1). In addition, specific applications will be presented on a new tool which utilizes machine vision technology to calibrate beam current, record dose rates and total accumulated dose within the sample area, and include this information directly into the image metadata for subsequent analysis (Figure 2.) With these advances, utilization of the AXON platform for in-situ experiments easily enables the application of FAIR principles to in-situ data management, and facilitates more robust analysis, data mining and review of in-situ experiments by outside researchers, increasing productivity and ultimately elevating the field of in-situ TEM.

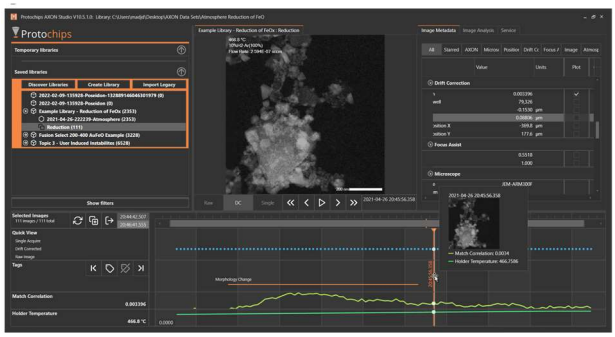

Fig. 1 Alignment and analysis of metadata

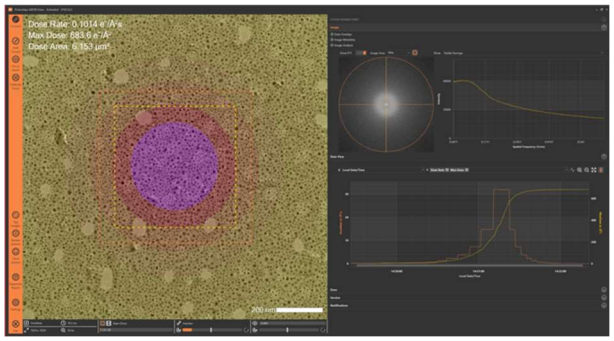

Fig. 2. Visualization of dose metadata

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IMPROVING SAMPLE DISPERSION WITH AN ADDITIONAL ULTRATHIN CARBON LAYER

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Sample preparation is a major roadblock in the cryo-EM workflow; identifying conditions in which the sample is well-distributed across the holes in random orientations can often seem like a black art to the novice cryo-electron microscopist. Grid choice and grid surface modifications can make a big difference to sample quality; but identifying the best support for a new project can be challenging. One additional option which can often improve particle distribution is an additional ultrathin carbon (UTC) layer [1], and Quantifoil offers additional UTC layers in a range of thicknesses for most holey supports. The layer acts in several ways to improve particle distribution:

- Increasing the number of particles due to adsorption of biomolecules onto carbon prior to blotting.
- Improving particle distribution due to interaction with a continuous carbon surface across the hole as well as the support.
- Reducing the number of particles adopting a preferred orientation.

Ultrathin carbon layers have been used with a wide range of biomolecules including: SARS-CoV2 proteins [2, 3] and membrane-embedded complexes, such as *Helicobacter pylori* outer membrane complex (OMC) [4] as well as a wide range of other biological macromolecules.

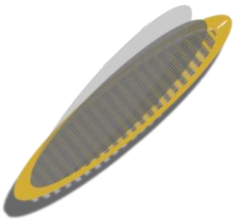

Fig. 1 Sample support with extra layer of ultrathin carbon

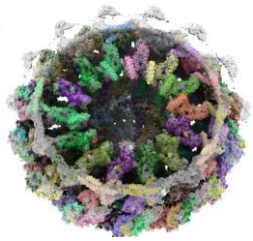

Fig. 2. *Helicobacter pylori* OMC from data collected on a sample support with a UTC [4]

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DYNAMICAL STUDIES OF ADVANCED MATERIALS COMBINING *IN SITU* 4D-sPED MAPPING AND PIXELATED DETECTORS

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Automatic crystal orientation & phase mapping (ASTAR technique) can be applied in a variety of materials, is known also as 4D-STEM technique [1, 2]. We apply a dedicated acquisition process (named as 4D-SPED "scanning Precession Electron Diffraction") where beam precession (to improve ED patterns quality [3]) in TEM is used in combination with beam scanning. ASTAR technique is very similar to the traditional EBSD-SEM method, where the major advantage is the significant improved spatial resolution that can be down to 1-3 nm (in case of FEG-TEM). In the standard configuration, a special CCD camera is used (able to collect a 100 fps) with dedicated scanning 4D-SPED DigiSTAR hardware. Local crystallographic orientation/phases are identified through an original algorithm that compares experimental ED spot patterns with pre-calculated (simulated) templates for all possible orientations and/or phases [4]. We have studied many advanced materials using 4D –SPED ASTAR, to investigate local properties and/or study of nanosized samples. In addition, *in situ* methods have been extensively applied towards dynamical studies. The combination of the 4D-SPED ASTAR with the new generation of pixelated detectors, allows the use of extremely low electron dose (up to 0.001 e/Å2/sec) with significantly fast acquisition times (500-1000fps). The use of pixelated detectors has critical advantages over conventional CCD, due to the absence of electronic detection noise (allowing to work at extremely low dose). Combining pixelated detectors (like QD & ASI Medipix,Dectris,PnCCD etc…) with NanoMEGAS 4D-SPED ASTAR system, not only acquisition time is greatly reduced, but more critically, data quality is enhanced, allowing in situ dynamical studies by sequential mapping even for beam sensitive samples.

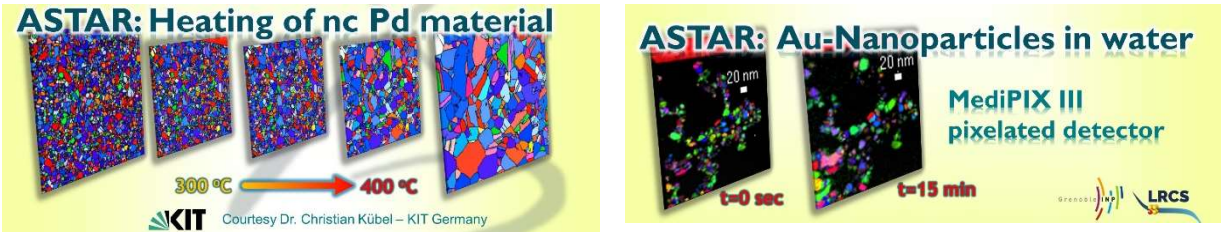

Fig. 1 *In situ* ASTAR applications using pixelated detector

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THE ARCHITECTURE OF ORGANELLE CONTACT SITES

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Organelles interact through close apposition of their membranes. Such contact sites serve the exchange of lipid molecules, calcium transport and organelle biogenesis. The mechanisms by which lipids are transferred across organelle contact sites are poorly understood because little is known about the underpinning supramolecular structure. We aim to reveal the architecture composed of proteins and apposing membranes and thereby shed light on how these microenvironments facilitate the transport of lipid molecules between organelles. Towards this goal, we employ correlative light and electron microscopy approaches including electron cryo-tomography. We complement these tools with live fluorescence imaging as well as biochemistry and genetic perturbations.

SECRETION SYSTEMS IN ACTION

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Many bacterial pathogens rely on secretion systems to establish infection. They assemble into large, membrane embedded macromolecular machines that often function as protein transport systems to secrete virulence factors. The type III secretion systems (T3SSs) or injectisomes assemble a needle complex containing a continuous conduit crossing the bacterial envelope and the host cell membrane to mediate effector protein translocation [1]. However, the molecular principles underlying type III secretion remain elusive. Here, I will discuss how we have established an experimental system to trap and visualize needle complexes from Salmonella enterica serovar Typhimurium in action by cryoEM, and how we used both manual and easy-to-be-used semi-automated atomic model refinement (StarMap: Rosetta-based refinement controlled from ChimeraX) [2] to obtain complete atomic models of the needle complex engaged with the effector protein SptP in two functional states. We uncovered the complete 800Å-long secretion conduit and unravelled the critical role of the export apparatus subcomplex in type III secretion. Unfolded substrates enter the export apparatus through a hydrophilic constriction formed by SpaQ proteins, which enables side chain-independent substrate transport. Above, a methionine gasket formed by SpaP proteins, which functions as a gate that dilates to accommodate substrates while preventing leaky pore formation. Following gate penetration, a moveable SpaR loop first folds up to then support substrate transport. Together, these findings establish the molecular basis for substrate translocation through T3SSs and explain tight control of gate opening by reorienting specific side-chains within the export apparatus [3].

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MACROMOLECULAR ORGANIZATION OF ATG18 OLIGOMERS

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Atg18 is a core protein in macroautophagy, a highly conserved eukaryotic pathway critical for the cell's stress response and homeostasis. The precise structural role of Atg18 in the autophagy process remains to be established. Atg18 belongs to the PROPPIN family consisting of seven WD40 repeats that form a beta propeller motif. Atg18 has been shown to bind Atg2 and Atg9 and thereby participates in the elongation of phagophores. Atg18 has a conserved PIP/PIP2-binding motif (FRRGT) that constitutes a specific membrane anchor. We demonstrate how Atg18 can form filaments in low-salt solution by solving the helical structure to near-atomic resolution using electron cryo-microscopy. The oligomerization sites are conserved even under high-salt conditions, as we demonstrate by single particle analysis of this small 55 kDa protein. We reconstituted Atg18 with liposomes and elucidated the macromolecular organization of Atg18 oligomers on cellular membranes using subtomogram averaging. The structure reveals that oligomerization motifs present in helical filaments and in solution are also observed between juxtaposed membranes, demonstrating how Atg18 may function to remodel and extend phagophore membranes in concert with the larger autophagy machinery.

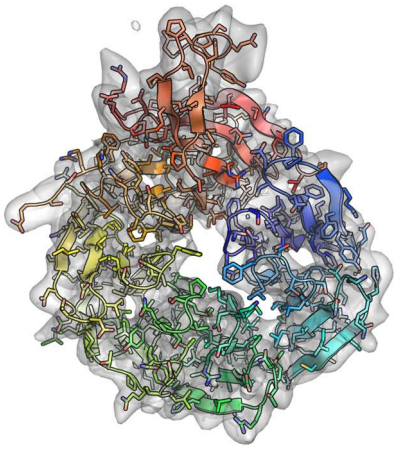

Fig. 1. 55 kDa Atg18 structure from single particle analysis

TEMPORAL DYNAMICS OF CHARGE BUILD IN CRYO-ELECTRON MICROSCOPY

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It is well known that insulating samples can accumulate electric charges from exposure to an electron beam. How the accumulation of charge affects imaging parameters and sample stability in transmission electron microscopy is poorly understood. To quantify these effects, it is important to know how the charge is distributed within the sample and how it builds up over time. In the present study, we determine the spatial distribution and temporal dynamics of charge accumulation on vitreous ice samples with embedded proteins through a combination of modeling and Fresnel diffraction experiments. Our data reveals a rapid evolution of the charge state on ice upon initial exposure to the electron beam accompanied by charge gradients at the interfaces between ice and carbon films. We demonstrate that ice film movement and charge state variations occur upon electron beam exposure and are dose-rate dependent. Both affect the image defocus through a combination of sample height changes and lensing effects. Our results may be used as a guide to improve sample preparation and data processing for imaging of dose-sensitive samples.

AMYLOID FIBRILS AND THE INTERPRETATION OF COFACTOR DENSITIES

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Amyloid fibrils are involved in many neurodegenerative diseases. It has become clear in the last few years that the aggregation of amyloidogenic proteins and peptides into fibrils, and therefore also the resulting fibril structure strongly depends on the environment conditions. In particular, co-solutes play an important role in driving the aggregation and determining the specific structure. In cryo-EM reconstructions, additional co-solute densities have been observed in several cases, but their interpretation is challenging. One example of a disease relevant amyloid is α-synuclein, which forms neuronal inclusions called Lewy bodies in Parkinson's disease. It is known that lipid membranes are important for the aggregation of α-synuclein. We present six different structures of α-synuclein fibrils grown in the presence of lipids, which show micelle-like lipid densities as well as specific lipid-fibril interactions. We interpret the lipid densities with extensive molecular dynamics simulations.

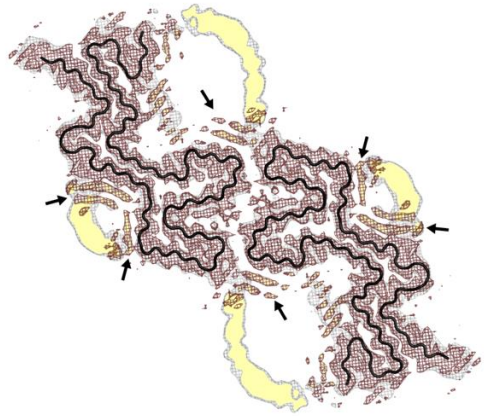

Fig. 1: The cross-section of an α-synuclein fibril with micelle-like lipid densities

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ABERRATION CORRECTION IN TANDEM WITH MODELLING FOR ATOMIC RESOLUTION STRUCTURE DETERMINATION

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Aberration corrected electron microscopy has now been in use and under continuous development for a quarter of a century. This has enabled novel modes of atomic resolution imaging and spectroscopy with many applications to characterization of materials at the atomic scale. Some of these developments will be highlighted [1-13] as well as the importance of an understanding of the physics of the interaction of the probe with the specimen [14-17] in optimizing the information we can obtain about the structure of a specimen at atomic resolution. The probe can be elastically scattered or inelastically scattered in a number of ways [16] and it is often not possible to directly interpret data obtained on modern aberration corrected microscopes and modelling is then an essential adjunct to experiment [9,11]. Further improvements in microscope design, spectrometers, x-ray detectors and the capabilities of fast-readout pixel detectors will facilitate greater accuracy and further applications. Some new ideas for structure determination in 3D [18], which would benefit from further improvements in resolution, will be discussed.

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PROGRESS IN ABERRATION-CORRECTED ELECTRON MICROSCOPY IN MATERIALS SCIENCE

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Aberration-corrected electron optics in transmission electron microscopes has opened the door to experimental studies of the atomic structure of materials with sub-ångström resolution and pico-meter precision resolution in two and three dimensions. This ability to directly image the arrangement of individual atoms provides crucial insights into the correlation between atomic structure and properties, for example in catalytic structures, electronics, photonics, plasmonics, photovoltaics and quantum devices. It also enables understanding of structural evolution during synthesis, self-assembly and patterning of nanostructures. An additional advantage of aberration correction is that it makes true atomic resolution possible also at lower acceleration voltages. The reduction in the point-spread effect resulting from aberration correction provides higher image contrast and improved signal-to-noise also for electron energy loss and x-ray spectroscopy. The combination higher signal-to-noise ratio, leading to a lower electron dose during observation and data recording, and an optimised acceleration voltage minimises the probability of material structure alteration due to the interaction between the impinging electrons and the specimen during e.g. imaging, diffraction, spectroscopy and in situ studies.

Examples of atomic site specific strain and its effect on catalytic activity [1], three dimensional orientation and distribution of individual dopant species in semiconducting polymers and their effect on conductivity [2], epitaxial growth of Pb on InAs nanowires forming superconductor-semiconductor for quantum devices [3] and growth induced strain in seed-mediated colloidally grown Au nanorods [4] will illustrate the importance of aberration correction for basic understanding of properties of materials and the design of new materials with optimised performance.

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IN-SITU HREM STUDIES OF NANOCATALYSTS AND FERROELECTRIC DOMAINS UNDER ENVIRONMENTAL CONDITIONS

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The dynamic behavior of catalysts under gas condition is the key to understand its mechanism with high performance. We solved the imaging problem of absorbed molecules and established a novel ETEM method to visualize the catalytic reaction at the atomic level, which was successfully employed to precisely determine the active sites for water-gas shift reaction and unveil the catalytic mechanism of this reaction [1]. An unique epitaxial rotation of metal nanoparticle on support during reactions has also been observed [2]. These findings provide valuable information regarding the reaction “black box”, which is critical for understanding the catalytic mechanism and developing high quality catalysts with superior performance.

Here we report also an investigation of the real-time topological transformations of polar structures at an atomic level. [3, 4] The redistribution of charge in various topological structures has been demonstrated under an external bias. This atomic level observation of dynamic evolution reveals the potential of modulating ferroelectric domain patterns in ferroelectric nano-devices.

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MAGNETIC DICHROISM INVESTIGATED IN THE C_S/C_C CORRECTED ELECTRON MICROSCOPE

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Electron magnetic circular dichroism (EMCD), first experimentally demonstrated in a transmission electron microscope (TEM) by Schattchneider et al. [1] is a magnetic spectroscopy technique as analogous to X-ray magnetic circular dichroism (XMCD). In principle EMCD can offer higher spatial resolution due to the short de Broglie wavelength of high-energy electrons compared to XMCD. However, under convergent beam illumination in a scanning TEM, the better spatial resolution, the larger convergent angle, the poorer signal-to-noise ratio (SNR) of EMCD spectra. With a major breakthrough of a spherical/chromatic aberration (C_S/C_C) corrector, the imaging resolution of elastically and inelastically scattered electrons was improved significantly by minimizing the delocalization, [2-4] which allows us to push the spatial resolution of EMCD into atomic level. Here, we make major advances via the ground-breaking combination of spatially resolved electron energy loss spectroscopy (SR-EELS), C_S/C_C correction and EMCD under parallel beam illumination to achieve chemical and magnetic imaging with atomic plane resolution. [5] In the example of $CaTiO_3/SrTiO_3$ superlattice thin films, we demonstrated atomic plane resolved (APR) EELS imaging of Ca and Ti from the individual atomic planes across $CaTiO_3/SrTiO_3$ interfaces. In the example of Sr_2FeMoO_6, we demonstrated APR EMCD imaging of Fe from the individual atomic planes. [5] Combining with advanced capability of structural and chemical imaging by using aberration-corrected TEM, all the information including magnetic polarization, atomic configurations and chemical states can be accessed simultaneously from the very same sample region at the atomic scale. [6]

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IMPORTANCE OF ABERRATION CORRECTION FOR IN SITU HIGH RESOLUTION TEM

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The attempt to obtain meaningful and reproducible in situ recordings, at atomic resolution, of material changes under controlled, externally applied stimuli (eg. temperature, gas, voltage, stress) adds a level of experimental difficulty to TEM observations. Not only do the events need to be followed at very high magnification as they traverse the specimen, but the imaging conditions can be changing sufficiently that they need to be taken into account. The improvements to resolution, image quality, stability, recording and specimen space afforded by aberration-corrected instruments clearly have significant influence on achieving high quality data, as illustrated for instance by NCSI images of cerium and oxygen surface migration on ceria thin films [1]. However additional benefits can also arise, which might not have previously been thought to be important. For example, maintaining the 0.1nm resolution level at lower voltages allowed Koh et al. to study the in situ oxidation of carbon nanotubes (CNT) under field-emitting conditions without electron beam knock-on damage to the CNT's [2,3]. Dionne et al. have revealed in situ the hydrogenation and de-hydrogenation mechanisms of palladium nanoparticales for hydrogen storage applications, using non-centered dark field imaging as a function of hydrogen pressure in an aberration-corrected ETEM [4,5]. Thus, aberration-corrected imaging greatly benefits, in many ways, the practice of in situ TEM experimentation as applied to basic research studies of important scientific and engineering problems.

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IMAGING THE PROPERTIES OF ATOMS AND FIELDS AT THE PICOMETER SCALE INSIDE MATERIALS AND DEVICES

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Electron microscopes use electrons with wavelengths of a few picometers, and are potentially capable of imaging individual atoms in solids at a resolution ultimately set by the intrinsic size of an atom. Even with the rapid advances in aberration-corrector technology, both residual aberrations in the electron lenses and multiple scattering of the incident beam inside the sample, the best resolution possible was an order of magnitude worse than this limit. However, with recent advances in detector technology and phase-retrieval algorithms such as ptychography, the resolution of the electron microscope is now limited only by the dose to the sample, and thermal vibrations of the atoms themselves [1]. These approaches have allowed us to image the detailed vibrational envelopes of individual atom columns as well as locating interstitial dopant atoms that would be hidden by channeling of the probe with conventional imaging modes. Ptychography also provides the sensitivity need to image the internal structures of both magnetic and ferroelectric vortices, skyrmions and merons, including their singular points that are critical for accurately describing the topological properties of these field textures.

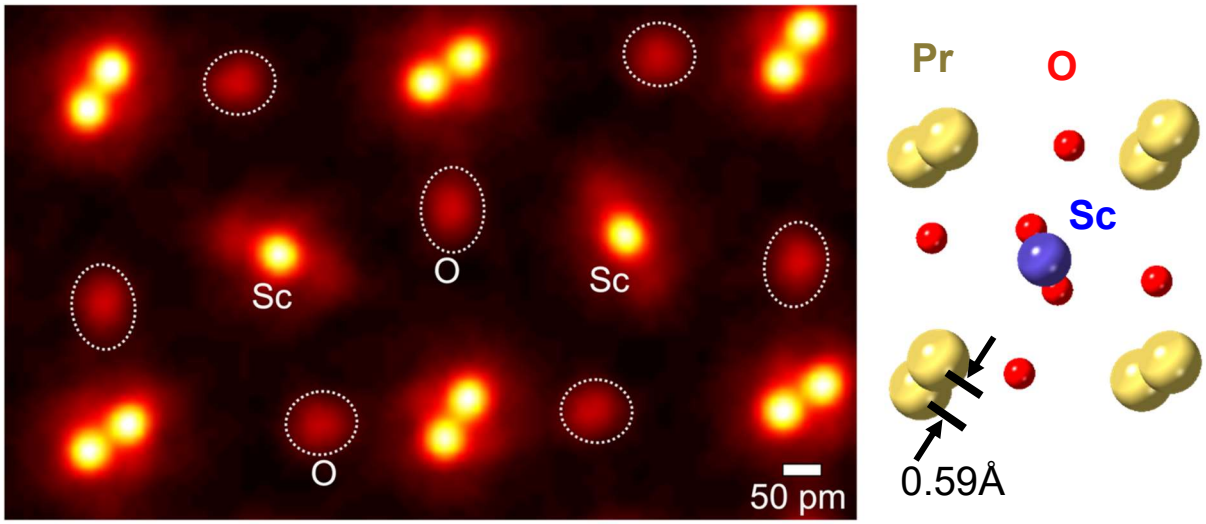

Fig. 1 Multislice Ptychographic reconstruction from a 21-nm thick $DyScO_3$ recorded at 300 keV showing the anisotropic thermal vibrations on the oxygen sites

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QUANTITATIVE HIGH-RESOLUTION TRANSMISSION ELECTRON MICROSCOPY OF ENERGY CONVERSION MATERIALS

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Transmission electron microscopy (TEM) is an essential tool for characterizing the nanoscale and atomic structures of materials, offering critical insight into their fundamental physical properties. Modern TEM has been revolutionized by the development of hardware spherical and chromatic aberration correctors, which, in combination with high-brightness electron sources, monochromators and excellent mechanical and thermal stability, allows deep-sub-Ångstrom spatial resolution to be achieved with high beam current density, bringing new opportunities for tackling topical problems in materials science. In conventional TEM (CTEM), the application of a negative spherical aberration (C_S) coefficient to the objective lens compensated with a positive focus value leads to the invention of a novel imaging mode, negative C_S imaging (NCSI), which can yield strong image contrast that is localized as much as possible on the respective atomic columns for both heavy and light elements [1]. This high image contrast, combined with the easily-corrected image distortion (owing to the parallel beam illumination) and the computer-based quantitative image analysis, allow ultra-high (from several up to 1 picometer) precision measurement of atom positions, which has been applied to a vast variety of materials, including but not limited to (anti-) ferroelectrics, semiconductors, nanocrystals and their interface and surface structures.

In this contribution, we will first introduce the NCSI technique; a particular focus will be on how to achieve quantitative measurements of atomic features. Then we will present our recent studies on quantification of the point defects in energy conversion materials by using quantitative NCSI technique. One of the examples is the direct observation of the off-center rattlers in filled skutterudite thermoelectrics [2], thus providing atomic-scale evidence for solving this long-term debate.

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[2] The author thanks Chun-Lin Jia, Felix Gunkel, Feng Zhang, Da-Wei Wang, Juri Barthel, Xian-Kui Wei, Fengshan Zheng, Penghan Lu, Zhen-Hua Ge, Di Wu and Rafal E. Dunin-Borkowski for collaborations.

UNDERSTANDING ELECTRON-BEAM-SPECIMEN INTERACTIONS AT LOW ACCELERATING VOLTAGES TO TAILOR PROPERTIES OF LOW-DIMENSIONAL INORGANIC AND ORGANIC MATERIALS

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Low-dimensional materials exhibit properties, which often differ strongly from those of the bulk counterparts and offer unique opportunities for new and miniaturized electronic and optical devices [1]. In situ electron microscopy allows, moreover, to functionalize the thin material while imaging. Here we present recent results using our unique chromatic and spherical aberration-corrected *SALVE* instrument both in imaging and spectroscopy modes [2]. We first discuss the formation of defects in two-dimensional inorganic and organic crystals. For transition metal di-chalcogenides (TMDs) we present results at electron energies below the knock-on threshold in the range between 20-80kV and understand the role of electronic excitations. We show that the formation of vacancies is possible at electron voltages nearly half of the knock-on threshold and quantify the damage [3,4]. Further, we analyse in-situ structural and chemical modifications of different freestanding transition metal phosphorus tri-chalcogenides (TMPTs). We predict the displacement thresholds, electronic properties, and the displacement cross-section of single vacancy S and P in all materials by ab-initio calculations and using these results, to understand the observed structural changes. As the TMPTs are often very oxygen-sensitive, they were prepared with the help of a newly-developed polymer-assisted sample preparation method [5]. We also present studies on the structure of two-dimensional polymer crystals and show that an accelerating voltage of 120kV is optimal to image on the molecular level the structure [6]. Furthermore, we present 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 [7]. On the more fundamental base we show that differentiating between the bond nature between two metal atoms is now possible [8].

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LATEST DEVELOPMENTS FROM HITACHI

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The combination of imaging and chemical analysis at the atomic scale is driving research & development in advanced nanomaterials and electronic devices. The ability of a TEM to perform these atomic-scale studies quickly and routinely while acquiring a wide range of analytical data is key to future technological developments. Hitachi's HF5000 FE-TEM, with spherical aberration corrector, has been developed to achieve sub-angstrom(Å) imaging combined with high-sensitivity elemental analysis. The HF5000 builds on features from Hitachi's dedicated STEM HD-2700 including Hitachi's own fully automated aberration corrector, dual symmetrical SDD EDX and Cs-corrected SE imaging.
I will present examples ranging from simultaneous STEM and SEM observations to EDX analysis and in-situ gas and heating experiments.

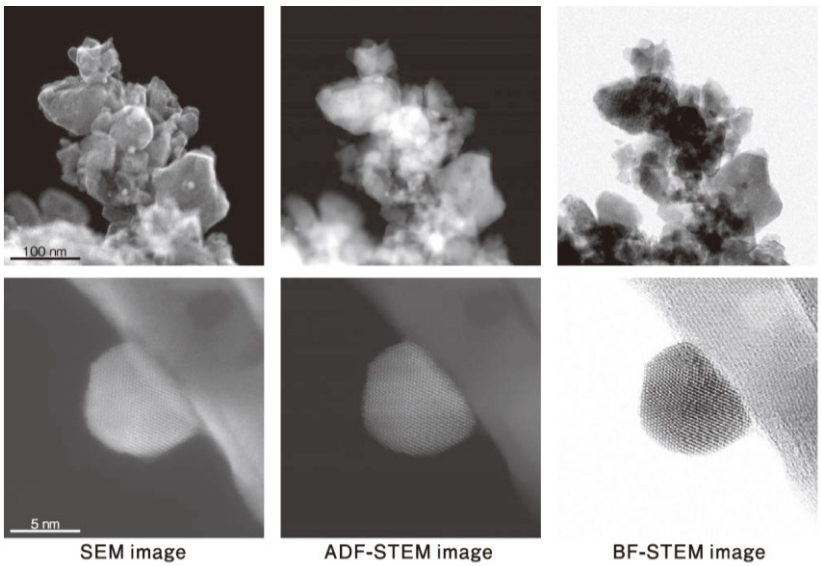

Figure 1: Simultaneous SEM & STEM imaging, thanks to true SE detection capability. This offers correlation of surface and internal information and insights to the 3D structure of a specimen, without the need to perform 3D tomography. Au/CeO2 catalyst SEM/ ADF-/ BF-STEM images (upper row), and respective high resolution Au particle images (lower row).

LET'S DESIGN TOGETHER YOUR NEXT 4D STEM EXPERIMENT

Daniel Stroppa[1*], Matthias Meffert[1], Darya Bachevskaya[1], Luca Piazza[1]

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4D STEM is one of the most exciting TEM techniques today, as it allows the extraction of a great deal of samples' information by the analysis of local electron scattering [1]. In this flash-talk we briefly comment on the most relevant experimental parameters that are relevant to the design of 4D STEM experiments, with focus on pixelated detectors present [2] and next-generation technology.

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[1] C. Ophus, Microscopy and Microanalysis **25**, 563-582 (2019).
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PTYCHOGRAPHY WITH MERLIN EM DETECTOR

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At the University of Glasgow, experiments with hybrid pixel array counting detectors started with a Medipix2 detector [1]. It was clear that direct electron detection and hardware-based electron counting can offer advantageous imaging capabilities. Subsequently, a Medipix3 detector with a Merlin readout system was commercialised as a MerlinEM detector by a collaboration between the University of Glasgow and Quantum Detectors Ltd. With more than 50 systems around the world, the detector has been applied in multiple experimental configurations.

The detector has been mainly applied in scanning transmission electron microscopy (STEM), however, diffraction and EELS applications are also wide-ranging. [2]
In STEM, the ability to collect a full distribution of electrons for each probe position with a millisecond and better timescales can be readily used in DPC [3] and ptychography [4, 5]. Moreover, this wealth of newly available information also enables more quantitative and low-dose approaches to standard techniques and the potential for the development of new ones.

In this short talk, we will focus on examples of ptychography performed with the MerlinEM detector.

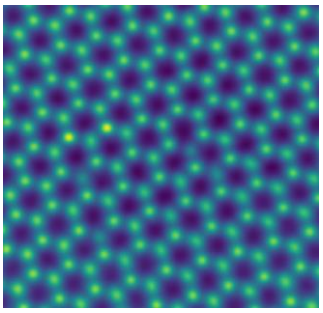

Fig. 1. Ptychography with MerlinEM. Image of single gold atoms in $MoSe_2$. Image courtesy Dr Shoucong Ning, NUS Singapore.

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CEPSTRAL SCANNING TRANSMISSION ELECTRON MICROSCOPY FOR ORDERED AND DISORDERED MATERIALS

J.M. Zuo[1*], S. Pidarpathy[1], Y.T. Shao[2], H.W. Hsiao[1], H. Ni[1], H. Hou[1], D. P. Abraham[3]

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The study of order and disorder is a fundamental theme in materials science and condense matter physics. Examples include the doping of semiconductors, advanced alloys with complex compositions, and nanocrystalline and amorphous materials in energy and environmental technologies, which can be viewed as minute, significant and strong departures from perfect crystallinity, respectively. Transmission electron microscopy (TEM) has played a predominant role in the discovery of disorder within order or order within disorder, from atomic resolution imaging of crystal defects and fluctuation analysis of short- and medium-range ordering in amorphous solids. However, large gaps remain in our ability for the interrogation of significantly and strongly disordered materials. Here we introduce the concept of cepstral STEM (scanning TEM) for filling these gaps [1][2]. The basic idea is to collect large amounts of nanodiffraction patterns using scanning electron nanodiffraction (SEND) and Fourier transform the logarithmic intensities into cepstral patterns to detect the harmonics captured by diffraction and use the quefrency signals for imaging and structural analysis (Fig. 1). This talk will detail this method and demonstrate its effectiveness through applications to semiconductors, high entropy alloys and silicon anode in lithium batteries.

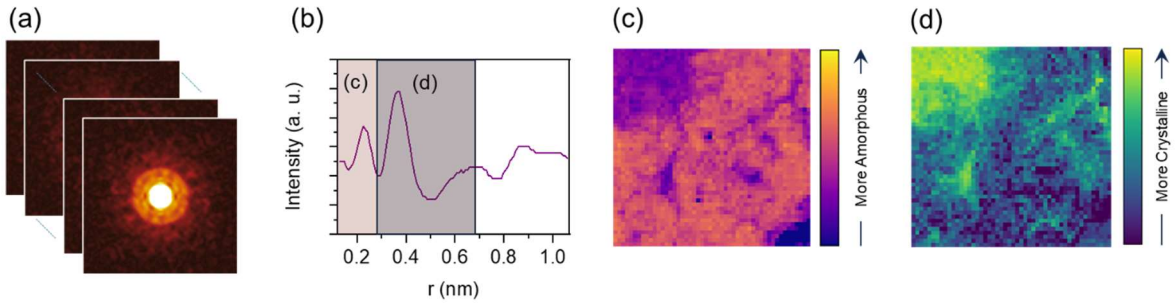

Figure 1. Cepstral STEM for imaging silicon anode. (a) Starting cepstrum stack computed from SEND dataset with (b) corresponding radial cepstrum profile. Image reconstruction of (c) amorphous material and (d) crystalline material.

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EXPLORING MATERIALS STRUCTURE AND PROPERTIES ON THE NANOSCALE BY COMPUTER-AUGMENTED ELECTRON MICROSCOPY

Christoph T. Koch[1*], Marcel Schloz[1], Alberto Eljarrat[1], Sherjeel Shabih[1], Benedikt Haas[1], Thomas C. Pekin[1], Anton Gladyshev[1], Markus Kühbach[1]

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Modern electron microscopes (EMs) can produce images with a spatial resolution of less than 50 pm, electron energy loss spectra with a spectral resolution better than 5 meV, and with electron pulses shorter than 1 fs. Pushing the limits of how precisely experimental data can be acquired comes at the price of increased complexity of operation, increased cost of ownership and with that, the need for making more effective use of beam time. At the same time, although some problems are obviously easier to solve with improved resolution in space, energy, and time, recording data with higher fidelity does not necessarily increase our capability to solve relevant materials science problems more effectively. In this talk we will present some recent examples of increases in the amount and precision of materials information being extracted when advanced data analysis workflows are applied to high-quality experimental data. The examples range from various holographic approaches [1], to the extraction of the bulk dielectric function from relativistic EELS data [2], to the recovery of 3D atomic structure from ptychographic datasets. The reward for developing state of the art EM data reconstruction and knowledge extraction tools grows with the number of people applying them to relevant problems. At the same time, modern data science techniques may potentially be applied to extract otherwise inaccessible information from EM data, if sufficiently large amounts of carefully documented and curated data are available. We will also shortly present solutions recently developed within the FAIRmat project [3] for harmonizing data from various sources and for lowering the threshold for non-experts to apply state of the art EM-data processing tools, without having to install any software on their local computer.

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[1] M. Schloz, T.C. Pekin, et al., "*Overcoming information reduced data and experimentally uncertain parameters in ptychography with regularized optimization*", Optics Express Vol. 28 (2021) 28306
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ROLE OF IONIZATION IN IMAGING AND SPECTROSCOPY UTILIZING FAST ELECTRONS THAT HAVE EXCITED PHONONS

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Atomic resolution scanning transmission electron microscopy, based on counting fast electrons that have been scattered to large angles after exciting a phonon, so-called high-angle annular dark-field (HAADF) imaging, is widely used in materials science. Recently atomic resolution phonon spectroscopy has been demonstrated. In both cases experiments are usually modeled taking into account only elastic scattering and the inelastic scattering due to phonon excitation. However, other inelastic processes, such as plasmon excitation and single electron excitation, also play a role. We apply extended multislice simulations using the *μSTEM* software [1] in order to investigate the role of ionization and its influence on imaging and spectroscopy. Inelastic scattering due to ionization is mainly forward peaked, which has implications for phonon spectroscopy with a detector in the forward direction. Nevertheless, a substantial fraction of electrons scattered by phonon excitation to larger angles have also lost significant amounts of energy due to also being involved in an ionization event. We find that when properly normalized, simulations predict an optimum sample thickness for maximum intensity in phonon spectroscopy utilizing electrons scattered to large angles, and that ignoring the contribution of large-angle ionization scattering in simulations can lead to a bias in the quantitative analysis HAADF STEM images. In general, the significance of large angle scattering due to ionization increases for lower beam energy. Consistent with experimental data [2], the simulations also show that ionization scattering is stronger for light elements than for heavy elements.

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2D MATERIAL HETEROSTRUCTURE LIQUID CELLS : A PLATFORM FOR ATOMIC RESOLUTION STEM IMAGING WITH LIQUIDS.

S J Haigh[1], N. Clark[1], Y Zou[1], D Kelly[1], M Lozada Higalgo[1], R Gorbachev[1]

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Liquid-phase transmission electron microscopy (TEM) offers a unique combination of nanometer spatial resolution and millisecond temporal resolution. Conventional liquid cell holders for scanning transmission electron microscopy (STEM) imaging and analysis are powerful tools for probing nanoscale behaviour at the solid-liquid interface. Nonetheless the presence of silicon nitride membranes, tens of nanometers in the thickness, as well as a relatively thick liquid layer (often >100 nm) compromises the instruments ultimate imaging resolution as well as the sensitivity of spectroscopy techniques. Graphene liquid cells provide excellent atomic resolution imaging and are fully compatible with spectroscopic analysis, but their construction often lacks reproducibility and the hermetic seal of the cells often fail with prolonged imaging. This talk I will discuss the 2D heterostructure approach to liquid cell STEM, which combines atomic resolution imaging, sensitive spectroscopic analysis with robust imaging capabilities even at high spatial resolution [1]. The platform builds on established expertise within Manchester's National Graphene Institute of 2D materials nanochannel fluidics [2]. I will show how we have been applying this approach for advancing our understanding of solid-liquid interfaces and liquid phase chemical reactions. We demonstrate the potential of the approach for in situ atomic resolution imaging and elemental analysis, by investigating the time evolution of calcium carbonate synthesis, from the earliest stages of liquid-liquid phase separated nanodroplet precursors to crystalline calcite [3]. I will also demonstrate the application of the technique to studying the dynamic motion and preferred resting sites of single atom metallic species on surfaces in aqueous salt solution, revealing a closer match to theoretical calculations than is achievable with ex situ vacuum phase imaging. The approach provides exciting new opportunities for studying the dynamic behaviour of nanomaterials interacting with liquids. [4]

References:

[1] Nanometer resolution elemental mapping in graphene-based TEM liquid cells, Kelly et al Nano Letters (2018) **18 (2)**, 1168-1174.

[2] Capillary condensation under atomic-scale confinement, Q Yang et al, Nature 588 (7837), 250-253 (2020); Ballistic molecular transport through two-dimensional channels, A Keerthi et al, Nature **558** (7710), 420-424 (2018).

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THICKNESS DEPENDENT COATING BREAKING DURING BATTERY CYCLING BY *IN SITU* TEM

Shibabrata Basak[1,2,*], Amir H. Tavabi[2], Chandramohan George[3], Joachim Mayer[2], Rafal E. Dunin-Borkowski[2], Rüdiger-A. Eichel[1]

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[2]Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons and Peter Grünberg Institute, Forschungszentrum Jülich GmbH, 52425 Jülich, Germany
[3] Dyson School of Design Engineering, Imperial College London, SW7 2AZ, London, UK

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Si has gained considerable attraction as an anode material in Li-ion batteries, in which composite electrodes that have different amounts of Si and carbon (with a theoretical capacity for Si of above ~4000 $mAhg^{-1}$ based on a $Li_{22}S_5$ stoichiometry) offer an enhancement in capacity when compared with pure graphite powder (~370 $mAhg^{-1}$). However, the breaking of Si particles because of strain caused by repeated lithiation (during battery charge/discharge cycles) limits exploitation of their full capacity and rate capabilities. In particular, the large volume change (>300%) of Si particles upon lithiation can result in particle pulverisation, accompanied by excessive solid electrolyte interphase (SEI) formation. The size-dependent cracking and shape-dependent lithiation behavior of Si has been reported, which provided indications about how the mechanical characteristics of Si particles evolve during battery cycling and affect their electrochemical performance. Even nanosized particles that do not easily break [1] during charge/discharge cycles owing to improved strain relaxation are still plagued by excessive electrolyte consumption associated with multiple SEI formation events, which causes rapid depletion of cyclable Li. Conformal coating of silicon (Si) anode particles is a common strategy for improving their mechanical integrity, to mitigate battery capacity fading due to particle volume expansion, which can result in particle crumbling due to lithiation induced strain and excessive solid–electrolyte interface formation. Here, we use *in situ* transmission electron microscopy in an open cell to show that TiO_2 coatings on Si/SiO_2 particles undergo thickness dependent rupture on battery cycling where thicker coatings crumble more readily than thinner (~5 nm) coatings, which corroborates the difference in their capacities [2].

References:
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NOVEL THEORY OF THE STRUCTURE OF THE ELECTRON

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A novel theory is outlined which represents a promising alternative to the Standard Model and to the Dirac theory of the electron and explains the origin of the spin, the mass, and the sign of the charge of elementary particles. Massless particles with opposite angular momentum attract each other forming massive particles. In particular, the electron is a four-dimensional top composed of two photons with opposite helicity. The force between the constituents of the electron originates from a hyper-symmetric four-dimensional (4D) potential, which is a solution of the 4D Poisson equation. The 4D rotational Hamiltonian of the composite system depends on the 4D radius and on three angles, one of which is imaginary. This angle describes the rotation in Minkowski space of the time-like axis with respect to one of the 3D axis representing a Lorentz transformation. The results of the novel approach show that massive particles can only be stable if they have a characteristic four-dimensional elementary radius. This radius is an eigenvalue of the rotational energy, which determines the mass of the particle. The sign of the charge differs for the left-handed and the right-handed space-time rotation. The eigenvalues of the 4D angular are related with the Compton wavelength and determine the mass of the compound particle. Hence, the novel theory explains the origin of mass without needing the Higgs field. We demonstrate the feasibility of the novel theory by revisiting the hydrogen atom. The resulting fine-structure formula differs from that derived by the Dirac theory because it accounts for the Lamb shift.

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POSTER PROGRAMME

EMS Extension

MONDAY MAY 9TH, 2022

POSTER SESSION A — **MATERIALS SCIENCE**
"Vaalsbroekerhof" castle ground floor

PA01 ABERRATION-CORRECTED STEM CHARACTERIZATION OF INTERCALATED LaCoO3/SrTiO3 LAYERS FORMING NANOSTRUCTURED THERMOELECTRIC SUPERLATTICES

Enrique Carbo-Argibay, International Iberian Nanotechnology Laboratory, (Portugal)

PA02 ATOMIC SCALE OBSERVATIONS OF AG SEGREGATION IN A HIGH ANGLE GRAIN BOUNDARY IN CU

Lena Frommeyer, Max-Planck-Institut für Eisenforschung GmbH Germany

PA03 THERMALLY ACTIVATED MICROSTRUCTURAL REFINEMENT IN □-TIAL ALLOYED WITH NB

Heike Gabrisch, Helmholtz-Zentrum Hereon (Germany)

PA04 STRUCTURE ANALYSIS OF MOLECULAR MATERIALS WITH HIGH NONLINEAR OPTICAL PROPERTIES

Johannes Haust, Philipps-University Marburg (Germany)

PA05 ATOMIC-SCALE DETERMINATION OF CATION AND MAGNETIC ORDER OF THE TRIPLE PEROVSKITE $SR_3FE_2REO_9$

Ping-Luen Ho, University of Oxford (UK)

PA06 CHARACTERIZATION AND IMPACT OF STACKING FAULTS IN YTTRIUM DOPED BARIUM ZIRCONATE

Dylan Jennings, Forschungszentrum Jülich (Germany)

PA07 FOCUSED ION BEAM MILLING TECHNIQUES FOR TEM SPECIMEN PREPARATION OF SOLID BULK SAMPLES

Lidia Kibkalo, Forschungszentrum Jülich (Germany)

PA08 INTERFACE CHARACTERIZATION OF COLD SINTERED BaZr0.7Ce0.2Y0.1O3-δ PEROVSKITE MEMBRANES

Moritz Kindelmann, RWTH Aachen University (Germany)

PA09 STRUCTURAL CHARACTERIZATION OF 2D MATERIALS AND NANOSTRUCTURES

Oliver Maßmeyer, Philipps-Universität Marburg (Germany)

PA10 INVESTIGATION OF BULK ZNPD CATALYSTS USING HIGH RESOLUTION ELECTRON MICROSCOPY

Ansgar Meise, Forschungszentrum Jülich (Germany)

PA11 THE EFFECT OF EPITAXIAL GROWTH DIRECTION ON THE PROPERTIES OF HIGHLY DOPED P-ALGAAS/N-GAINP INTERFACES

Vita Mergner, Forschungszentrum Jülich (Germany)

PA12 CORRELATING 3D GRAIN ORIENTATION MAPPING WITH ATOM PROBE TOMOGRAPHY IN NANOCRYSTALLINE ALLOYS

Saurabh M. Das, Max-Planck-Institut für Eisenforschung (Germany)

PA13 UNDERSTANDING GAS ADSORPTION OF PAN-BASED CARBON NANOFIBERS

Junbeom Park, Forschungszentrum Jülich (Germany)

PA14 INTERMETALLICS IDENTIFICATION IN LEAN MG ALLOYS BY COMBINING EDS, DIFFRACTION AND HAADF STEM

Robin Schäublin, ETH Zürich (Ch)

PA15 STRUCTURE AND DEFECT ANALYSIS OF SUPERCONDUCTING NbN THIN-LAYERS

Michael Steiner, Johannes Kepler University (Germany)

PA16 ATOMIC-SCALE TUNING OF CHARGE DISTRIBUTION BY STRAIN ENGINEERING IN COMPLEX OXIDE HETEROSTRUCTURES

Eren Suyolcu, Max Planck Institute for Solid State Research (Germany)

PA17 NANOSTRUCTURE OF METAL POLY(HEPTAZINE IMIDES)

Nadezda Tarakina, MPI of Colloids and Interfaces (Germany)

PA18 LOW-DOSE PERFORMANCE OF IDPC-STEM

Eric Van Cappellen, Thermo Fisher Scientific (NL)

PA19 UNVEILING ATOMIC-SCALE CHARGE DISTRIBUTION AT COMPLEX OXIDE INTERFACES

Hongguang Wang, Max Planck Institute for Solid State Research (Germany)

PA20 CONTINUOUS ILLUMINATION PICOSECOND IMAGING OF MAGNETISATION DYNAMICS IN A TRANSMISSION ELECTRON MICROSCOPE

T. Weßels, University of Duisburg-Essen (Germany)

PA21 MAPPING OXYGEN COLUMNS IN MAGNETITE CRYSTALS USING DIFFERENTIAL PHASE CONTRAST STEM IMAGING

Xuyang Zhou, Max-Planck-Institut für Eisenforschung (Germany)

POSTER SESSION B — ELECTRON OPTICS
"Jachtkelder" castle ground floor

PB01 PERFORMANCE ESTIMATES FOR A GROUND-POTENTIAL MONOCHROMATOR

Felix Börrnert, CEOS GmbH (Germany)

PB02 CEFID: A FLEXIBLE PLATFORM FOR SPECTROSCOPIC EXPERIMENTS

Giulio Guzzinati, CEOS GmbH (Germany)

PB03 HIGH COHERENCE ELECTRON SOURCES FOR ULTRAFAST TRANSMISSION ELECTRON MICROSCOPY BASED ON PHOTOASSISTED FIELD EMISSION

Rudolf Haindl, Max Planck Institute for Multidisciplinary Sciences (Germany)

PB04 COMBINATION OF ABERRATION CORRECTION WITH A THIN-FILM ZERNIKE PHASE PLATE

Simon Hettler, Universidad de Zaragoza (Spain)

PB05 THE NEXT GENERATION OF IN-SITU OPEN CELL ENVIRONMENTAL DOUBLE ABERRATION CORRECTED E-(S)TEM: ARTEMIS

Leonardo Lari, University of York (UK)

PB06 THE USER ADJUSTABLE POLE-PIECE – A TEM HEART TRANSPLANT

Patrick McBean, Trinity College Dublin (Ireland)

PB07 ENERGY DISCRIMINATION USING CMOS-COMPATIBLE ARRAYS OF PHOTODIODES IN SCANNING ELECTRON MICROSCOPY

Jorge Sáenz-Noval, University of Cádiz (Spain)

PB08 CAN A PROGRAMMABLE PHASE PLATE SERVE AS AN ADAPTIVE Cs CORRECTOR IN THE TEM?

Francisco Vega, University of Antwerp (Belgium)

TUESDAY MAY 10TH, 2022

POSTER SESSION C — **METHOD DEVELOPMENT & DATA SCIENCE / THEORY**
"Vaalsbroekerhof" castle ground floor

PC01 SINGLE SCAN STEM EMCD IN 3-BEAM ORIENTATION

Hasan Ali, Stockholm University (Sweden)

PC02 REDUCING ELECTRON BEAM DAMAGE THROUGH ALTERNATIVE STEM SCANNING STRATEGIES

Armand Béché, University of Antwerp (Belgium)

PC03 THE EFFECT OF DYNAMICAL SCATTERING ON PHASE RETRIEVAL IN 4D-STEM

Laura Clark, University of Leeds (UK)

PC04 IMPROVEMENTS IN DATA PROCESSING OF STEM-DPC WITH DISTORTED BRIGHT FIELD DISK

Sivert Dagenborg, NTNU (Norway)

PC05 TRANSIENT HEXATIC PHASE OBSERVED BY HIGH-COHERENCE ULTRAFAST ELECTRON DIFFRACTION

Till Domröse, Max Planck Institute for Multidisciplinary Sciences (Germany)

PC06 OPTIMIZATION OF IMAGING CONDITIONS FOR COMPOSITION DETERMINATION BY ANNULAR DARK FIELD STEM

Saleh Firoozabadi, Philipps-Universität Marburg (Germany)

PC07 DIRECT ELECTRON PTYCHOGRAPHY OF STRONG PHASE OBJECTS

Chuang Gao, University of Antwerp (Belgium)

PC08 ATOMIC RESOLUTION STEM MAPPING OF MOMENTUM TRANSFER AND ELECTRON ENERGY LOSS

Benedikt Haas, Humboldt-Universität zu Berlin (Germany)

PC09 PTYCHOGRAPHY FOR CHARGE TRANSFER SENSITIVITY AND 3D STRUCTURE DETERMINATION IN 2D MATERIALS

Christoph Hofer, University of Antwerp (Belgium)

PC10 FAST 4D STEM ACQUISITION WITH EVENT BASED ELECTRON DETECTION

Daen Jannis, University of Antwerp (Belgium)

PC11 MAGNETIC FIELD MAPPING OF 2-DIMENSIONAL AND 3-DIMENSIONAL FRUSTRATED MAGNETS USING OFF-AXIS ELECTRON HOLOGRAPHY

András Kovács, Forschungszentrum Jülich (Germany)

PC12 APERTURED EMCD AT ATOMIC SIZED ELECTRON BEAMS

Klaus Leifer, Uppsala University (Sweden)

PC13 OPTIMISING DOSE AND SAMPLING EFFICIENCY IN PHASE CONTRAST ELECTRON MICROSCOPY

Peng-Han Lu, Forschungszentrum Jülich (Germany)

PC14 A NEW APPROACH FOR 3D QUANTITATIVE STEM USING DEFOCUS CORRECTED ELECTRON PTYCHOGRAPHY

Ali Mostaed, University of Oxford (UK)

PC15 PRECESSION SEGMENTATION TO INCREASE SPED IMAGE RESOLUTION

Gregory Nordahl Norwegian University of Science and Technology (Norway)

PC16 REAL TIME INTEGRATION CENTER OF MASS (RICOM) RECONSTRUCTION FOR 4D-STEM

Chu-Ping Yu, University of Antwerp (Belgium)

PC17 FAST AUTOMATIC ANALYTICAL PARTICLE ANALYSIS USING AN AI GUIDED SMART SCAN STRATEGY

Bert Freitag, Thermo Fisher Scientific (NL)

PC18 PHASE OBJECT RECONSTRUCTION FOR 4D-STEM WITH MACHINE LEARNING

Thomas Friedrich, University of Antwerp (Belgium)

PC19 4D-STEM FOR 3D STEREO IMAGING AND AUTOMATIC 3D RECONSTRUCTION OF CURVILINEAR OBJECTS

Gulnaz Ganeeva, [1]Electron Spectrometry and Microscopy Laboratory (CH)

PC20 AUTOMATED ANALYSIS OF HIGH-RESOLUTION TRANSMISSION ELECTRON MICROSCOPY IMAGES OF METALLIC NANOPARTICLES USING NEURAL NETWORKS

Nina Gumbiowski, University of Duisburg-Essen (Germany)

PC21 AUTOMATED ALIGNMENTS WITH REAL-TIME ELECTRON RAY TRACING

David Landers, University of Limerick (Ireland)

PC22 LARGE AREA MULTISLICE SIMULATIONS A NOVEL APPROACH

Stephen Mullins, University of Limerick (Ireland)

PC23 ADAPTABLE AND AUTOMATED ANALYSIS FOR VARIOUS NANOPARTICLE SYSTEMS USING NNPIPE

Kevin Treder, University of Oxford (UK)

PC24 SYNTHETIC DATA, MACHINE LEARNING AND EDGE DETECTION TO MEASURE PARTICLES IN TRANSMISSION ELECTRON MICROSCOPY

Eoin Walsh, University of Limerick (Ireland)

POSTER SESSION D — LIFE SCIENCE
"Jachtkelder" castle ground floor

PD01 ENHANCING THE DOSE LIMITED SIGNAL FROM THICK BIOLOGICAL SPECIMENS WITH CC CORRECTION

Joshua Dickerson, MRC Laboratory of Molecular Biology (UK)

PD02 THE 3D STRUCTURE OF LIPIDIC FIBRILS OF α-SYNUCLEIN

Benedikt Frieg, Forschungszentrum Jülich (Germany)

PD03 α-SYNUCLEIN POLYMORPHS IN NEURODEGENERATION

James A. Geraets, Forschungszentrum Jülich (Germany)

PD04 MICROSTRUCTURAL CHARACTERIZATION OF CELLULOSE NANOFIBRES USING SCANNING ELECTRON DIFFRACTION

Mathias Nero, Stockholm University (Sweden)

PD05 VISUALISATION OF NATIVE ATG9 COMPLEXES USING IN SITU CRYO-EM

Claire Ortmann de Percin Northumberland, Forschungszentrum Jülich (Germany)

PD06 MEMBRANE ANALYSIS TOOLKIT – A QUANTITATIVE WAY TO ANALYSE PROTEIN LIPID MIXTURES FROM CRYO-EM IMAGES

Philipp Schönnenbeck Forschungszentrum Jülich (Germany)

PD07 CRYO-EM STRUCTURE OF A□UPP(1-42)□19-24 FIBRILS

Mara Zielinski, Forschungszentrum Jülich (Germany)

MONDAY MAY 9TH, 2022

AND

TUESDAY MAY 10TH, 2022

POSTER SESSION S — **SPONSORS**
"Vaalsbroekerhof" castle ground floor

PS01	HYBRID-PIXEL DETECTORS FOR TEM BY DECTRIS Luca Piazza, DECTRIS Ltd. (CH)
PS02	PULSED LASER INTEGRATION AND ELECTROSTRATIC SYSTEMS FOR DODE MODULATION AND TEMPORAL RESOLUTION TEM Manuel Reinhard, JEOL GmbH (Germany)
PS03	CLIMATE - EXPOSE YOUR SAMPLE TO A CONTROLLED GAS ENVIRONMENT WITH PRESSURES UP TO 2 BAR AND SWITCH GASSES IN SECONDS Eva Bladt, DENSsolutions BV (NL)
PS04	MERLIN EM, HYBRID PIXEL ARRAY COUNTING DETECTOR FOR TRANSMISSION ELECTRON MICROSCOPY Matus Krajnak, Quantum Detectors Ltd. (UK)
PS05	DYNAMICAL STUDIES OF ADVANCED MATERIALS COMBINING IN SITU 4D-sPED MAPPING AND PIXELATED DETECTORS Alan Robins, NanoMEGAS SPRL (Belgium)
PS06	AXON DOSE: A MACHINE VISION SOLUTION FOR ACCURATE, QUANTIFIABLE DOSE MANAGEMENT IN THE TRANSMISSION ELECTRON MICROSCOPE David Nackashi, Protochips Inc. (USA)
PS07	IMPROVING SAMPLE DISPERSION WITH AN ADDITIONAL ULTRATHIN CARBON LAYER Claire Naylor, Quantifoil (Germany)

PS08 DEDICATED WORKFLOWS FOR ATOMIC STEM BASED ANALYSIS ON LITHIUM BASED BATTERY MATERIALS

Eric Van Cappellen, Thermo Fisher Scientific (USA)

PS09 QUANTITATIVE MAPPING OF LITHIUM IN THE SEM USING COMPOSITION BY DIFFERENCE METHOD

Saleh Gorji, Gatan Inc. (USA)

PS10 LIQUID HELIUM CRYO-TEM

Denys Sutter, condenZero GmbH (CH)

PICO 2022

25 Years of Aberration Correction

SEVENTH CONFERENCE ON FRONTIERS OF ABERRATION CORRECTED ELECTRON MICROSCOPY

celebrating

25 years of aberration correction

Kasteel Vaalsbroek

May 08th – May 12th

2022

POSTER ABSTRACTS

EMS Extension

ABERRATION-CORRECTED STEM CHARACTERIZATION OF INTERCALATED $LaCoO_3/SrTiO_3$ LAYERS FORMING NANOSTRUCTURED THERMOELECTRIC SUPERLATTICES

Enrique Carbo-Argibay[1*], David Bugallo[2], Eric Langenberg[3], Noa Varela Dominguez[2], Adolfo Otero Fumega[4,5], Victor Pardo[4], Irene Lucas[6], Luis Morellón[6], and Francisco Rivadulla[2]

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The contribution of coherent and incoherent phonons to thermal conductivity [1] of nanostructured superlattices has been studied in a set of $LaCoO_3/SrTiO_3$ (LCO/STO) multilayer samples by tuning their periodicities and thicknesses. The combination of Aberration-Corrected Scanning Transmission Electron Microscopy (AC-STEM) characterization and Focused Ion Beam (FIB) as sample preparation method is really powerful to understand the structure and chemical composition of these structures at atomic level. In this work [2], we have followed that approach to prepare several TEM lamellas of nanostructured superlattices made of intercalated LCO/STO layers. A thorough AC-STEM analysis of those TEM lamellas, combining imaging and energy dispersive X-Ray spectroscopy (EDX), has been carried out in order to elucidate the multilayer structure of these materials and correlates it with their thermoelectric properties, concluding that small variations in the periodicity and thickness of the layers can be used to control the thermal conductivity of these superlattices.

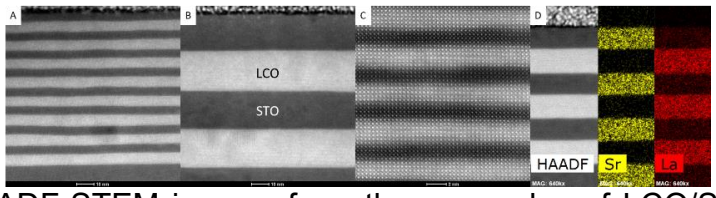

Fig. 1: (A-C) HAADF-STEM images from three samples of LCO/STO with different thickness and periodicity. (D) EDX maps showing the distribution of Sr and La.

References:
[1] M Maldovan, Nat. Mater., Vol. 14, (2015), 667.
[2] D Bugallo *et al*, J. Phys. Chem. Lett., Vol. 12, (2021), 11878.

ATOMIC SCALE OBSERVATIONS OF AG SEGREGATION IN A HIGH ANGLE GRAIN BOUNDARY IN CU

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Segregation to grain boundaries (GBs) can lead to dramatic changes of materials properties [1]. One prominent example is bismuth GB segregation in copper causing GB embrittlement [2]. Thermodynamic models can predict and explain segregation effects in general. However, atomistic origins and atomic scale observations of segregation within the GB structure are rarely explored since they can only be investigated by atomic resolution imaging and spectroscopy. In this work, we studied Ag segregation in Cu as a model system due to its large difference in atomic number and immiscibility, which acts as a driving force for Ag to segregate to the GB. An epitaxially grown <111> Cu thin film was used to analyze the atomic structure of pure and segregated Σ37c <111> {347} tilt grain boundaries. Site-specific lift outs of such GBs obtained by using a focused ion beam were investigated with aberration-corrected HAADF-STEM. As shown in Fig. 1, the clean GB structure is composed of a double-square (green) and a zipper unit (blue). Controlled segregation was achieved by depositing a 100 nm-thin Ag layer on the Cu film and subsequent annealing at 600°C for 16h. Interestingly, the Ag enriched GB adopt similar atomic structures (Fig. 2). Near atomic-resolution energy dispersive X-ray spectroscopy (EDS) was performed to resolve the Ag positions within the GB. Ag enrichment was found to be in a periodic arrangement with maxima each 0.85 nm. As shown in Fig. 2, the intensity peaks coincide with the zipper unit. Atomistic simulations are used to explore the underlying mechanisms leading to the observed Ag segregation pattern. Its impact on interfacial properties will be discussed.

Acknowledgment: This project has received funding from the European Research Council (Grant agreement No. 787446; GB-CORRELATE).

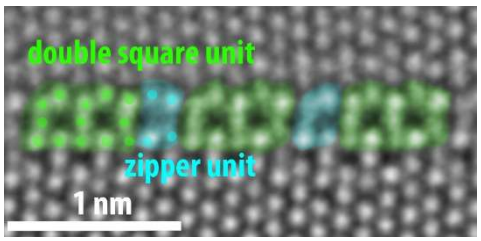

Fig. 1 HAADF-STEM image of a Σ37c <111> {347} GB in pure Cu.

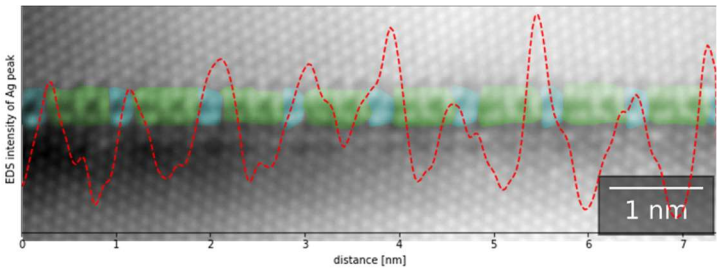

Fig. 2. HAADF-STEM image of a Σ37c <111> {347} Cu GB, overlayed with the EDS intensity of Ag.

References:
[1] P. Lejcek, Springer Series in Materials Science **136** (2010).
[2] E.D. Hondeos, D.McLean, Philosophical Magazine **29**, 771-796 (1974).

THERMALLY ACTIVATED MICROSTRUCTURAL REFINEMENT IN γ-TIAL ALLOYED WITH NB

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γ-Titanium aluminides are structural materials for applications in turbines of aero-engines. At Al contents between 40 - 48 at.-% their microstructure consists of the two ordered phases, α_2 (hexagonal) and γ (tetragonal) that form lamellar colonies. Upon alloying with Nb, the α_2-phase becomes metastable. In the temperature range between 450 - 680°C continuous transformations involving lattice distortions and diffusion are observed in α_2-lamellae. The product is a mixture of hexagonal α_2-phase and orthorhombic O-phase. Measurements in the alloy Ti-42Al-8.5Nb show, that the size of O-phase domains decreases during annealing at 550 °C while simultaneously Nb partitions to O-phase domains.
The effect of these microstructural changes on mechanical properties has been monitored by in-situ-heating Resonant Ultrasound Spectroscopy [2]. A small but distinct increase in the elastic constants is detected. The correlation between microstructure and elastic constants shows that the transformation from hexagonal to orthorhombic symmetry in the beginning of the annealing experiment has the largest impact on the observed increase.

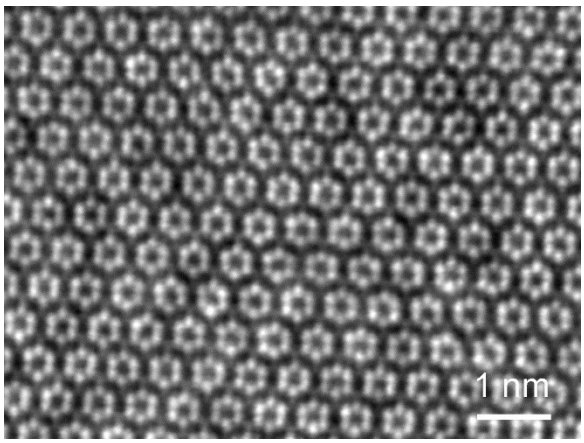

Fig. 1 Hexagonal α_2 phase in [0001] projection.

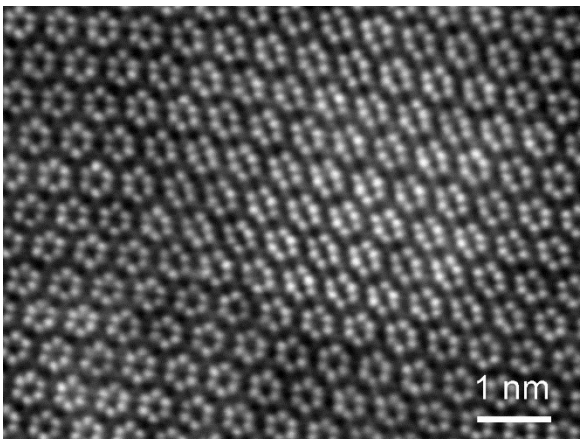

Fig. 2. [0001] projection of α_2 and O-phases after transformation at 550°C.

References:
[1] Marcus Willi Rackel et al., Acta Materialia **121**, 343-351 (2016).
[2] P. Sedlák et al., Benchmark Examples Experimental Mechanics **54** (6), 1073-1085 (2014).

STRUCTURE ANALYSIS OF MOLECULAR MATERIALS WITH HIGH NONLINEAR OPTICAL PROPERTIES

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Adamantane-type molecular cluster materials with the general formula $[(RT)_4S_6]$ (R = organic substituent; T = Ge, Sn) show high nonlinear optical properties[1]. Depending on R and T either second harmonic generation or efficient, directional white-light emission[2] (WLE) of so far unknown origin occur. We study the structure of these materials by means of transmission electron microscopy (TEM), because it opens the path for spatially resolved investigations of nanoscale volumes. Since the majority of the materials that show the WLE have no crystalline structure, the so called pair distribution functions g(r) are obtained from TEM diffraction patterns as shown in figure 1. First the scattering background is corrected[3] and subtracted to get the structure factor S(k). The diffuse rings visible in the diffraction pattern lead to the peaks in S(k) and these are linked to the structure of the sample. g(r) is then the Fourier transformation (FT) of S(k) and as a probability function of the spacing between the atoms contains information about frequently occurring next neighbor distances. Our results are in a good agreement with previous density functional theory (DFT) calculations[1] as well as X-ray diffraction measurements[4]. With our method we can now investigate the relationship between structural and optical properties for better understanding the nature of the WLE.

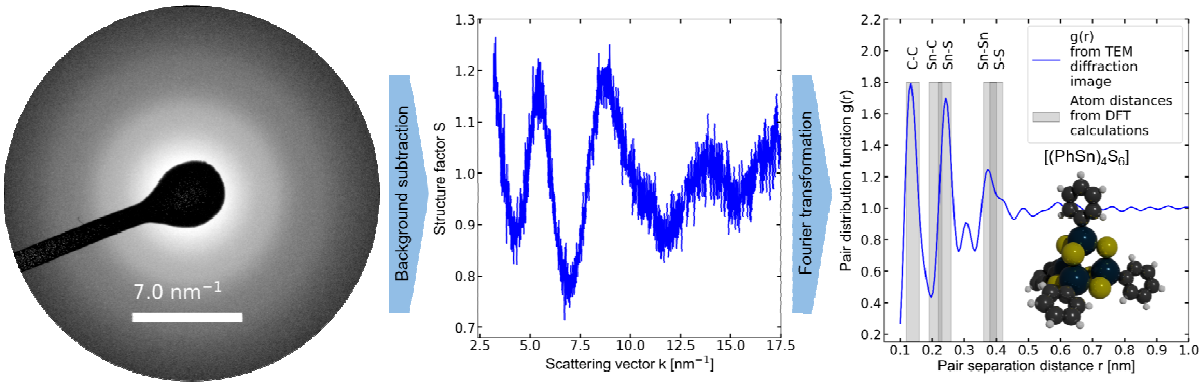

Fig 1 An examplary procedure for obtaining g(r) for a $[(PhSn)_4S_6]$ sample (Ph = phenyl). The diffraction pattern is log scaled for better visualization.

References:
[1] Dehnen, S. *et al. ChemPhotoChem* (2021).
[2] Rosemann, N. W. *et al. Science (80-.).* **352**, 1301–1304 (2016).
[3] Tran, D. T., Svensson, G. & Tai, C.-W. *J. Appl. Crystallogr.* **50**, 304–312 (2017).
[4] Klee, B. D. *et al. Phys. Status Solidi Basic Res.* **255**, 1–6 (2018).

ATOMIC-SCALE DETERMINATION OF CATION AND MAGNETIC ORDER OF THE TRIPLE PEROVSKITE $SR_3FE_2REO_9$

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Perovskite oxides (ABO_3) that have various magnetic cations located at two or three crystallographically-equivalent B sites have attracted considerable interest as a result of their tunable magnetic properties [1]. Nanoscale inhomogeneity in cation order on the B sites can lead to different magnetic ground states and electronic band structures in local sample regions [2,3]. Here, we determine the cation order on the atomic scale in a nanosized $Sr_3Fe_2ReO_9$ phase that has a triple perovskite structure using aberration-corrected analytical transmission electron microscopy (TEM), revealing that the Fe and Re cations form a layer repeat with -[Fe-Fe-Re]$_n$- sequences in the pseudo-cubic $\langle 111 \rangle$ direction. To the best of our knowledge, this triple perovskite $Sr_3Fe_2ReO_9$ phase has not been reported before. Based on a relaxed theoretical model that is consistent with the experimental images, density functional theory calculations are performed to determine the magnetic ground states and exchange parameters of the newly-discovered $Sr_3Fe_2ReO_9$ phase, in which the nearest-neighbour Fe and Re cations are coupled antiferromagnetically. This combination of aberration-corrected analytical TEM and *ab initio* calculations provides physical insight into cation order and magnetic coupling in perovskite oxides at the atomic level.

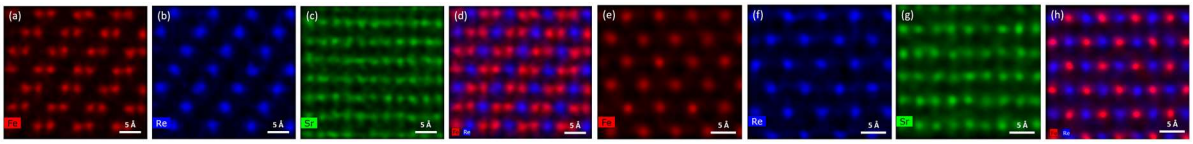

Figure 1. Atomic-resolution elemental mapping of $Sr_3Fe_2ReO_9$ (a-d) and Sr_2FeReO_6 (e-h) perovskite structures viewed along $\langle 011 \rangle_{pc}$. These Fe maps (red), Re maps (blue), Sr maps (green), and mixed B sites of cation ordered maps show obvious differences in their positions of cations with colours, Fe-Fe-Re and Fe-Re, respectively.

References:
[1] S. Vasala, M. Karppinen, Prog Solid State Chem **43**, 1-36 (2015).
[2] T-W Lim, S-D Kim, K-D Sung et al., Sci Rep **6**, 19746 (2016).
[3] P.-L. Ho, C.-P.Yu, Q. Zhang et al., Ultramicroscopy **193**, 137-142 (2018).

CHARACTERIZATION AND IMPACT OF STACKING FAULTS IN YTTRIUM DOPED BARIUM ZIRCONATE

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Yttrium doped barium zirconate (BZY) is a perovskite ceramic of interest for a variety of energy applications, including as a catalyst support material and as an electrolyte for solid state fuel and electrolytic cells. Yttrium doping introduces oxygen vacancies into the structure, allowing for the incorporation of hydroxyl groups and conduction of protons throughout the material. Of great interest to material performance is the presence and character of two-dimensional defects in the material, which often (such as in the case of grain boundaries) are blocking to protonic conduction. The work presented here discusses the discovery of stacking faults in conventionally sintered BZY20 ($BaZr_{0.8}Y_{0.2}O_{3-\delta}$), a set of defects that have not been previously observed in this material composition. Characterization of stacking faults using S/TEM is shown, indicating that the stacking faults lie preferentially on the {100} planes in the material. Additionally, results indicate that the presence of stacking faults is tied to the A/B ratio in the material (i.e. the ratio between Ba and Y + Zr), as no stacking faults are observed when the material is synthesized to be A site deficient (A/B ratio = 0.995). Finally, electrochemical impedance spectroscopy is utilized to analyze the impact of the defects on the electrochemical performance of the material.

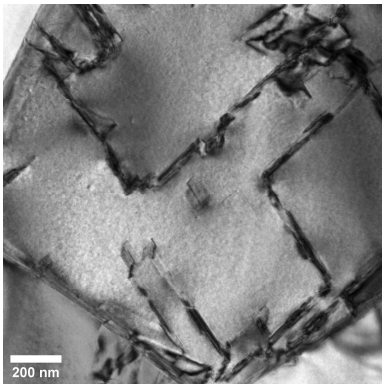

Fig. 1 BF TEM image showing stacking faults in a conventionally sintered BZY20 sample.

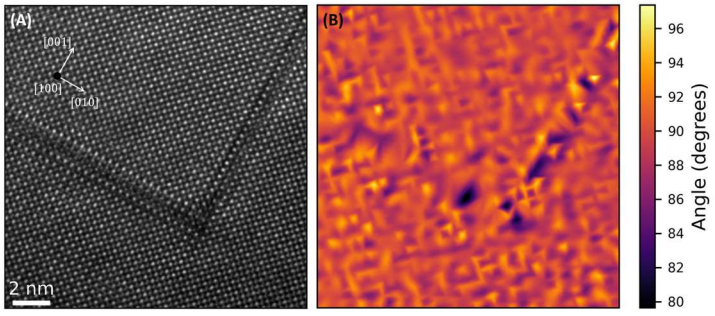

Fig. 2. (A) HRTEM image of stacking fault in BZY20, and (B) a map of angles between the (010) and (001) planes in the image.

FOCUSED ION BEAM MILLING TECHNIQUES FOR TEM SPECIMEN PREPARATION OF SOLID BULK SAMPLES

Lidia Kibkalo[1]*, Maximilian Kruth[1], Lea Risters[1]

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Specimen preparation for the transmission electron microscopy analysis is on the one hand a necessary step, on the other hand a very challenging task. All of the existing methods for the TEM-specimen preparation follow one common principle – thinning and polishing of the bulk sample until the desired thickness. Furthermore, a high quality ultra-thin TEM-specimen should not only preserve the material properties on the nm scale, but also have a homogeneous and undamaged surface to avoid the measurement artefacts. In contrast to time-consuming conventional TEM-specimen preparation methods, the focused ion beam (FIB) sputtering in a dual beam SEM system allows to produce high-quality specimens for various TEM studies (semi-) automatically and quickly. Different TEM based techniques require specific considerations during the specimen preparation. In this work, we discuss various methods of specimen preparation of bulk samples using focused Ga ion beams in FEI Helios dual-beam scanning electron microscopes.

INTERFACE CHARACTERIZATION OF COLD SINTERED $BaZr_{0.7}Ce_{0.2}Y_{0.1}O_{3-\delta}$ PEROVSKITE MEMBRANES

Moritz Kindelmann[1,2,3*], Ashok Vayyala[1,2], Laura Almar[4], Sonia Escolastico[4], Christian Rodenbücher[5], Wendelin Deibert[3], Wilhelm Albert Meulenberg[3], Martin Bram[3], Jose Serra[4], Joachim Mayer[1,2], O. Guillon[3,6]

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[6]Jülich Aachen Research Alliance, JARA-Energy, 52425, Jülich, Germany

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Proton conducting perovskite (ABO_3) ceramics based on yttrium doped barium cerium zirconate ($BaZr_{1-x}Ce_xY_{0.1}O_{3-\delta}$, BZCY) have a great potential for intermediate temperature energy conversion applications like proton conduction fuel cells and electrolyzers (PCFC/PCEC) as well as membrane reactors. Especially the zirconium rich composition $BaZr_{0.7}Ce_{0.2}Y_{0.1}O_{3-\delta}$ (BZCY72) combines high chemical stability against CO_2 atmosphere and a good total conductivity. However, the electrochemical performance of proton conducting perovskites like BZCY is highly dependent on their microstructure and the associated interfacial properties. The novel low temperature processing route we developed in this study for BZCY72 using the cold sintering process (CSP) yields nanoscaled microstructures, which strongly differ from conventionally processed BZCY72. Nevertheless, electrochemical properties match the performance of conventionally processed BZCY72 membranes sintered at significantly higher temperatures (1600°C, 5h). Therefore, the structure and chemistry at the grain boundaries of cold and conventionally processed BZCY72 were characterized by aberration corrected transmission electron microscopy and atom probe tomography and correlated to the electrochemical properties. Our work introduces a new pathway for processing of barium zirconates at intermediate temperatures between 1200 and 1300°C, while maintaining electrochemical performance, chemical robustness and relative densities of state-of-the-art sintered membranes.

STRUCTURAL CHARACTERIZATION OF 2D MATERIALS AND NANOSTRUCTURES

Oliver Maßmeyer[1*], Shubhada Patil[1], Johannes Glowatzki[1], Robin Günkel[1], Andreas Beyer[1], Kerstin Volz[1]

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Two-dimensional materials received a lot of interest over the past decade. Especially Van der Waals 2D materials, such as transition metal dichalcogenides (TMDCs), and their heterostructures, exhibit semiconducting properties, opening the path for novel applications. For these device structures, bottom-up synthesis methods, like metal-organic chemical vapor deposition (MOCVD) are currently explored to realize uniform semiconducting 2D monolayers and heterostructures [1-2]. To reveal the orientation relation between the deposited 2D layers inside the heterostructures and with respect to the substrate we investigated different nano-structures grown by MOCVD, such as GaS on sapphire or WS_2 deposited on monolayer graphene on sapphire. These 2D nanostructures were either be transferred to a TEM-Grid by an etchant-free transfer method, which employs poly(methyl-methacrylate) (PMMA)[3] or by preparation of electron transparent focused ion beam (FIB) lamellae using a JEOL JB 4601. The samples are then characterized by precession electron diffraction (PED) in a JEOL 3010 TEM and in an aberration-corrected JEOL 2200 FS STEM in conjunction with a fast pixelated pn-CCD detector, opening the possibility to analyze the samples by means of 4D-STEM [4]. A phase map and the diffraction patterns of a cross section of a GaS nanostructure grown on sapphire (red), consisting of a grain (blue) and GaS tail (green) is depicted in fig 1. The diffraction patterns indicate an in-plane misorientation between the upper and lower GaS layers, as well as an epitaxial relation of the lower GaS and the sapphire. This and further results obtained by 4D-STEM will be discussed in the presentation.

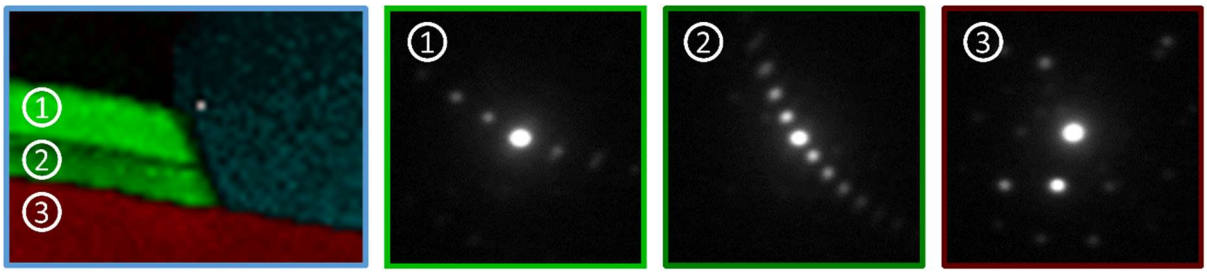

Fig. 1. PED Phase map of a GaS nanostructure and diffraction patterns from different parts of the GaS nanostructure as indicated.

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INVESTIGATION OF BULK ZNPD CATALYSTS USING HIGH RESOLUTION ELECTRON MICROSCOPY

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Methanol Steam Reforming (MSR) is an important reaction converting methanol into hydrogen ($CH_3OH + H_2O \rightarrow 3\ H_2 + CO_2$) holding promise for easy fuel handling in hydrogen based applications like fuel cells. ZnPd/ZnO nanoparticles prove to be promising MSR catalysts due to their high CO2 selectivity, stability and activity.[1] Their activity and selectivity depends very sensitively on their Zn/Pd ratio, even though the relevant intermetallic PdZn phase has a wide compositonal existance range.[2] In order to elucidate the relationship between composition, microstructure, and catalytic performance, ZnPd samples with different composition were characterized using high resolution transmission electron microscopy (HRTEM) and energy dispersive X-ray spectroscopy (EDX). Furthermore, first identical location and in-situ experiments under MSR conditions are conducted to elucidate the microstructural changes of the ZnPd system under realistic operation conditions.

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THE EFFECT OF EPITAXIAL GROWTH DIRECTION ON THE PROPERTIES OF HIGHLY DOPED P-ALGAAS/N-GAINP INTERFACES

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III-V compound semiconductors are used in various optoelectronic devices including LEDs and solar cells. Understanding the nature and quality of hetero-interfaces is still an active field of research. Here we investigated the transition of p-AlGaAs to n-GaInP which includes an exchange of group III and group V atoms as well as a change of the doping source. All layers were grown by metal organic vapor phase epitaxy in an Aixtron 2600 G4 reactor. It has been found that the IV-characteristics of this interface are sensitive to the growth direction. For instance, growing the junction upright or inverted significantly affects tunneling characteristics. The origin of this difference is unknown. Hence, a detailed analysis of the structure, dopant distribution and electric fields at the interface is required to improve the understanding and, thus, the device production. Here, we present a thorough study of the interfaces of inverted and upright grown n-GaInP/p-AlGaAs junctions by means of conventional transmission electron microscopy (TEM) imaging, electron diffraction and aberration-corrected scanning TEM (STEM) combined with energy dispersive X-ray spectroscopy and electron energy-loss spectroscopy. The electron transparent specimens with varying geometries were prepared using focused Ga ion beam (FIB) sputtering in a dual-beam scanning electron microscope. The surface damage of the FIB specimens was reduced using a low-energy (<1 keV) Ar ion beam sputtering. The structural and chemical analyses are combined with a preliminary measurement of the electrostatic potential distribution using off-axis electron holography.

CORRELATING 3D GRAIN ORIENTATION MAPPING WITH ATOM PROBE TOMOGRAPHY IN NANOCRYSTALLINE ALLOYS

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The stability of nanocrystalline (NC) alloys is strongly tied to grain boundary (GB) segregation phenomena [1]. To reveal GB segregation with the highest possible spatial and chemical resolution in nanomaterials, the crystallographic information obtained from transmission electron microscopy (TEM) needs to be correlated with the chemical information obtained from atom probe tomography (APT) on the same specimen [2]. In this correlative method development, 4D-scanning precession electron diffraction (4D-SPED) tomography of a needle shaped specimen is correlated with APT to obtain a grain orientation mapping with high spatial resolution to identify all degrees of freedom of the GBs and solute distribution in three-dimensions. Here, (Fig. 1), a ~1 nm-sized incident beam is precessed by 1° while being scanned over a needle shape Ni-W NC-alloy to realize quasi-kinematical scattering conditions. Nanobeam scanning precession electron diffraction (SPED) patterns are acquired for a ±80° tilt range with 10º tilt increments. The diffraction patterns are recorded using a scintillator coupled CMOS camera (TVIPS TemCam-XF416) for capturing reflections with high signal-to-noise ratio, dynamic range and read-out speed. A multi-indexing approach, coupled with automated crystal orientation mapping (ACOM), is being used to extract information (Fig. 2) from overlapping grains [3]. Further, we will discuss implications on our 3D understanding of segregation effects in nanocrystalline alloys.

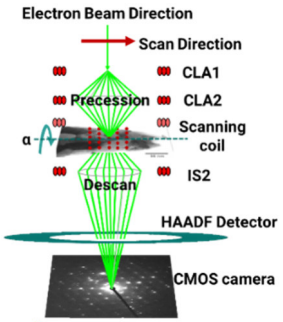

Fig. 1. 4D-SPED data acquisition schematic.

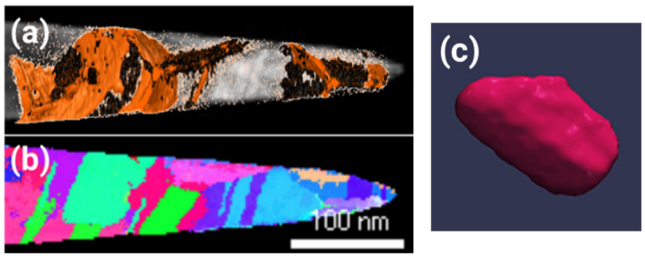

Fig. 2. (a) Projected 3D atom map over VDF image of APT tip. (b) grain orientation by 4D-SPED analysis. (c) reconstructed grain.

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UNDERSTANDING GAS ADSORPTION OF PAN-BASED CARBON NANOFIBERS

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Polyacrylonitrile-based carbon nanofibers (PAN-based CNFs) have great potential to be used for carbon dioxide (CO_2) capture due to their excellent CO_2 adsorption properties. The porous structure of PAN-based CNFs originates from their turbostratic structure, which is composed of numerous disordered stacks of graphitic layers. During the carbonization process, the internal structure is arranged toward the ordered graphitic structure, which significantly influences the gas adsorption properties of PAN-based CNFs. However, the relation between structural transformation and CO_2 capture is still not clear enough to tune the PAN-based CNFs. In this paper, we show that, with increasing carbonization temperature, the arrangement of the PAN-based CNF's structure along the stack and lateral directions takes place independently: gradually aligning and merging along the stack direction and enlarging along the lateral direction. Further, we correlate the structural arrangement and the CO_2 adsorption properties of the PAN-based CNFs to propose a comprehensive structural mechanism. This mechanism provides the knowledge to understand and tailor the gas adsorption properties of PAN-based CNFs.

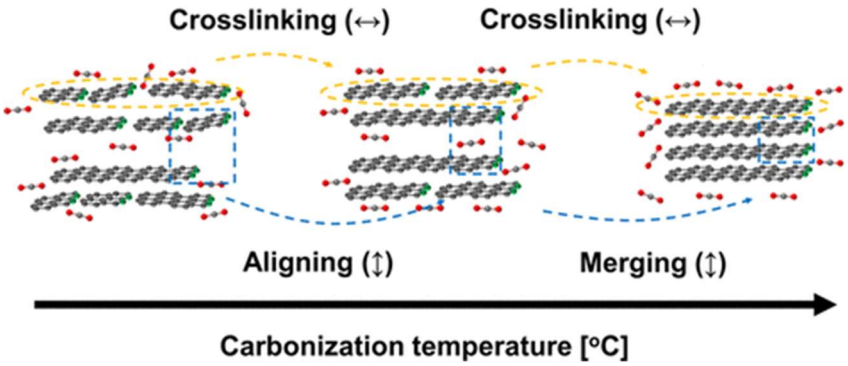

Fig. 1: Shows the schematic representation of structural evolution of PAN-based CNF during sintering.

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INTERMETALLICS IDENTIFICATION IN LEAN MG ALLOYS BY COMBINING EDS, DIFFRACTION AND HAADF STEM

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Transmission electron microscopy is an amazing technique to solve materials science puzzles. In the present work we use it at its limits to tackle the crystallographic structure of binary and ternary intermetallic precipitates made of calcium, magnesium and zinc in a lean Mg alloys envisaged as a material for implants, whose biodegradation depends on precipitation [1]. While the equilibrium binary Mg–Ca compound is known to have a C14 hexagonal structure, its precursor phases are still unclear. It appears that the precipitation sequence is as follows: Guinier-Preston zone $\Longrightarrow$ hexagonal phase $\Longrightarrow$ C14 phase, as identified by diffraction and EDS. Moreover, while the ternary Ca–Mg–Zn phase structure has been postulated in 2003 to be the trigonal phase $Ca_2Mg_6Zn_3$, and many other structures have been proposed since then, it remains unclear. In 2016 we proposed a new phase, a hexagonal $Ca_2Mg_5Zn_5$, on the base of X-rays and electron diffraction [2], whose composition incidentally coincides with the one proposed in 1933 by Paris [3]. However, in lean Mg alloys doubts remain on the ternary precipitates structure, in particular with respect to the known isostructural phase $Ca_3Mg_{11}Zn_4$ [4]. Here we combine diffraction, atomically-resolved EDS, and Cs-corrected HAADF STEM imaging to conclude firmly on its identity. We applied image simulations to interpret the obtained HAADF STEM images, using xSTEM [5], Dr. Probe [6] and jEMS [7]. The comparison between software indicates that the inclusion of the frozen lattice model in the simulation scheme is essential for a good match to the experimental image for thick areas. This may relate to the way the electrons are absorbed by channelling on the crystal atomic columns. The approach allowed confirming the $Ca_2Mg_5Zn_5$ underlying crystallography, while the composition, deduced by EDS, leaning towards the one of $Ca_3Mg_{11}Zn_4$ is achieved by adjusting the lattice occupancy ratios in the $Ca_2Mg_5Zn_5$ structure.

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STRUCTURE AND DEFECT ANALYSIS OF SUPERCONDUCTING NbN THIN-LAYERS

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Transition-metal nitrides (TMN) represent a class of materials relevant for their superconductivity and excellent mechanical properties [1]. Interestingly, rock salt δ-NbN possesses the highest reported superconducting transition temperature (T_C~17.3K) among TMN [2], along with remarkable mechanical and elastic properties. While superconductivity is well established in high quality NbN layers [1-3], a systematic analysis of the structure and of relevant defects in thin-film NbN is still in its infancy.

Here, the epitaxial growth by reactive magnetron-sputtering (MS) and atomic layer deposition (ALD) of NbN onto *c*-sapphire, and its comprehensive characterization *via* optical microscopy (OM), atomic force microscopy (AFM), x-ray diffraction (XRD) and transmission electron microscopy (TEM) are reported. The collected XRD data suggest that the deposited NbN films are highly crystalline and stabilize in a (111)-oriented δ-NbN phase. The analysis of the structure and the evolution of defects have been carried out with TEM. The diffraction patterns of the NbN films confirm a cubic structure and a $(111)\ [1\bar{1}0]$// $(0001)\ [10\bar{1}0]$ epitaxial relation [4]. Fast-Fourier Transformations (FFT) and inverse FFTs allow establishing the *d*-spacing and hence the lattice parameters, which are found to be consistent with the XRD data. Moreover, *via* a combination of bright-field and dark-field imaging, the various dislocation types, as well as their propagation are visualized.

These findings pave the way to the optimization of high quality δ-NbN thin-films with mechanical and superconducting properties on demand. Moreover, wide perspectives are envisaged for the fabrication of heterostructures based on NbN in combination with ferromagnets for fostering the onset of long-range superconducting proximity effects, such as the generation of spin-polarized currents [3].

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ATOMIC-SCALE TUNING OF CHARGE DISTRIBUTION BY STRAIN ENGINEERING IN COMPLEX OXIDE HETEROSTRUCTURES

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Due to the challenge of disentangling intrinsic and extrinsic effects at complex oxide interfaces, the combined effects of epitaxial strain and charge transfer mechanisms have been rarely studied. Here, we reveal the local charge distribution in manganite slabs by means of high-resolution scanning transmission electron microscopy (STEM) and spectroscopy via investigating how the strain locally alters the electronic and magnetic properties of La_2CuO_4–$La_{0.5}Sr_{0.5}MnO_3$ (LCO–LSMO) heterostructures (Fig 1.). The charge rearrangement results in two different magnetic phases, an interfacial ferromagnetically reduced layer and an enhanced ferromagnetic metallic region away from the interfaces. In addition, the magnitude of the charge redistribution can be controlled via epitaxial strain, which further influences the macroscopic physical properties in a way opposed to strain effects reported on single-phase films. [1]

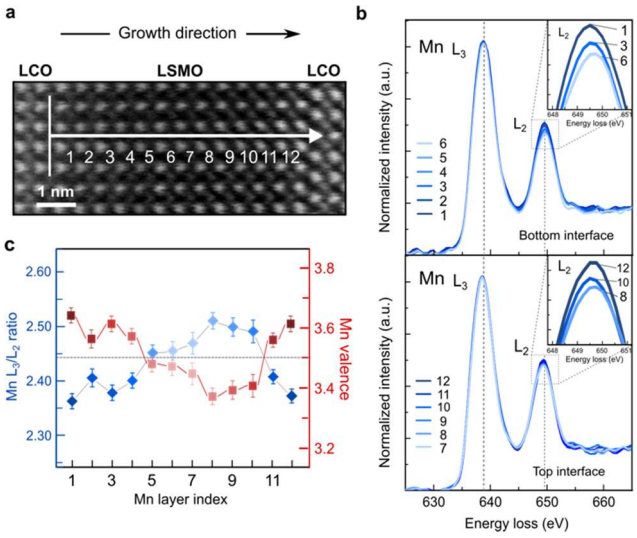

Fig. 1. (a) STEM-HAADF image of LCO/LSMO/LCO trilayer grown on LSAT substrate, where the white arrow indicates the region that EELS spectra were acquired. (b) Layer-resolved Mn $L_{2,3}$ edge spectra collected from the bottom interface to the top interface, layers 1-12 in (a). (c) Local variation of the Mn L_3/L_2 intensity ratio (blue) and the corresponding Mn valence (red) within the LSMO. [1]

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NANOSTRUCTURE OF METAL POLY(HEPTAZINE IMIDES)

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Poly(heptazine imides) have become a benchmark carbon nitride materials in photocatalysis [1, 2]. Direct unambiguous structural solutions of these compounds using X-ray powder diffraction is in many cases not possible due to a high concentration of defects/disorder in these systems. We now combined X-ray powder diffraction (XRD) with low-dose HRTEM imaging and energy filtered electron radial distribution function (EF-eRDF) analysis for solving defect crystal structures of several metal poly(heptazine imides). Our results show how specific structural features influence catalytic performance, in particular related to hydrogen evolution reaction.
We solved and refined structures of poly(heptazine imide) salts of sodium (Na-PHI), potassium (K-PHI), magnesium (Mg-PHI), iron (Fe-PHI) as well as poly(heptazine imide) acid (H-PHI), obtained by ion exchange reaction from salts. Based on low-dose HRTEM and XRD data, and general assumptions about the structure from earlier works [1], starting models for refinement were proposed. PHI salts were found to crystalize in a trigonal unit cell; their idealized structure consists of heptazine units that are placed on top of each other in a so called AAA stacking, forming continuous channels along the *c* direction. In most salts, metal atoms tend to preferably occupy positions between the center of the channels and bridging nitrogen atoms of the heptazine units. During refinement an additional type of anisotropic broadening function had to be introduced, suggesting the presence of disorder. We found that the type of defects present in the samples is strongly dependent on the metal cation in the salt and the synthesis procedure. Thus, low-dose HRTEM images of K-PHI lamellar nanocrystallites display unit cell distortions, faults in the sequences of CN-layer stacking, edge and screw dislocations as well as rippling of CN-layers. Na-PHI, Mg-PHI and Fe-PHI polycrystalline flakes consist of many rotational stacking domains. The rotation of layers within a domain by a random (usually only 2-8°) angle leads to the formation of “flower-like structures”, while at specific angles of rotation Moiré pattern lattices are formed. Disorder in the distribution of metal atoms is common to all salts. The EF-eRDFs of the salts revealed specific details about their local structure and the mean size of the domains based on coherent scattering length.
We gratefully acknowledge financial support by the Max Planck Society.

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LOW-DOSE PERFORMANCE OF IDPC-STEM

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iDPC-STEM is utilized as the integration (inverse gradient) of the DPC *vector* field to resolve the iDPC *scalar* field, Lazić, et al. [1]. For a thin sample, the DPC vector represents the projected electric field, which, when integrated into iDPC, yields the scalar, projected electrostatic potential field of the sample [1, 2]. Besides imaging all elements of the periodic table, iDPC-STEM is also a low-dose imaging technique with a proven performance record on a variety of beam sensitive materials [3, 4, 5, 6]. Here we will further demonstrate this ability by adding iDPC-STEM into the list of existing and newly introduced STEM techniques, specifically optimized for low-dose [7]. Figure 1 shows iDPC-STEM simulations at different total electron doses to match the simulations performed by Ooe et al. [7].

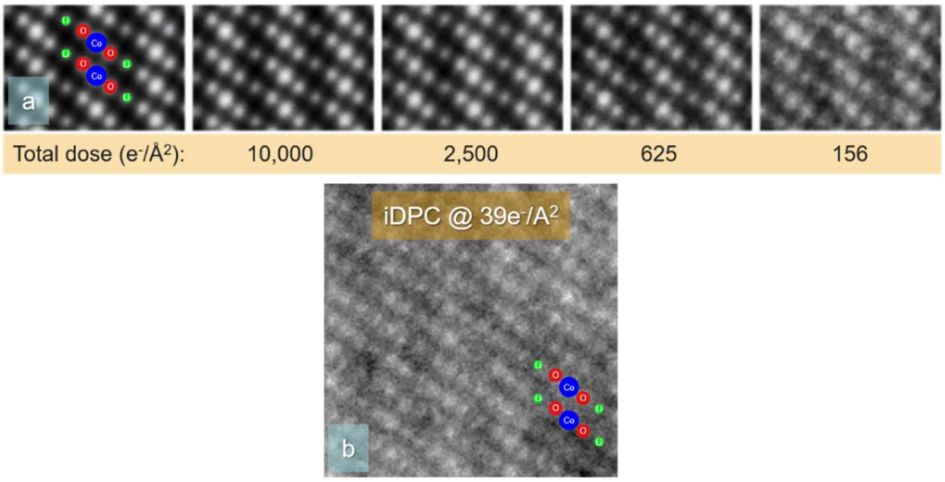

Fig. 1 iDPC-STEM simulation of $LiCoO_2$ [010] at 120kV. **(a)** The simulations from left to right are done for the following total doses: infinite, 10000 $e^-/Å^2$, 2500 $e^-/Å^2$, 625 $e^-/Å^2$, and 156 $e^-/Å^2$ to match the OBF simulations in [7]. **(b)** The total electron dose was further reduced to 39 $e^-/Å^2$, 4 times less than the lowest dose shown in [7].

Various experimental iDPC-STEM results will be shown including extremely beam sensitive MOF's [3] and Zeolites [4].

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UNVEILING ATOMIC-SCALE CHARGE DISTRIBUTION AT COMPLEX OXIDE INTERFACES

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Heterointerfaces in complex oxide materials promote emergent physical properties like colossal magnetoresistance, magneto-electric coupling, fractional quantum Hall Effect, and high-T_c superconductivity, which are not found in their bulk constituents. These properties can be attributed to the reconstruction of charge, spin, and orbital ordering at interfaces. Of particular concern is the charge distribution at interfaces, which is a crucial factor in controlling the interface transport behavior. However, the study of the charge distribution is very challenging due to its small length scale and the intricate structure and chemistry at interfaces. Furthermore, the underlying origin of the interfacial charge distribution has been rarely studied in-depth and is still poorly understood. In this study [1], we performed atomic-scale investigations of the microstructure and electronic structure at $SrTiO_3$ (STO) - $SrMnO_3$ (SMO) heterointerfaces by high-angle annular dark-field and annular bright-field imaging and by electron energy-loss spectroscopy (EELS) using an aberration-corrected scanning transmission electron microscope (STEM). EELS analysis confirms that the charge density attains the maximum of 0.13 ± 0.07 e^-/unit cell (uc) at the first SMO monolayer (Fig. 1). Based on quantitative atomic-scale STEM analysis and first-principle calculations, we explore the origin of the interfacial charge accumulation in terms of epitaxial strain-favored oxygen vacancies, cationic interdiffusion, interfacial charge transfer, and space-charge effects. This work provides a comprehensive description of the charge distribution and related mechanisms at the SMO/STO heterointerfaces, which is beneficial for the functionality manipulation via charge engineering at interfaces.

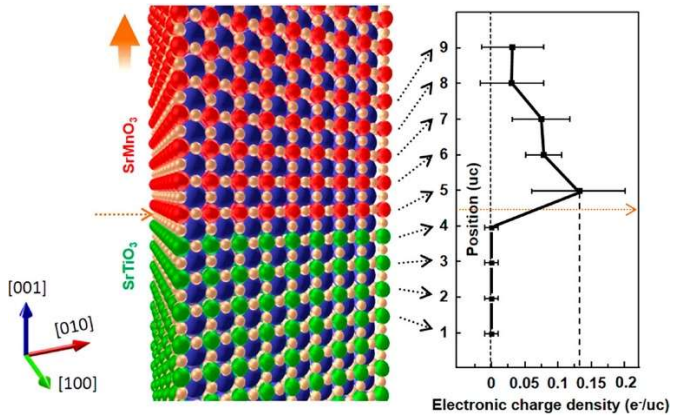

Fig. 1 Electronic charge density as a function of atomic position near the interface

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CONTINUOUS ILLUMINATION PICOSECOND IMAGING OF MAGNETISATION DYNAMICS IN A TRANSMISSION ELECTRON MICROSCOPE

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Studies of magnetic nanostructures using transmission electron microscopy (TEM) often require high spatial and temporal resolution. In fact, while achieving sub-nanometre spatial resolution in modern TEMs is straightforward, the temporal resolution is limited to the microsecond range in general. Here, we present the possibility to access reversible dynamics in the picosecond range applying a fast delay line detector (DLD) at an aberration-corrected transmission electron microscope [1].
DLDs measure the arrival time of every electron which was assigned to a specific phase based on the synchronisation with the microwave magnetic excitation that was applied using a custom-made holder. The limits of the setup were probed with low angle diffraction experiments where the electron beam was deflected by microwave magnetic fields. There, an intrinsic temporal resolution of about 120 ps was achieved in a frequency range from 0.1 to 2.3 GHz. As a proof of concept, the magnetic vortex core gyration excited by microwave magnetic fields in a permalloy disk with 1.6 µm in diameter and 130 nm in thickness. The deflection of the electron beam due to the Lorentz force was used to visualise the position of the vortex core employing Lorentz TEM. The resonance frequency was found to be around 420 MHz and the radius of the gyration was in the range of a few tens of nanometers for the applied microwave excitation. Both results are in good agreement with supporting micromagnetic simulations. Hence, combining TEM with fast detectors offers the possibility to study dynamics in real space with an improved temporal resolution by six order of magnitude compared to standard TEM cameras. This work is supported by the DFG through CRC/TRR 270 (Project ID 405553726), the European Union's Horizon 2020 Research and Innovation Programme (Grant No. 856538 and 823717) and ETH Zurich.

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MAPPING OXYGEN COLUMNS IN MAGNETITE CRYSTALS USING DIFFERENTIAL PHASE CONTRAST STEM IMAGING

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Differential phase contrast (DPC) imaging in the scanning transmission electron microscope (STEM) is an emerging technique to observe light elements with low atomic number and to determine the local charge density of crystalline materials at atomic resolution [1, 2]. DPC images reconstructed from four-dimensional (4D) STEM data have extended the application to reveal information about electrical fields and with this indirectly information on local atomic bonding at sub-Ångstrom resolution [3]. In this talk, we will present our calibration of the electron microscope pixel array detector (EMPAD) using polycrystalline Au samples and compare our DPC measurements on $SrTiO_3$ (STO) single-crystalline samples to results published in literature [2, 3]. Furthermore, we have applied the DPC technique to map oxygen columns and charge density maps in magnetite (Fe_3O_4) crystals and at the interface between magnetite and iron. The talk will also discuss data processing methods to increase the signal-to-noise ratio and steps to correct scanning artifacts. Direct experimental observations of charge transfer between neighboring atoms at magnetite-iron interfaces will provide novel insights into our fundamental understanding of iron corrosion and the contribution of the interface structure in catalytic processes.

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PERFORMANCE ESTIMATES FOR A GROUND-POTENTIAL MONOCHROMATOR

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Electron energy loss spectroscopy with high energy resolution is interesting for a wide range of applications ranging from analytics of organic matter at very small dimensions to determining the optical properties in two-dimensional materials. In (scanning) transmission electron microscopy ((S)TEM), the energy resolving power of the instrument was determined by the type of the electron source and limited to about 300 meV before the advent of electron monochromators for (S)TEM instruments about two decades ago. In the mean time, several different designs of monochromators have been implemented and can be bought as an option to most high-end microscopes [1-4]. There are two possible positions for monochromators along the optical path: Before or after the accelerator, that is, at high potential or at ground potential, respectively. While most monochromator implementations today are at high potential, ground-potential monochromators have some notable advantages, such as less sensitivity to selection-slit interactions and magnetic stray fields, independence of acceleration voltage instabilities, no Boersch effect within the monochromator, and the possibility to retrofit it to existing instruments. One of the major general drawbacks in the use of a monochromator for imaging or spectroscopy is the loss of current in the electron beam, and thus, loss of signal. Therefore, the most interesting parameter besides the actual energy resolution is the available current at this energy resolution for a certain primary electron energy. Here, we evaluate the possible performance of a ground-potential monochromator. To this end, we summarise the physical background for the determination of the performance parameters that we present. All calculations are independent from the geometry and design of the actual monochromator. As examples, numbers are given for an instrument equipped with a cold field emission electron source operated at primary electron energies of 300 keV and 30 keV. This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement ID 823717 "ESTEEM3" and grant agreement ID 951215 "MORE-TEM".

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CEFID: A FLEXIBLE PLATFORM FOR SPECTROSCOPIC EXPERIMENTS

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TEM-related instrumentation has been developing rapidly in the last few years and a state-of-the-art experiment often involves a variety of third-party components ranging from advanced in-situ specimen holders and new generation detectors to pulsed lasers and programmable phase plates. The recently developed CEOS Energy Filtering and Imaging Device (CEFID) is an energy filter and spectrometer offering the flexibility to implement ambitious and unprecedented experiments. The filter design comprises highly optimised and stable optics up to the energy-selecting slit, and a flexible and minimalist projective stage [1]. This gives high performance and stability, while allowing to hop between modes (imaging, spectroscopic dispersions) with little to no re-tuning. The Python/Qt-based graphical software used for the filter operation implements interactive and automated procedures for alignments, common workflows ranging from EFTEM to 4D-STEM, and tools for on-the-fly analysis from live DFT to EELS maps computation. A wide range of detectors (Fig. 1) and scan generators from different manufacturers has already been integrated and can be used for both the tuning and data acquisition. The software is highly extendable and offers a scripting and plug-in API in python and a remote control interface for the integration into third-party software. The flexibility and compatibility make the filter a platform for complex experiments where different tools need to work in unison, such as the synchronization of acquisition with in-situ stimuli, or photon-induced near-field electron microscopy [2].

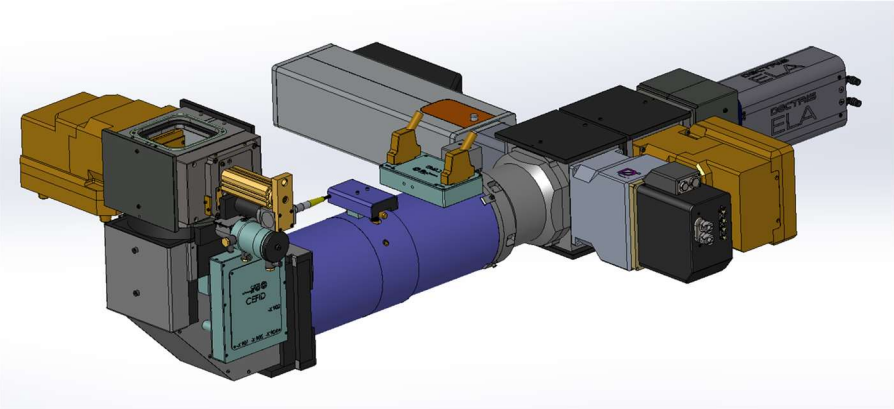

Fig. 1: Rendering of CEFID outfitted with five cameras (TVIPS XF416R, ASI Cheetah, Direct Electron DE-16 and Dectris ELA) and a retractable ADF detector developed by CEOS. Each detector has its specific strengths which make it ideal for a class of experiments.

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HIGH COHERENCE ELECTRON SOURCES FOR ULTRAFAST TRANSMISSION ELECTRON MICROSCOPY BASED ON PHOTOASSISTED FIELD EMISSION

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Ultrafast transmission electron microscopy combines femtosecond time resolution with a nanometer spatial probe. Laser-triggered Schottky field emitters feature one-photon photoemission with a high beam brightness [1,2]. A promising candidate for a further enhancement of the beam coherence is photoassisted field emission from tungsten tips [3-5], offering the high coherence and the narrow energy spread of cold field emitters [6], while maintaining the flexibility of linear photoemission. In this study, we laser-trigger one-photon photoemission from W(310)-nanotips (tip diameter ~120 nm). Using Fowler-Nordheim plots [7], we can attribute the main electron emission process to photoassisted field emission [3-5], i.e. a transient photoexcitation of electrons to a higher energy level resulting in an increased tunneling probability. With a frequency-tunable laser, we experimentally verify a linear relation of the laser photon energy and the reduced effective work function [3,8] of W(310). We expect improved ultrafast imaging, spectroscopy and holography conditions from the combination of flexible time-structuring from one-photon photoemission with the high coherence from photoassisted field emitters. Future work will be focused on improving the emission lifetime of laser-driven photoassisted field emitters, thus making them readily accessible in ultrafast transmission electron microscopy.

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COMBINATION OF ABERRATION CORRECTION WITH A THIN-FILM ZERNIKE PHASE PLATE

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Aberration-corrected transmission electron microscopy allows imaging at highest resolution. However, in a corrected microscope under focused conditions, phase-contrast transfer is minimum. This leads to weak contrast of many nanomaterial specimens, which mainly modify the phase of the transmitted electron wave. Physical phase plates (PPs) allow the enhancement of phase contrast, but have been applied mainly to biological specimens [1]. Here we present the application of a thin-film Zernike PP, implemented in an aberration-corrected microscope, to nanomaterial samples [2]. Fig. 1 shows the comparison of two focused images acquired from the identical specimen region (a) with and (d) without PP. Although a minor defocus difference might affect the contrast of the lattice fringes of the $Fe_{3-\delta}O_4$ nanoparticles [3], the main effect on contrast may be attributed to the PP action, especially at intermediate spatial frequencies. We performed a detailed study of the behavior of the PP and calculated phase-contrast transfer functions considering convergent illumination. The results prove that the application of PPs in aberration-corrected TEM is highly promising. [4]

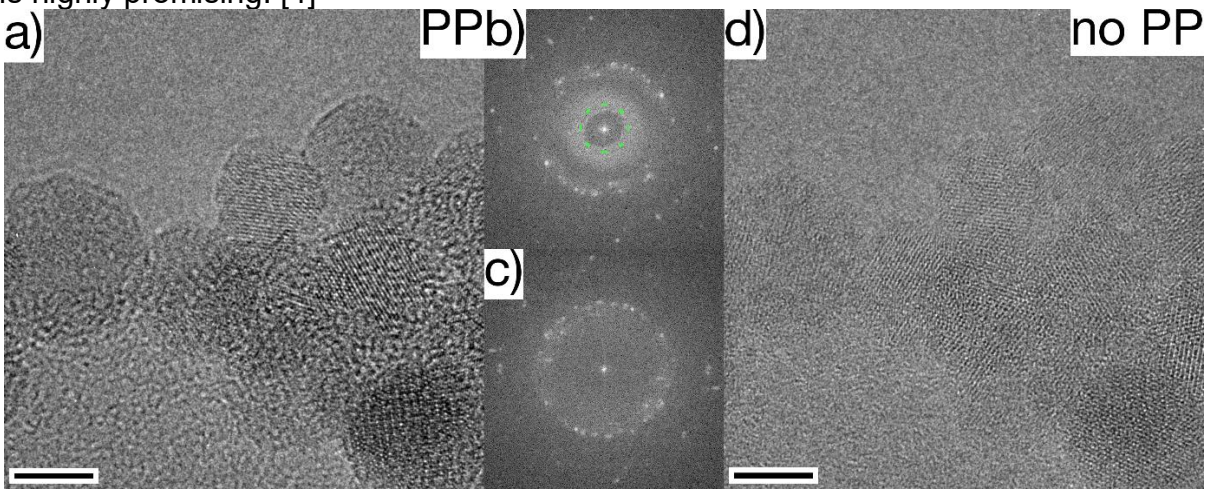

Fig. 1: (a,d) HRTEM images of $Fe_{3-\delta}O_4$ NPs and (b,c) corresponding power spectra (size of 14.3 nm^{-1} x 14.3 nm^{-1}) acquired (a,b) with and (c,d) without Zernike PP (hole radius of 5 µm). Contrast settings for (a,d) are identical. Scale bar: (a,d) 5 nm.

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THE NEXT GENERATION OF *IN-SITU* OPEN CELL ENVIRONMENTAL DOUBLE ABERRATION CORRECTED E-(S)TEM: ARTEMIS

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Atomic resolution electron microscopy is a powerful and increasingly important tool for nanomaterials characterisation that is vital to solutions of key problems in energy, IT, health, safety, transport, the environment and economy. York is in the process of installation of an *in-situ* open cell environmental TEM/STEM facility based on a JEOL NeoArm cold FEG double corrected instrument (ARTEMIS). Recent progress in vacuum modifications and open cell gas injection systems will provide a much-improved version of the current proof-of-principle ETEM/STEM based on the first generation of aberration corrected microscope JEOL 2200FS. Here we will discuss the wide range of capabilities associated with the machine based on variable voltage, gas/vapour injection system, diffraction and EDX/EELS spectroscopy, and the new science enabled by this facility. In addition, we will present *in-situ* results, that includes oxidation/reduction and H_2O vapour on thin films and nanoparticles in low pressure open cell environment, that were performed on the first generation environmental TEM/STEM where single atom visualisation by HAADF-STEM in an open cell controlled gas reaction environment and temperature was demonstrated.

Acknowledgement:
We thank the EPSRC (UK) for funding, through capital infrastructure grant, ‘Aberration-Corrected Scanning Transmission Electron Microscope with atomic resolution spectroscopy under controlled environmental conditions: AC-eSTEM’; EP/S033394/1.

THE USER ADJUSTABLE POLE-PIECE – A TEM HEART TRANSPLANT

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The transmission electron microscope (TEM) is a powerful characterisation instrument for both physical and life sciences. However, it represents a significant investment of often several million dollars followed by the running costs, cooling/energy bills, support staff salaries, and maintenance contracts for perhaps a further 15 years [1]. Many consider the objective lens pole-piece to be the heart of the TEM, with the pole-piece gap specification being crucial in dictating the capabilities and performance of the instrument [2]. While a smaller gap results in reduced spherical and chromatic aberrations, and improved resolution [3], a larger gap offers wider flexibility for sample-tilting, tomography, in-situ experiments, or energy-dispersive x-ray spectroscopy (EDX) collection efficiency. As the former is typically preferred by the physical science community, but the latter by the analytical/life-science communities, the result is that two (or more) columns are often needed, or alternatively, a single intermediate gap may be commissioned but compromises must be made. As an alternative approach, we propose a User Adjustable Pole-piece (UAP) with a pole gap that can be adjusted by the microscopist to suit a variety of experiments, reducing wasteful duplication [4]. To be of practical use, the proposed UAP must be; adjustable while under high vacuum, mechanically stable with ultra-precise alignment about the optic axis, and be realignable to a good tuning without a specialist engineer present. In this presentation we will elaborate on our progress in the design and manufacture of the UAP. We will present our preliminary results from multi-physics modelling simulations which confirm that the larger pole gaps yield better tilt/access for experiments, while the smaller gaps yield reduced aberrations. Furthermore, these simulations allowed us to evaluate geometric predictions for EDX collection solid-angles [5] and the attainable tilt-ranges of these customised holders. [6].

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ENERGY DISCRIMINATION USING CMOS-COMPATIBLE ARRAYS OF PHOTODIODES IN SCANNING ELECTRON MICROSCOPY

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Pixel array detectors for electron microscopy (or EMPADs) have recently been developed with spectral sensitivity (e.g., *Medipix3* family). In this sense, each photodiode measures the electric charge freed on its substrate by individual electrons. Nonetheless, these EMPADs have poor energy resolution. In this work, we have fabricated an EMPAD sensor for electrons with improved spectral sensitivity and application in the range of energies of scanning electron microscopy (SEM) between 1-30 keV [1, 2]. The proposed EMPAD has a 64x44 Active Pixels Sensors (APS) matrix fabricated in Complementary Metal Oxide Semiconductor (CMOS) technology of 180 nm. The Figure below shows the vertically stacked diodes, pixel, and matrix layout of the proposed EMPAD.

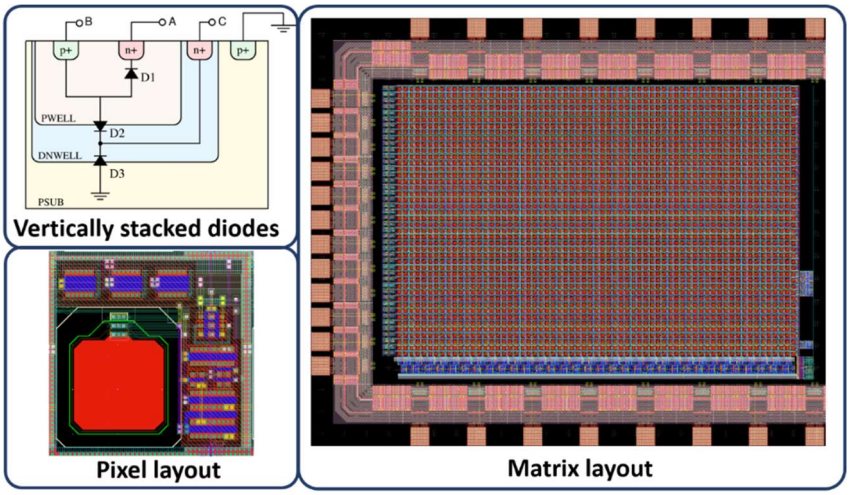

Fig. 1 Vertically stacked diodes, pixel, and matrix layout.

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CAN A PROGRAMMABLE PHASE PLATE SERVE AS AN ADAPTIVE C_s CORRECTOR IN THE TEM?

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Current progress in programmable electrostatic phase plates (PPs) raises several questions about their usefulness for specific applications [1]. Here, we explore different designs for such PPs to correct spherical aberration in the Transmission Electron Microscope (TEM). More specifically, we theoretically investigate whether a PP could provide down to Ångström spatial resolution on a conventional uncorrected TEM. Some of the proposed designs show a probe size (d_{50}) down to ≈ 0.93 Å, proving that (at least theoretically) it is possible to correct spherical aberration past the 1 Å limit using a programmable PP. We discuss the possibility of implementing an adaptive algorithm to tune such a PP as a compact automated alternative to multipole arrays and present an early experimental realization of such a setup, showing its feasibility.

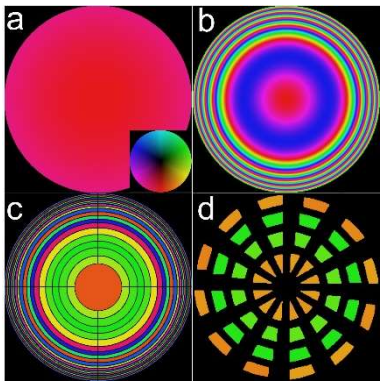

Fig.1. C_s on a round aperture: *1µm for* (a), *1.2mm* for (b). Concentric ring design with 20 segments at *15mrad* (c), Segmented design with 48 segments at *10mrad* (d).

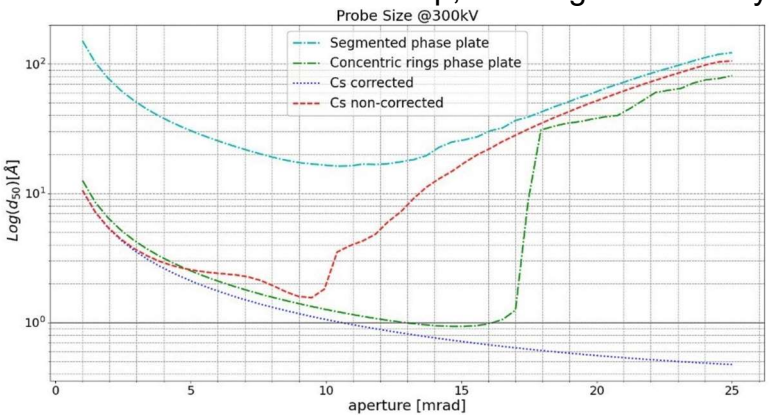

Fig.2. Simulated probe size (log scale) assuming *300keV* and C_s*=1.2mm* for the phase plates and non-corrected aperture, and C_s*=1µm* for a corrected aperture. The black line shows the 1 Å limit. The Concentric Ring design is capable of breaking the 1 Å giving a ≈ 40% improvement in probe size over the non-corrected instrument.

References:

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SINGLE SCAN STEM EMCD IN 3-BEAM ORIENTATION

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Electron magnetic circular dichroism (EMCD)[1] is a transmission electron microscope (TEM) based technique for nanoscale characterization of element specific magnetic moments of the materials. A classical EMCD experiment requires the acquisition of two or four off-axis EELS spectra located at conjugate scattering angles in the reciprocal space, with the sample tilted to two or three beam orientation respectively. In STEM mode, the spatial resolution of the EMCD analysis is defined by the size of the electron probe as well as how good the multiple datasets are spatially aligned to each other. Sample drift can result in a significant degradation of the spatial resolution of analysis compared to the actual probe size [2]. In this work, we built a quadruple aperture to acquire the four off-axis EELS spectra in 3-beam orientation in a single STEM scan. This ensures a perfect spatial registration among the 4 EMCD component spectra. We detected an EMCD signal for bcc Fe and measured the magnetic orbital to spin moment's ratio (m_L/m_S).

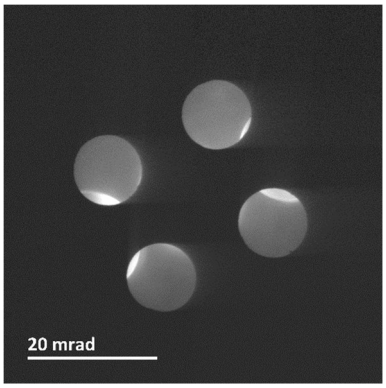

Fig. 1 CCD image of quadruple aperture overlaid on diffraction pattern in 3-beam orientation

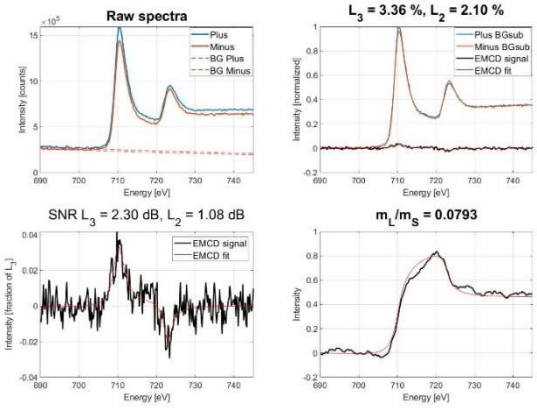

Fig. 2. EMCD signal obtained using the quadruple aperture and the measured mL/mS value

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REDUCING ELECTRON BEAM DAMAGE THROUGH ALTERNATIVE STEM SCANNING STRATEGIES

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Transmission electron microscopes (TEM) provide unique capabilities to characterize matter down to the atomic scale. However, the effect of the highly energetic electrons used as probing particles/waves is far from negligible, especially on soft materials such as zeolite, organic or biological materials. This beam damage, with multiple potential physical origins, remains difficult to study and available results suffer from a general lack of reproducibility. This work attempts to reproducibly study the effect of the electron beam on a commercially available zeolite material while applying different scan patterns [1,2]. An alternative scanning sequence consisting of spatially separating 2 time consecutive scan points by a distance of an integer number of conventional scan steps and repeating and shifting multiple frame scans until all scan positions are visited (Figure 1a). Such acquisition results in an image presenting the exact same dose and dose rate as conventional raster scanning. We demonstrate experimentally that a significant and statistically relevant beam damage reduction is obtained as shown in Figure 1b and 1c. In order to understand the reason of such behavior, a threshold diffusion model was proposed which qualitatively reproduces the experimental observations (Figure 1d and 1e). We discuss the scope of the current findings and their potential applications on other types of electron beam sensitive materials.

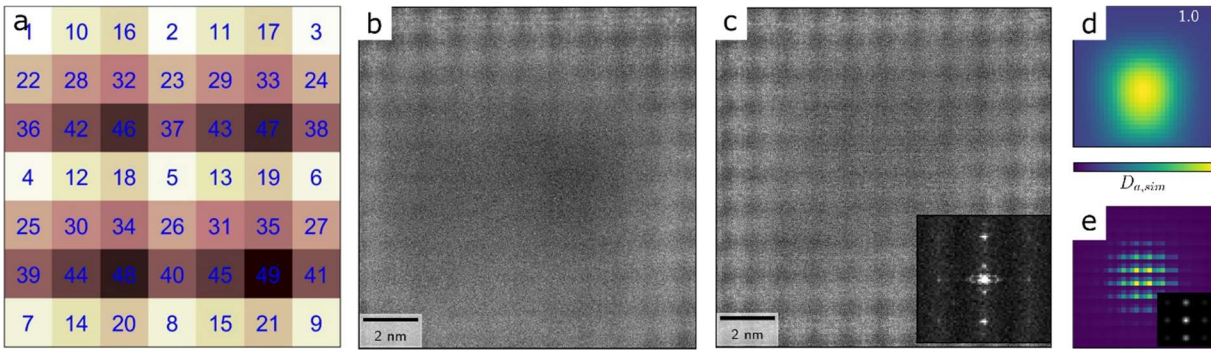

Fig. 1: a) Schematic representation of the alternative scanning sequence. b,c) Experimental visualization of the damaged area after 3 consecutive passes of raster (b) and alternative (c) scan patterns. d,e) Modelled electron beam damage in b and c.

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Acknowledgment: AB, AV, DJ and JV acknowledge financial support from the Flemish region through FWO grants G093417N, G042920N and the European Union's Horizon 2020 research & innovation program under grant agreement No 823717 ESTEEM3.

THE EFFECT OF DYNAMICAL SCATTERING ON PHASE RETRIEVAL IN 4D-STEM

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Imaging the complex transmission function of a sample is key to interpreting the electric and magnetic properties of the specimen, and as such 4D-STEM imaging techniques are crucial for our understanding of functional materials. Many of the algorithms used in the reconstruction of the transmission function rely on the multiplicative approximation and the (weak) phase object approximation, which are not valid for many realistic materials, particularly at high resolution. Herein we study the breakdown of simple phase imaging in thicker samples, using integrated centre of mass imaging, single side band ptychography and Wigner distribution deconvolution over a thickness series of simulated GaN. From these we determine optimal focal conditions for obtaining more interpretable data, and demonstrate the utility of post-acquisition aberration correction in 4D-STEM datasets.

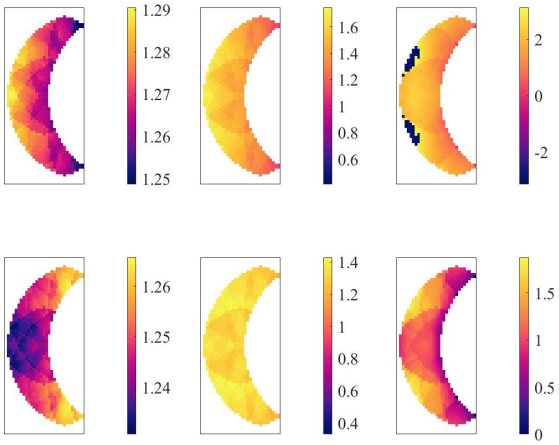

Fig. 1. Illustration of phase interference in double overlap regions for 3A, 31A and 175A thick samples (left to right) in the top surface focus case (upper panel) and the mid-plane focus condition (lower panel). Perceptually uniform colour map following Ref 1.

References:

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† Now at: imec, Kapeldreef 75, 3001 Leuven, Belgium

IMPROVEMENTS IN DATA PROCESSING OF STEM-DPC WITH DISTORTED BRIGHT FIELD DISK

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Pixelated electron detectors for use in scanning transmission electron microscopy (STEM) have both unlocked new ways of studying materials and greatly enhanced existing techniques. One example of the latter is STEM – differential phase contrast (DPC)[1]. When passing through an electromagnetic field in a material, the electron beam is deflected. By tracking the position of the bright field disk for each position in a STEM-scan, we can find the perpendicular magnetic or electric field the beam passed through. Any effect that either distorts or redistributes the intensity in the bright field disk complicates this. For the vacuum probe (Fig. 1a) finding the center position of the bright field disk is straight forward, but for the disk from a thick region (Fig. 1b) it becomes much trickier. These large disturbances in its shape severely complicate the data processing. This is further exacerbated by the immense data sizes: a small dataset consists of 65000 images, necessitating robust, efficient, and scalable algorithms. In this work, we will detail both the challenges and possible solutions for extracting the beam shifts from these heavily distorted bright field disks. Here, we will utilize open-source Python software such as HyperSpy and pyXem.

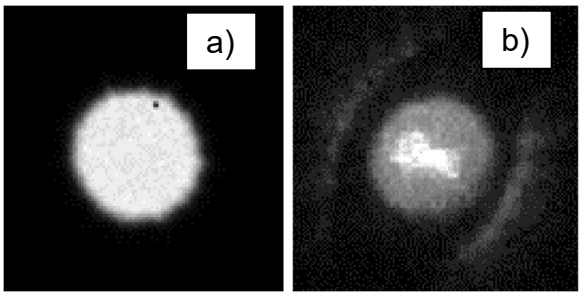

Fig. 1 Bright field disk taken from (a) vacuum and (b) thick region.

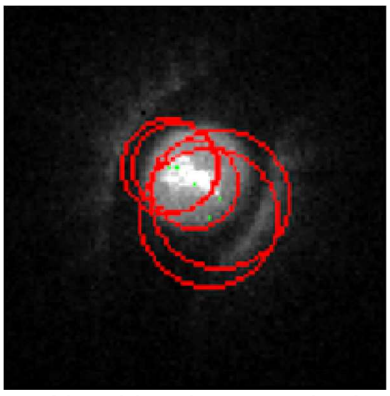

Fig. 2. Algorithm incorrectly detecting the disk due to distortions.

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TRANSIENT HEXATIC PHASE OBSERVED BY HIGH-COHERENCE ULTRAFAST ELECTRON DIFFRACTION

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Ultrafast transmission electron microscopy (UTEM) extends the capabilities of conventional TEM by femtosecond temporal resolution (Fig. 1a) [1]. This enables the investigation of ultrafast processes in spatially heterogeneous systems by means of imaging, diffraction and spectroscopy in a laser pump/electron probe approach. In this work, we track the formation of an incommensurate charge-density wave (IC CDW) phase by means of ultrafast electron diffraction (Fig. 1b). The high-coherence electron source of the Göttingen UTEM [2] allows us to infer the three-dimensional establishment of long-range order from CDW rocking curves at every stage of the dynamics. Within the material layers, the initially broadened shape of the CDW diffraction spots suggest a transient state with intact bond-orientational order, but reduced translational symmetry (Fig.1c). This state is characterised by a loss of stacking order evident from a broad distribution of scattered intensity along the out-of-plane direction (Fig.1d). We identify this quasi-2D intermediate as a transient hexatic phase [3] that is present prior to the establishment of the CDW stacking sequence.

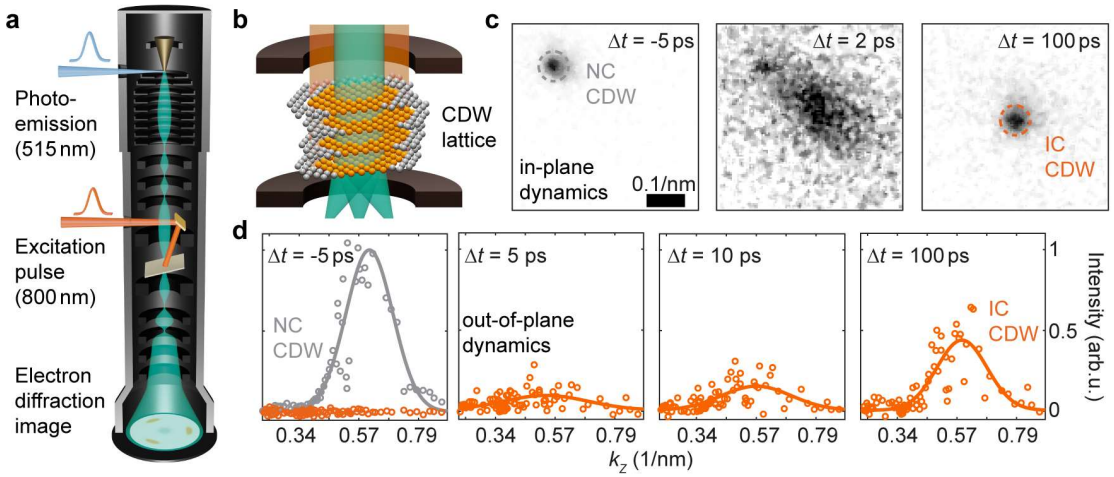

Fig. 1. **a** UTEM schematics. Photoemitted ultrashort electron pulses (green) probe laser-induced structural dynamics. **b** The excitation drives a transition between two CDW phases in a layered material (grey: nearly-commensurate (NC) CDW; orange: IC CDW). **c** In-plane CDW diffraction spot shape at different temporal delays Δ*t*. **d** Rocking curve of the CDW reciprocal lattice rod at representative temporal delays.

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OPTIMIZATION OF IMAGING CONDITIONS FOR COMPOSITION DETERMINATION BY ANNULAR DARK FIELD STEM

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The determination of composition on an atomic scale is possible by comparing aberration-corrected high angle annular dark field scanning transmission electron microscopy (HAADF-STEM) images and complementary image simulations [1, 2]. Optimization of ADF imaging parameters raises the accuracy of the results. In this work, a simulation study is performed to investigate the effect of inner and outer detection angles, probe semi-convergence angle, electron dose, and image sampling. The simulation study is conducted on Ga(AsBi) and (GaIn)As at a range of thickness between 10 and 40 atoms per atomic column. The results reveal the critical role of probe semi-convergence and detection angles. The optimum semi-convergence angle for all investigated conditions is defined as around 21 mrad. The effect of detection angle varies by the material system and thickness. In addition, a minimum sampling of the image and a minimum electron dose are favorable. The applicability of the method is investigated by determining the composition of a sample containing a $GaAs_{1-x}Bi_x$ QW, applying the optimized parameters. Although choosing the semi-convergence angle, electron dose, and sampling is rather straightforward, flexible detection angles can be only obtained by 4D-STEM with the help of a fast, pixelated detector. Fig. 1 depicts an ADF image using the optimized parameters. The resulting concentration profile obtained by the optimized parameters shows good agreement with the XRD results (Fig. 2). For comparison, the result using the optimum detection angle (blue line) is compared to the one at a typical angular range (red line), which shows the significance of optimization of detection angle.

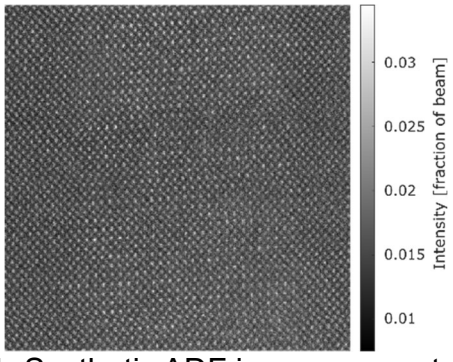

Fig. 1. Synthetic ADF image generated from a 4D data-set at the optimum detection angles. Reprinted from [3].

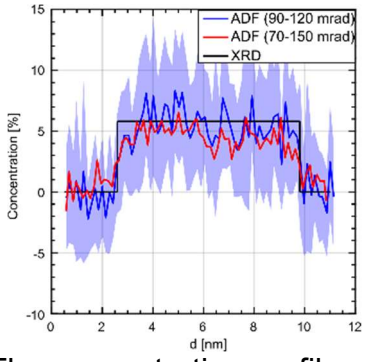

Fig. 2. The concentration profile achieved by ADF-STEM compared to XRD. Reprinted from [3].

References:
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DIRECT ELECTRON PTYCHOGRAPHY OF STRONG PHASE OBJECTS

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Developments in camera technology are making ptychography ever more promising [1]. Direct noniterative techniques such as the single side band (SSB) and Wigner distribution deconvolution (WDD) methods are fast, highly dose efficient and can clearly reveal light atoms [2-4]. However, the theory used to explain the SSB and WDD methods usually assumes the sample is thin and weak. Here we investigate the ability of direct ptychography to reach beyond the most simplistic theoretical descriptions and provide high quality images for typical materials science samples. Figure 1 displays experimental and simulated data from a $SrTiO_3$ sample with an estimated thickness of 28 nm. The experimental ptychographic images clearly show the Sr, Ti, and O columns. However, at zero defocus the simulated ptychography exhibits complex contrast reversals. Interestingly, by applying a small defocus, the contrast reversals are removed. A lower dose example with Fe_3O_4 using microsecond dwell time 4D STEM from a Timepix3 is shown in Figure 2. The clarity of the SSB image far exceeds that of the other signals, including the integrated center of mass (iCoM) image. We point out that defocus can be inserted post acquisition to remove contrast reversals in both SSB and WDD ptychography.

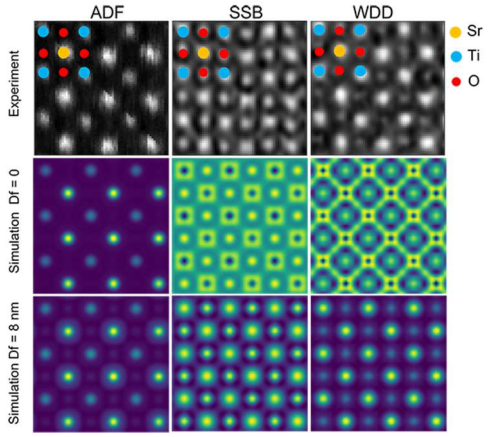

Fig. 1. $SrTiO_3$ experiment and simulations

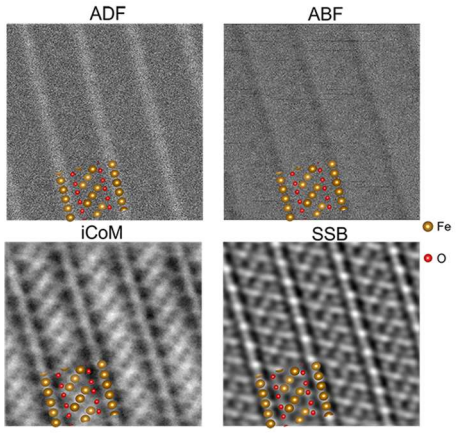

Fig. 2. Fe_3O_4 with Timepix3 data

References:
[1] D. Jannis et al., Ultramicroscopy **233**, 113423 (2022).
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[4] H. Yang et al., Ultramicroscopy **180,** 173-179 2017).

ATOMIC RESOLUTION STEM MAPPING OF MOMENTUM TRANSFER AND ELECTRON ENERGY LOSS

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Momentum-resolved STEM (4D-STEM) is an intensely researched area of electron microscopy with significant impact on the field as a whole [1]. Resolving this data also in energy-loss would allow for much more relevant information. To facilitate this, we use a high beam convergence angle and configure the spectrometer to single-focus EELS mode, which projects energy vs. projected momentum onto the detector, which we then record for every probe position. Consecutive scans with a 90° rotation in momentum space at the spectrometer entrance plane were acquired and non-rigidly registered [2] to obtain information about two momentum and one energy axis from the same location, as depicted in Fig. 1. An HAADF of the used monolayer hBN sample is depicted in (a) and of the template-matched region in (b) with a single momentum-resolved spectrum from the position marked in (b) given in (e). This allows not only to obtain core-loss maps, as depicted for B and N in (c) and (d), but also to obtain center-of-mass (COM) data as shown in (f) and the derived charge density in (g). Moreover, COM maps and their divergence can also be acquired from core-loss (here: B K-edge) electrons, as demonstrated in (h) and (i), which will offer novel capabilities of material analysis. This information should allow to retrieve directional information from non-degenerate electron orbitals and the acquisition scheme can also be extended to low-loss EELS.

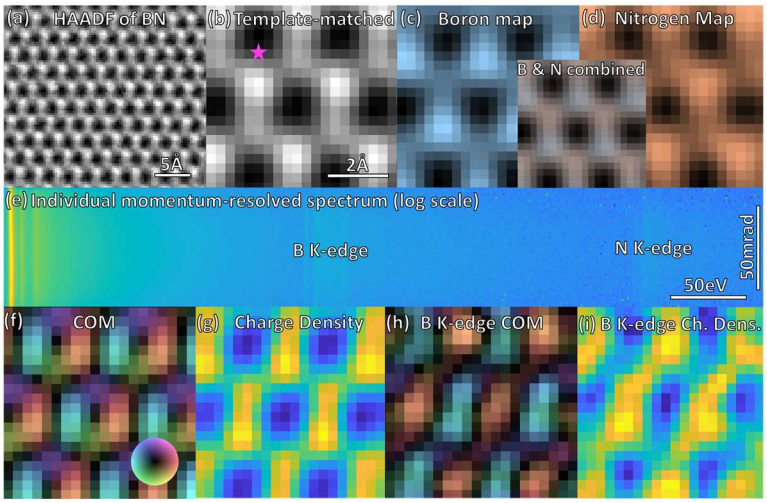

Fig. 1 5-dimensional STEM data consisting of two spatial, two momentum and an energy axis, allowing for core-loss mapping, conventional COM and core-loss COM.

References:
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[3] Acknowledgements: We thank Toma Susi (Universität Wien) for the hBN sample.

PTYCHOGRAPHY FOR CHARGE TRANSFER SENSITIVITY AND 3D STRUCTURE DETERMINATION IN 2D MATERIALS

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Microsecond dwell time 4D scanning transmission electron microscopy (STEM) enables seamless integration of ptychography into conventional rapid scan annular dark field (ADF) STEM workflows [1]. Single side band (SSB) ptychography is a simple and direct phase retrieval method providing high-quality phase images [2]. Here, we apply SSB to directly visualize atomic structures and defects in monolayer WS_2 which provides a much stronger signal than the ADF, especially for light atoms neighboring heavy elements such as the S in WS_2. The sensitivity of the SSB to charge density allows us to detect the effects of bonding (Fig 1). Furthermore, we show how to combine SSB with ADF to obtain the 3D structure of monolayer CuI at picometer precision at low doses using very few tilts [3]. This is possible because the shift of Cu and I can be reliably tracked when the sample is tilted and the SSB provides far clearer images at low doses, especially for the light elements (Fig 2).

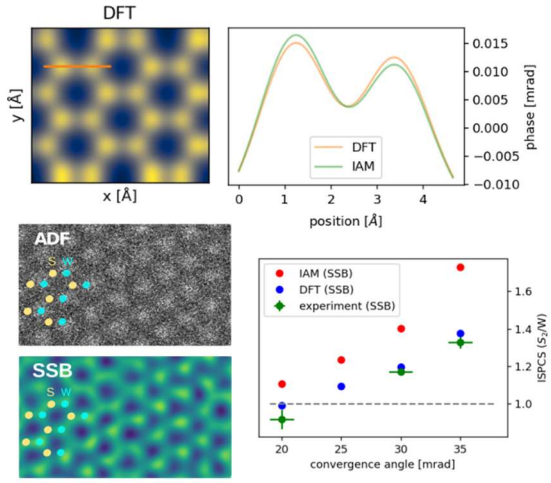

Fig.1: Simulated and experimental data of single layer WS_2

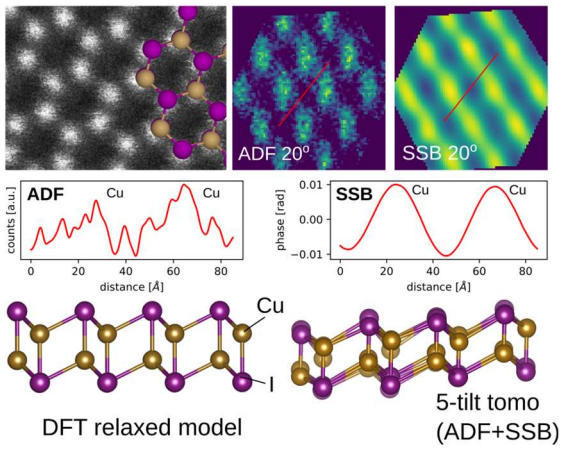

Fig.2: 3D reconstruction of monolayer Cu using ptychotomography

References:
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FAST 4D STEM ACQUISITION WITH EVENT BASED ELECTRON DETECTION

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Fast direct electron detection cameras are rapidly becoming one of the key components in a modern STEM setup. Such detectors allow one to collect a full diffraction pattern at every probe position in what is known as 4D STEM. The tremendous amount of data generated during these scans enables more dose efficient reconstruction algorithms, such as iCOM or SSB ptychography. However the speeds at which these detectors operate has remained an order of magnitude slower than conventional methods in which the probe is typically scanned with dwell times of 1 to 10 µs. In this work we demonstrate the use of an alternative event driven detector architecture to completely remove the restriction in scan speed for 4D STEM [1]. Event driven operation allows one to take advantage of event sparsity to achieve enhanced time resolution by avoiding the readout of pixels containing zero counts. In STEM one can reduce the dose imposed on a sample per unit area by reducing the probe current and dwell time, both of which increase the sparsity of the events in each probe position. Here, we present results from 4D STEM performed with a Timepix3 detector at dwell times of a few microseconds down to 100 ns at both 60 and 200 kV accelerating voltages and discuss the characterization of the Timpix3 detector.. Furthermore, we show how the speed enhancement facilitates multiply scanned 4D STEM to increase the signal-to-noise with minimal susceptibility to drift (see Fig. 1)

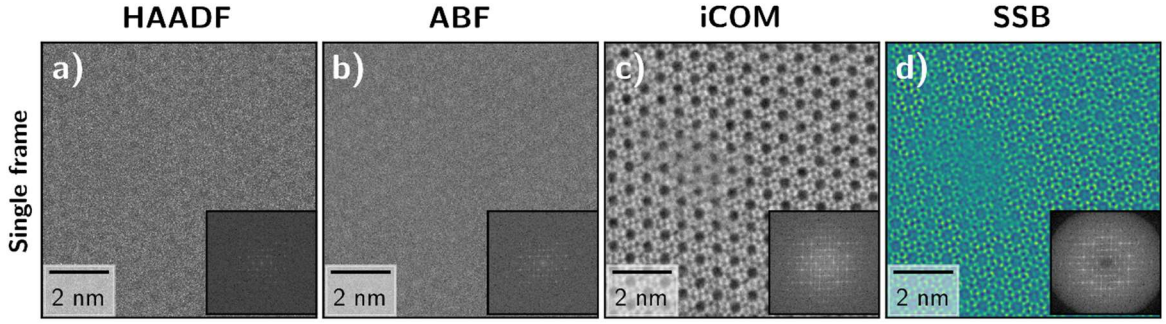

Fig 1. A 1024x1024 probe position scan at 1 µs dwell time of a silicate-1 zeolite. The reconstructed signals from the different methods are shown.

References:
[1]D. Jannis et al, Ultramicroscopy **233**, 113423, (2022).
[2]We acknowledge funding from G093417N, G042920N, No 823717 – ESTEEM3. and European Research Council Grant no. 802123-HDEM.

MAGNETIC FIELD MAPPING OF 2-DIMENSIONAL AND 3-DIMENSIONAL FRUSTRATED MAGNETS USING OFF-AXIS ELECTRON HOLOGRAPHY

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Artificial spin ices can be formed from geometrically-frustrated arrangements of two-dimensional magnets. Magnetostatic interactions between the magnets in such an array can lead to rich emergent behavior, including the formation of magnetic monopoles, chiral dynamics and phase transitions. The ability to map magnetic fields with high spatial resolution is essential to gain an understanding of this behavior. When compared with 2-dimensional systems, magnetic frustration in 3-dimensional systems has been studied much less intensively. Here, we use off-axis electron holography to measure magnetic fields in an artificial two-dimensional spin ice lattice formed from permalloy (Py) nanomagnets and in a three-dimensional Py magnetic gyroid structure. The square lattice of Py elements for chiral ice pattern was prepared on SiN membrane using lift-off lithography. The magnetic gyroid structures were fabricated using block co-polymer templates and electrodeposition of Py. Experiments were recorded both in magnetic-field-free conditions and in magnetic fields up to 1.5 T. The recorded electron optical phase images of the Py islands were analyzed using a model-based iterative reconstruction algorithm, in order to determine maps of projected in-plane magnetization. Magnetostatic interactions between the Py elements and the stray field distribution in chiral ice [2] were studied by applying both in-plane and out-of-plane magnetic fields to the sample. The experimental results were compared with micromagnetic simulations, which were created based on input from TEM images. The experimental results and micromagnetic simulations indicated that the gyroid structure was able to support many different magnetization configurations [3].

References:

[1] The authors acknowledge the collaboration with S. Gliga, S. Finizio, J. Llandro, A Kákay. This project has received funding from the ERC under the European Union's Horizon 2020 under grant agreement 856538 and the DFG under project No 405553726.
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[3] J. Llandro *et al.*, Nano Letters **20**, 3642 (2020)

APERTURED EMCD AT ATOMIC SIZED ELECTRON BEAMS

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The quest to observe an EELS signal that stems from a magnetic circular dichroic interaction has stimulated the development of several EELS based methods in the past decade. Hitherto it seems that only the original acquisition geometry, proposed by Schattschneider et al. was pursued for quantitative work. Using this approach, atomic plane resolution was demonstrated.[1] In original proposal, a crystalline, ferromagnetic sample is oriented in 2-beam orientation, i.e. away from a zone axis orientation. By nature, EMCD signal is small and can be perturbed by several factors such as sub-0.1 mrad orientation changes, non-optimal sample thickness as well as difficulties to position the electron probe on exactly the same atomic position for the acquisition of both corresponding q-dependent spectra. Here, we propose and apply an acquisition geometry in which the two q-dependent EELS spectra are acquired simultaneously thus erasing doubt on differences in sample orientation or electron probe position. For this purpose, a segmented aperture is introduced at the level of the entrance aperture of the spectrometer and spectra are acquired in the qE mode of the spectrometer. When the two aperture holes are positioned in the EELS entrance aperture in the 2-beam case orientation, we could demonstrate an improved signal to noise ratio and more stable signal as compared to the acquisition of two separate EELS spectra. Introducing a 4-hole aperture, we could simultaneously measure exact sample orientation and EMCD spectra. This made an exact measurement of the orientation dependence of the EMCD signal possible. Finally, we followed a theoretical work proposed by Rusz et al, which predicted an EMCD signal generated by an electron probe on the zone axis orientation.By introducing a more complex aperture into our spectrometer, we could, in fact demonstrate an EMCD signal on the zone axis orientation. In addition, this EMCD signal remained stable for convergence angles corresponding to electron probes smaller than a (111) lattice spacing in Fe crystal.

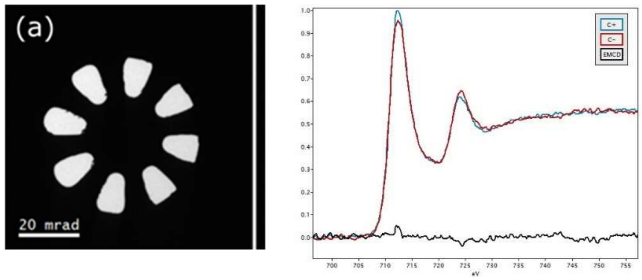

Fig.: a) Image of ventilator aperture, b) corresponding EELS and EMCD spectra.

References:
[1] Schattschneider,et al. (2006). Nat. 441(7092): 486. Rusz et al. (2016). Nat. Comm., 7: 12672; Wang et al. (2018). Nat. Mat. 17: 221. Negi et al. (2019). Phys.Rev.Lett. 122: 037201;. Ali et al. (2020). Phys.Rev.Res.

OPTIMISING DOSE AND SAMPLING EFFICIENCY IN PHASE CONTRAST ELECTRON MICROSCOPY

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Phase contrast electron microscopy has allowed the characterisation of otherwise unobservable phenomena. These include weak phase objects such as biological macromolecules and soft matter, very light elements particularly surrounded by heavy elements such as in 2D materials, oxides and semiconductors, free-space electromagnetic fields such as around field emission tips, electronic devices, and magnetic skyrmions. Those measurements are often hampered by two challenges. One is to image radiation-sensitive specimens at atomic resolution, preferably using ultralow dose and dose-efficient imaging methods, while the other is to map extended specimens or long-range fields with an extremely large field of view at nanometer (or better) resolution, ideally with minimum number of datasets and sampling-efficient imaging workflow. In this poster presentation, we will discuss how to tune parameters to optimise the dose and sampling for given spatial resolution, dose budget and field of view, and introduce a few different methods, particularly the variants of centre of mass and ptychographic imaging, for improved dose and sampling collection efficiency.

A NEW APPROACH FOR 3D QUANTITATIVE STEM USING DEFOCUS CORRECTED ELECTRON PTYCHOGRAPHY

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Electron ptychography is a powerful technique exploited to study the atomic structure of materials including those containing both light and heavy elements. In electron ptychography, first, a series of electron diffraction patterns (i.e. a 4D STEM dataset) are collected by scanning an electron beam (probe) across a specimen. Then, some mathematical algorithms (e.g. SSB, WDD and ePIE) are used to deconvolve the probe and object transfer functions from the 4D STEM dataset. The key assumption in these algorithms is that the probe function is constant for all the probe positions since the aberrations of the microscope's electromagnetic lenses are almost constant during the very short time of data acquisition. Although the lens aberrations can be assumed to be constant for each probe position in a dataset, the probe function is not unique since the geometry of the specimen at each probe position across the imaging area alter the defocus value of the probe (Figs. 1(a-c)). Thus, we have to use a defocus-corrected probe function for each probe position to calculate the object transfer functions. Here we show that it is possible to calculate the probe's defocus value using electron ptychography. Moreover, we demonstrate that 3D models of nanoparticles can be obtained from 4D-STEM datasets acquired simultaneously with HAADF images. Here, we calculated the number of Pd atoms for each atom column observed in a HAADF image from a Pd nanocube (Fig. 2(d)), and then we measured the height of those columns from their apparent defocus extracted from the WDD ptychographic phase reconstructed from a 4D-STEM dataset acquired simultaneously with the HAADF image. Finally, the 3D model of the Pd nanocube were simply reconstructed as we had the number of atoms in each column as well as the height of those columns (Fig. 2(e-g)). We expect this approach to be applicable to reconstruct not only an accurate ptychographic phase but also a 3D model of any other nanostructure.

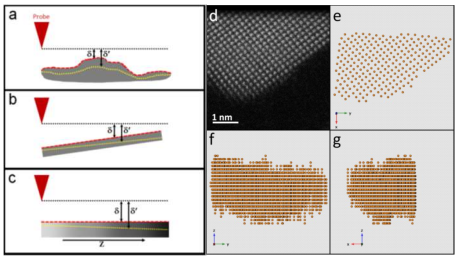

Fig. 1 Absolute defocus (δ) and apparent defocus (δ') levels for (a) a sample with an uneven surface, (b) tilted sample and (c) sample with compositional variation. (d) ADF image obtained from a Pd nanocube. (e-g) 3D reconstructed model in three different viewing direction for the nanocube shown in (d).

PRECESSION SEGMENTATION TO INCREASE SPED IMAGE RESOLUTION

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Scanning precession electron diffraction (SPED) has been utilized for various applications, e.g., for strain-, orientation-, and phase mapping, and shown significant advantages over alternative techniques. Precession allows us to sample more of reciprocal space, and the diffraction reflections present appear kinematic-like. Despite the advantages, SPED has some drawbacks, one of them being an increased difficulty of instrument alignment. A common encounter is the probe shift on the specimen, which can arise from aberrations due to a high off-axis beam tilt [1], or simply from a slight misalignment leading to an offset between the pivot point plane and the probe crossover or sample height. The probe shift is, however, periodic, and has the potential to be corrected. In this talk we introduce the concept of precession subframing to reduce the blur in images reconstructed from SPED scans. The subframing process relies on segmenting the precession path by recording an integer number of frames (> 1) at each scan position. The recorded dataset can then be sliced, and virtual images reconstructed from each sliced scan. If probe shifts are present, an apparent displacement of features between the reconstructed images can be seen, which can be minimized by rigid correction performed in SmartAlign [2]. Summing the corrected image reconstructions reveals a reduction in blur when compared to a non-corrected scan, as can be seen in Fig. 1. Precession subframing is easy to implement and can be used to fix small pivot point misalignments, as well as potentially increase the resolution in SPED images where aberrations at a high precession angle led to probe shifts.

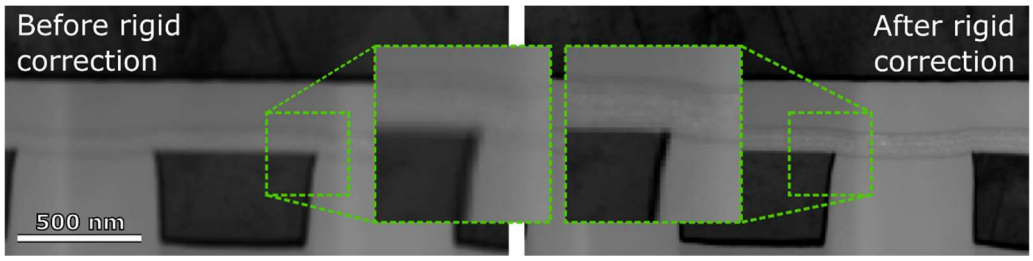

Fig. 1. Virtual bright field reconstructions of a segmented SPED scan of a CPU cross section sample. Image shows the reconstructions before and after rigid correction.

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[2] L. Jones et al., Adv Struct Chem Imaging **1**, 8-23 (2015).

REAL TIME INTEGRATION CENTER OF MASS (RICOM) RECONSTRUCTION FOR 4D-STEM

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Multiple reconstruction methods have been created to process the diffraction patterns collected in scanning transmission electron microscopy (STEM) experiments [1, 2]. They enable image reconstruction with higher resolution or with fewer electrons. While most of the reconstruction methods, with few exceptions [3], work after the complete dataset is collected, operations on the microscope such as searching and tuning of the optical system remains to rely on the more traditional imaging methods that generate live updated results. In this work, an imaging method, called real-time integrated center of mass (riCOM) is proposed, which allows quick and real-time reconstruction of a 2D image of the object by processing diffraction patterns separately and update their contribution to a larger array of the reconstructed image (Fig. 1). The proposed method shows reconstruction quality comparable with other non-iterative methods (Fig. 2). The discussion also covers how the frequency components and noise contribute to the reconstructed images. Moreover, it will be demonstrated how to obtain a real-time reconstruction while tuning the optical system including a discussion of the benefits as well as unexpected or unwanted effects it brings.

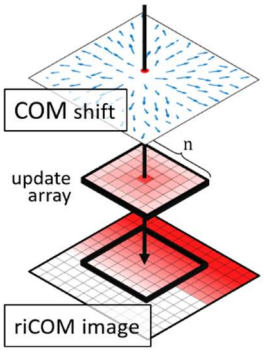

Fig. 1. A schematic of the riCOM reconstruction algorithm.

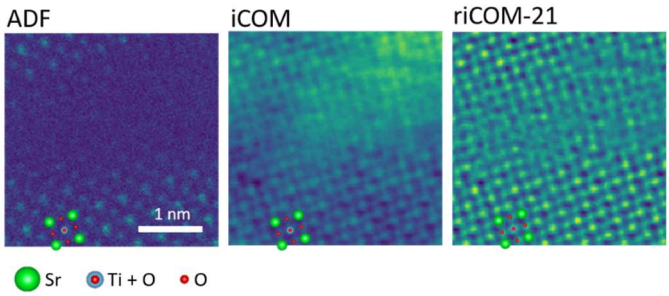

Fig. 2. Experimental results from $SrTiO_3$ of different imaging methods. RiCOM clearly shows the atomic structure and the ability to image light atomic columns.

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[4] The authors acknowledge financial support from the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation program (Grant Agreement No. 770887 PICOMETRICS), funding from the European Union's Horizon 2020 research and innovation program under grant agreement No. 823717 ESTEEM3, and funding from TOP BOF project. The direct electron detector was funded by the Hercules fund from the Flemish Government.

FAST AUTOMATIC ANALYTICAL PARTICLE ANALYSIS USING AN AI GUIDED SMART SCAN STRATEGY

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The shape, size, chemical composition and distribution of nanoparticles influence their properties and function - especially in catalysis applications. The relevance of such measurements depends greatly on a statistically significant analysis on sufficiently large numbers of particles in an efficient way. Therefore we developed a novel deep learning-driven fast workflow that spends most acquisition time on particle regions, and provides an automated analysis of the particles. We demonstrated a relative speed increase of two orders of magnitude - and a corresponding dose reduction - compared to the classical procedure based on full frame XEDS mapping. A trained neural networks identifies the position and shape of individual particles of interest in a first scan automatically and a second adaptive scan of only these identified ROIs speeds up the analytical measurement. The custom scan pattern functionality enables visiting individual pixel positions with precision higher than the original reference image pixel size. There are multiple options to control the XEDS scanning like visit only particle centers, or visiting small geometric areas (rectangle, circle) or scan the complete particle shape. We applied this adaptive scan particle workflow to characterize AgPd particles in a titanium oxide matrix and determined on more than 4000 particles their size, shape distribution and chemical variation. (Figure 1) Additionally the AI network is re-trained for similar applications and examples of the benefits of speed and dose reduction are given.

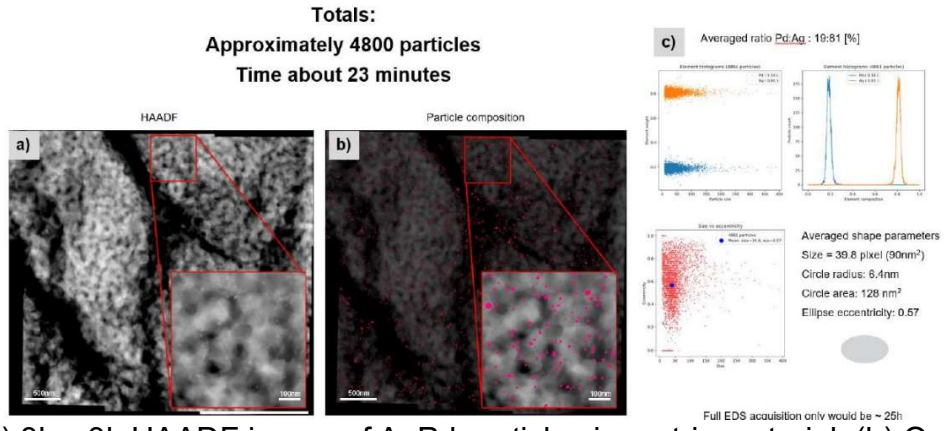

Fig. 1. (a) 3k x 3k HAADF image of AgPd particles in matrix material. (b) Overlay of identified particles using the trained network. (c) automatic statistical analysis of shape size, morphology, and composition of 4000 particles within 23 minutes total AI guided workflow time.

PHASE OBJECT RECONSTRUCTION FOR 4D-STEM WITH MACHINE LEARNING

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We explore the possibility to use machine learning for the live reconstruction of phase images from 4D scanning transmission electron microscopy (4D-STEM) data with super-resolution. The process is illustrated in figure 1: A patch of the phase object ① of a 4D-STEM dataset ② is reconstructed by extracting a 3x3 kernel of adjacent convergent beam electron diffraction patterns (CBEDs) ③, using a convolutional neural network (CNN) ④ to reconstruct the exit wave of the central CBED ⑤ and using the phase object approximation to reconstruct the object patch ⑥. Patches are then combined successively to yield the full phase object, which makes live processing during an experiment feasible. The CNN can retrieve phase information beyond the aperture angle, enabling super-resolution imaging. We evaluate the dose dependence and image contrast formation characteristics, showing a contrast dependence on thickness and atomic column type as well as good noise robustness. The combination of super-resolution and low dose imaging capabilities, as well as intuitive image contrast characteristics makes the approach unique among live imaging methods in 4D-STEM.

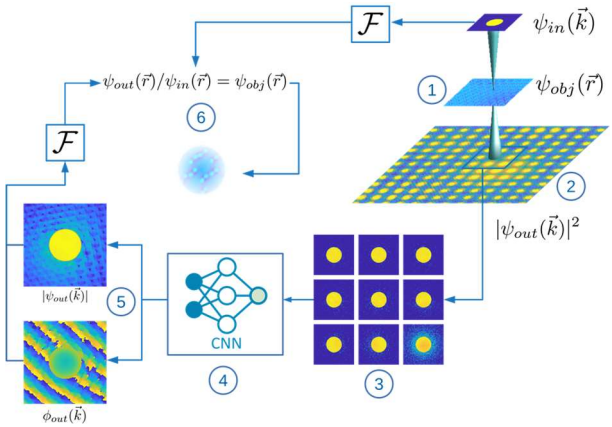

Fig. 1 General Workflow of the phase object reconstruction method

References:

[1] We acknowledge funding from the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation program (Grant Agreement No. 770887 PICOMETRICS and No. 823717 ESTEEM3), funding from the University of Antwerp through a TOP BOF project, funding through the Hercules fund from the Flemish Government and support by the FWO and FNRS within the 2Dto3D project of the EOS program (grant number 30489208).

4D-STEM FOR 3D STEREO IMAGING AND AUTOMATIC 3D RECONSTRUCTION OF CURVILINEAR OBJECTS

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Three-dimensional (3D) stereo reconstruction of curvilinear crystal defects known as dislocations can advantageously replace conventional electron tomography [1,2]. Hence, we developed a methodology that combines tilt-less 3D imaging [2] with deep learning convolutional neural networks (CNN) to automatically reconstruct the dislocations network in the 3D volume of a sample. Our algorithm automatically detects the dislocation structures and matches them across views without prior assumptions about their 3D shape [3]. By tilt-less 3D imaging on a fast pixelated STEM detector [4] and using our algorithm, we demonstrate the 3D reconstruction of dislocations from virtual stereo-images extracted from the 4D STEM dataset (Fig.1). This imaging technique delivers the possibility to obtain a larger stereo-angle between the views and to optimize the balance between SNR and angle resolution. Our automatic approach allows us to perform fast and reliable 3D reconstruction represented in the fractional crystallographic coordinates.

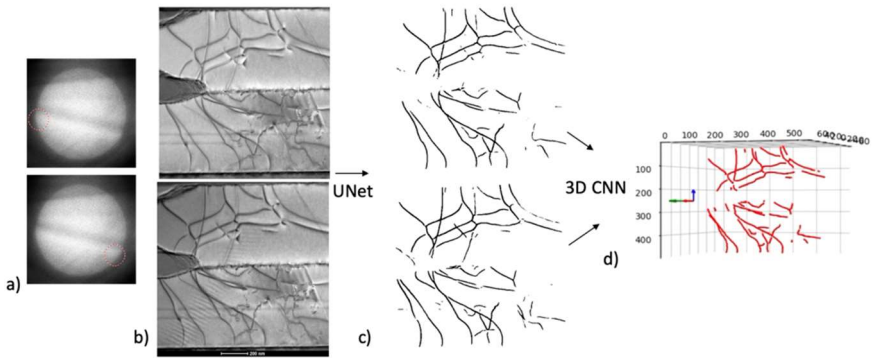

Fig. 1. 3D reconstruction procedure from 4D STEM dataset: a) STEM diffraction pattern in 2-beam diffraction condition **g**=(200) acquired on a pixelated detector, b) stereo-pair of virtual STEM images of TiAl alloy (stereo-angle is 6 degrees) from regions highlighted by circles in a), c) corresponding detected dislocations by UNet neural network, d) final 3D reconstruction by 3D CNN.

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[1] A Jácome et al., Ultramicroscopy, 195 (2018), 157-170.
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[4] C Ophus, Microscopy Microanalysis. (2019), 563-582.

AUTOMATED ANALYSIS OF HIGH-RESOLUTION TRANSMISSION ELECTRON MICROSCOPY IMAGES OF METALLIC NANOPARTICLES USING NEURAL NETWORKS

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Metallic nanoparticles are of high interest in catalytic or biomedical applications due to their high surface to volume ratio and their chemical and physical properties. These properties critically depend on the particle structure and size. Therefore, it is essential for nanoparticle research to get detailed data on their structure and size, as well as on the synthetic parameters that determine them. High-resolution transmission electron microscopy (HRTEM) is a prominent method to acquire such data. Nanoparticle size, morphology and also the crystal structure of individual particles can be extracted from HRTEM images. The analysis of HRTEM images, which can show up to hundreds of particles per image, is a tedious process when done manually. This becomes even more obvious if multiple images are to be analysed, either to get more statistically significant data or for the analysis of *in-situ* TEM experiments. Therefore, we have developed a MATLAB based automated image processing program which uses neural networks to detect and classify nanoparticles in HRTEM images. The program first uses a neural network to detect the particles in the HRTEM image by performing image segmentation. The network is trained on a large dataset of HRTEM images from nanoparticles of different metals and sizes. The segmentation image is then further processed to enable the extraction of shape-related information about the particle like circularity, convexity, equivalence diameter and Feret diameter. The particles in the image can then be further analysed in regards to their crystal structure. For this step, another neural network is used, which classifies the particles with respect to the crystallographic nature of individual particles (e.g. single-crystalline, polycrystalline). This program enables the automated analysis of large quantities of data and gives more insight into nanoparticle structures compared to a manual analysis.

AUTOMATED ALIGNMENTS WITH REAL-TIME ELECTRON RAY TRACING

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A considerable challenge for new users of a Transmission Electron Microscope (TEM) is to understand the microscope alignment state, and how the electron beam spot on the detector changes as they adjust each component. To assist in the teaching and training of new microscopists we have developed a ray tracing-based tool which aims to help TEM users to comprehend how changes to the alignment affect the beam. The approach we have taken is to use a numerical ray tracing algorithm, written in Python, which makes use of GPU parallelisation. The system presented here is a basic magnetic deflector lens system consisting of 2 deflectors and a round focusing lens, which will enable users to gain an understanding of how the tilt-pivot alignments work within the microscope[1]. Our objectives are twofold: To create an interactive platform that enables the exploration of automatic control schemes in a simulated TEM and create an educational tool to enhance the TEM community's knowledge of the primary TEM alignments, via a software-based teaching aid.

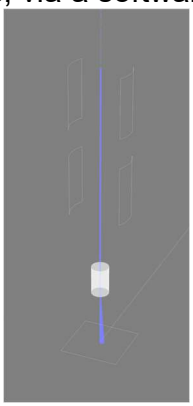

Fig. 1 Interactive 3D model of the TEM with three components. Two deflectors and a magnetic lens.

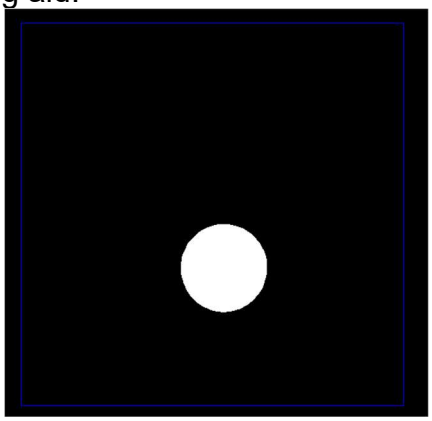

Fig. 2 Resulting beam spot on the detector plane.

References:

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LARGE AREA MULTISLICE SIMULATIONS A NOVEL APPROACH

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Multiple multislice algorithms are available for Transmission Electron Microscope (TEM) image simulations [1], including abTEM [2], and Dr. Probe [3], used to validate experimental images, a common limitation amongst these algorithms is the size of the field of view, primarily due to the limited bandwidth of memory on standard personal computers. A limited field of view can restrict the ability to validate and interpret an image, which can be particularly problematic for thick amorphous specimens, or large non-crystalline materials. One approach to overcome this problem is to split the model into a series of smaller regions, tiles, which can simulate each subregion/tile of the image separately and stitch the simulations together, each title fits into the available memory and is calculated *via* the standard multislice methodologies [4]. Here we present progress towards a tile based parallelised algorithm, which can simulate large field of view images, enabling validation and interpretation of transmission electron micrographs. Fig. 1 shows the buffer region used in the tile approach to insure there is continuity and consistency between the tiles.

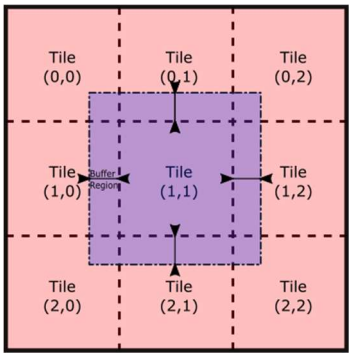

Fig. 1 The fragmenting of the atomic layer into tiles with a buffer region overlapping with the neighbouring tiles.

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ADAPTABLE AND AUTOMATED ANALYSIS FOR VARIOUS NANOPARTICLE SYSTEMS USING NNPIPE

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In the recent years, researchers successfully applied machine learning (ML) and deep learning (DL) [1] on electron microscopy (EM) data to solve problems in the domains of physical and life sciences [2]. Those approaches are of particular importance for experimental techniques such as in situ (Scanning) Transmission Electron Microscopy ((S)TEM) where large datasets are generated and for which manual or semi-automated analysis methods become unsuitable [3]. In the case of nanoparticle catalysts, many previous studies discuss systems in which nanoparticles are clearly distinguishable and are deposited on topologically uniform support materials. Furthermore, training data is often prepared by manual annotation introducing unintentional bias and workable image resolutions are limited by available GPU resources. Herein, we present nNPipe as an automated analysis method that consists of two standalone Convolutional Neural Networks (CNNs). The modular structure of nNPipe allows for processing of experimental images of various size at high speed without compromising on the inference precision. Large training datasets are obtained by using a smaller number computationally generated sample models of two topologically different nanoparticle catalyst systems. The network pipeline is subsequently applied on experimental 2048x2048 HRTEM images and high fidelity analysis performance is demonstrated. Lastly, we show adaptability towards new, nanoparticle systems by proposing a quick-deploying retraining scheme.

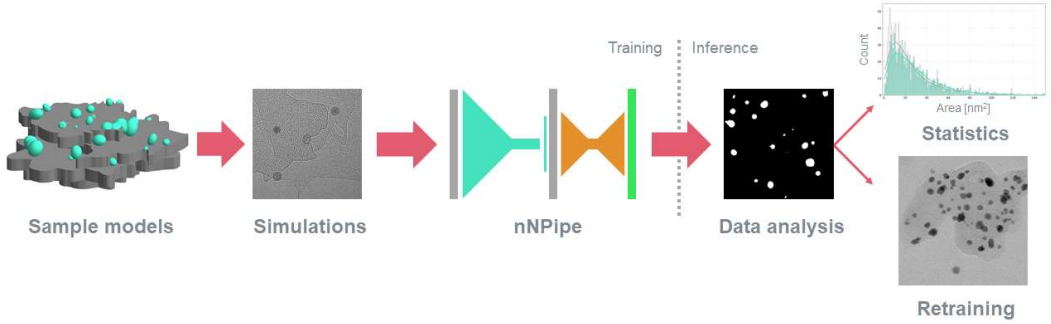

Fig. 1 Training and inference scheme of the nNPipe method

References:
[1] Y. LeCun, Y. Bengio, G. Hinton, Nature **521**, 436-444 (2015).
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SYNTHETIC DATA, MACHINE LEARNING AND EDGE DETECTION TO MEASURE PARTICLES IN TRANSMISSION ELECTRON MICROSCOPY

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As Transmission Electron Microscopes (TEM) develop, the data acquisition rate is quickening. Current *in situ* cameras exhibiting 23000 frames per second performance [1]. With an ever-increasing volume of data output, techniques are required to analyze these data. These data encompass a range of material characteristics, such as morphological, electronic and chemical structure change. This information will lead to a deeper understanding of how materials evolve in various environments. The application of Machine Learning (ML) is becoming ubiquitous across scientific domains, including the field of TEM [2] [3]. A sub-field of ML is supervised ML, where an algorithm is trained using a pre-labelled dataset. This has proven to be a state-of-the-art method of segmenting micrographs [4]. The rate-limiting step in supervised ML is labelling the data. To create a ML algorithm to segment particles of interest, a binary mask of each image in a dataset is required, indicating which pixels in the image represent particles, and which do not. We have developed synthetic data generators, which provide an automatically labelled dataset. The generator can produce any number of images, which are then used to train a supervised ML algorithm. We will present the application of this synthetic data strategy to the segmentation of nanoparticles in TEM micrographs, and the subsequent measurement of specimen size and morphology.

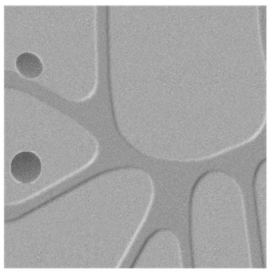

Fig. 1. Synthetic TEM image.

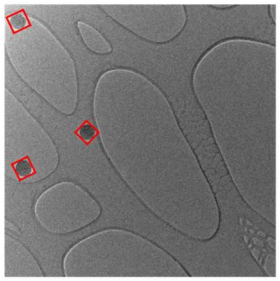

Fig. 2. Particles detected in real TEM image.

References:

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ENHANCING THE DOSE LIMITED SIGNAL FROM THICK BIOLOGICAL SPECIMENS WITH C_C CORRECTION

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The success of single particle electron cryomicroscopy has resulted in an exponential growth in the number of protein structures determined by electron microscopy [1]. As high-resolution structural information becomes abundant for a large number of biological molecules, a natural progression is to use this information to identify the position and orientation of these molecules in their native cellular environments. *In situ* imaging requires specimens that are thicker than those used for single particle analysis; this limits the obtainable signal due to multiple elastic and inelastic scattering from the surrounding water and other molecules. As a result, successful *in situ* imaging has so far been limited to large macromolecular complexes that are of order a million Daltons in mass. Here we investigate the potential of using chromatic aberration correction for improving phase contrast imaging of thick biological specimens. Although inelastic scattering causes a quantifiable amount of loss in spatial coherence, inelastically scattered electrons that carry elastically scattered information in their wavefront can be used to enhance the high-resolution phase contrast signal (Figure 1). Based on these measurements, we show that a phase plate in conjunction with chromatic aberration correction may be particularly useful for imaging smaller molecules inside cells.

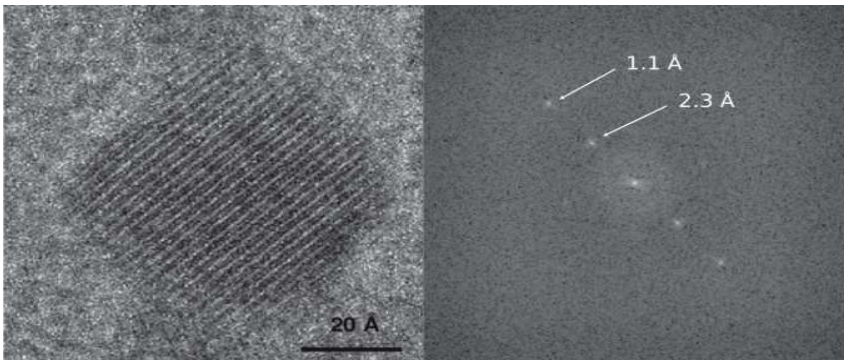

Fig. 1 Phase contrast image of a platinum particle on a thick aluminium/carbon foil, with a mass density equivalent to 4000 Å thick water ice. The micrograph was taken at 15 eV energy loss and 300 keV primary energy.

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THE 3D STRUCTURE OF LIPIDIC FIBRILS OF α-SYNUCLEIN

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The pathological causes for α-synucleinopathies, including Parkinson's disease (PD), multiple system atrophy, and dementia with Lewy bodies (DLB), are largely unknown, but accumulating evidence suggests that these diseases are related to the misfolding and aggregation of α-synuclein (αSyn) into amyloid fibrils [1,2]. A pathological hallmark for PD and DLB is the presence of large neuronal inclusions called Lewy bodies (LB) and αSyn fibrils have been identified as one constituent of LB [3,4]. Studies on the composition of LB extracted postmortem from brain tissue of PD-confirmed patients revealed that lipids and membranous organelles are a significant component of LB [5,6]. However, although molecular interactions between αSyn and lipids have been discussed as relevant for PD pathology for decades [7], insights into the interactions of αSyn fibrils with phospholipids have remained elusive. Here, we present the cryo-electron microscopy (cryo-EM) structures of six αSyn fibrils aggregated in the presence of phospholipid vesicles [8], providing structural evidence for specific lipid-fibril interactions. These structures reveal that phospholipids favor a novel protofilament fold and mediate the unique arrangement of protofilaments. Furthermore, our findings also provide a structural rationale for fibril-induced lipid extraction, a mechanism likely to be involved in the development of α-synucleinopathies.

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α-SYNUCLEIN POLYMORPHS IN NEURODEGENERATION

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Synucleinopathies, such as Parkinson's disease (PD) and Multiple System Atrophy (MSA), are neurodegenerative diseases featuring the intracellular fibrilization of the protein alpha-synuclein (αSyn). Many polymorphs of αSyn fibrils exist, and αSyn growth is able to be templated by pre-existing fibrils. Here we obtained fibrils by seeding monomeric αSyn with PD and MSA patients' brain homogenates using protein misfolding cyclic amplification (PMCA), and determined their structures using cryo-electron microscopy to near-atomic resolution. The two fibrillar polymorphs have a similar protofilament fold to each other and other previously determined *in vitro* and *ex vivo* αSyn fibrils, yet differ in their inter-protofilament interface and helical symmetry. We further characterize the fibrils in primary mouse oligodendroglia, showing that those amplified from MSA are the more potent in recruiting endogenous αSyn and effecting a redistribution of the phosphoprotein TPPP/p25α from the myelin sheath to the cell soma; both are critical features of oligodendroglial dysfunction underpinning MSA pathology, suggesting these structural differences could manifest clinically.

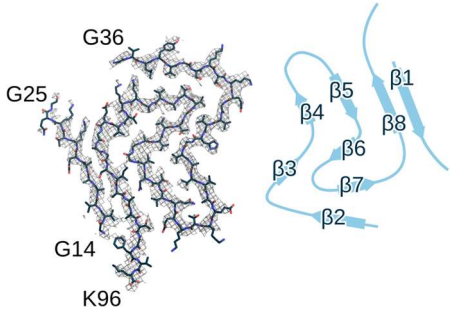

Fig. 1. PD polymorph cross-section.

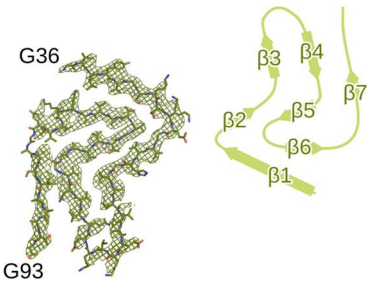

Fig. 2. MSA polymorph cross-section.

MICROSTRUCTURAL CHARACTERIZATION OF CELLULOSE NANOFIBRES USING SCANNING ELECTRON DIFFRACTION

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Scanning electron diffraction (SED) is a 4D-STEM technique in which a nanometer-sized, near parallel probe is scanned across the sample. Acquisition of diffraction patterns at each beam position results in a 4D data set, simultaneously sampling both real and reciprocal space. The use of direct electron detectors and low dose conditions have now enabled studies of beam sensitive materials [1]. Virtual bright-field and dark-field images are later constructed in post-processing through precise positioning of virtual apertures, using the open-source software pyXem.
This current work reveals the microstructure of cellulose nanofibres where discrete Bragg discs confirm the crystalline nature of the fibres (fig.1). Further analysis of the diffraction patterns shows that they represent two different orientations of the cellulose crystal structure, which implies a twisting of the fibre. These characteristics are important for future applications of cellulose-based hybrid materials.

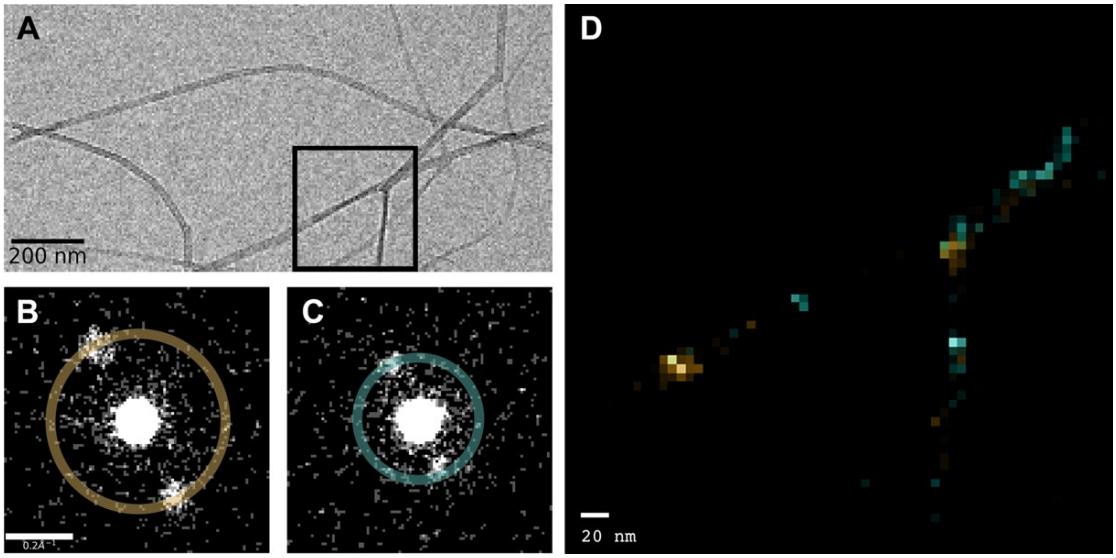

Figure 1. (A) Virtual bright-field image showing individual cellulose nanofibres extracted from tunicate. (B, C) Diffraction patterns from the SED data with annular virtual apertures are marked in yellow and green. (D) A combined virtual dark-field image, using the apertures from B and C, reveals the crystalline domains in two different orientations along with the fibres.

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VISUALISATION OF NATIVE ATG9 COMPLEXES USING IN SITU CRYO-EM

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Autophagy is a conserved process that allows for recycling of cellular components by sequestration into a double-membrane-bounded compartment which fuses with the lysosome for degradation. Among the core machinery that monitors the steps towards autophagy is Atg9, the only multi-spanning membrane protein. Atg9 forms a complex with Atg2 and Atg18 and is essential for extending the isolation membrane. So-called Atg9 vesicles, stored in the cytoplasm, provide a lipid basis for the isolation membrane. Recently a scramblase activity has been identified in both the human and yeast Atg9 [1,2]. In order to elucidate the exact mechanism of the Atg9-Atg2-Atg18 complex, I observed native Atg9 complexes and surrounding membrane environment using cryo-confocal microscopy followed by *in situ* tomography. Thus far, I have screened a range of *S. cerevisiae* knock-out mutants, tested different fluorophores and growth conditions. These optimizations enriched Atg9-containing punctae around lysosomal membranes and will improve the success of localization for down-stream FIB-milling and any associated image analysis such as subtomogram averaging.

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MEMBRANE ANALYSIS TOOLKIT – A QUANTITATIVE WAY TO ANALYSE PROTEIN LIPID MIXTURES FROM CRYO-EM IMAGES

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Cryo-EM is a powerful tool to study protein membrane interactions as it delivers high-resolution images of vitrified macromolecules including biological membranes. The quantitative analysis of protein-induced effects on the membrane structure is a challenging and time-consuming task as they are not amenable to established single-particle image reconstruction techniques. However, due to the high contrast of phospholipid bilayers, the images are very suitable for detailed analysis and can yield characteristic lipid features and membrane shapes. Currently, localisation of such membrane structures and the extraction of their features is often performed interactively and is, therefore, subjective and a time-consuming task. This is particularly true for larger data sets collected by automated electron microscopes because the number of available images is often too large to be analysed interactively. The here developed “membrane analysis toolkit” aims to automate the membrane analysis and facilitate quantitative comparison of different functional datasets, e.g., capturing the variations such as protein mutations and lipid compositions. The membrane structures are localized using convolutional neural networks and sequentially analysed by a variety of image processing techniques. Typical determined features are membrane thickness, curvature and distance to neighbouring structures along the membranes in addition to shape information about the continuous membrane structures like total area and shape assignments. The newly developed membrane analysis toolkit is available in a Napari-based GUI implementation and was applied to images of the bacterial membrane-remodeling protein IM30. By extracting lipid features from different IM30 datasets, different effects on lipid shapes could be quantitatively established. The membrane analysis toolkit can be used to any other protein lipid mixtures and can lead to a more quantitative understanding of the membrane-active proteins.

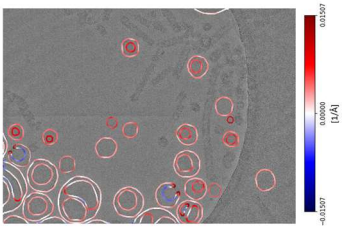

Fig. 1 Curvature of vesicles

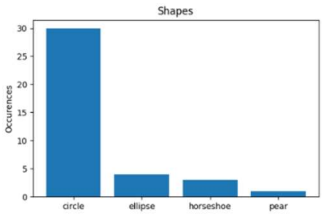

Fig. 2. Shape distribution

CRYO-EM STRUCTURE OF AβUPP(1-42)$_{\Delta 19\text{-}24}$ FIBRILS

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Today, 32 pathogenic mutations in the amyloid precursor protein (APP) gene are known. Most of them are point mutations that cause familial Alzheimer's disease (AD) by an increased production or a change in the conformation of amyloid-beta (Aβ). The Uppsala APP mutation (Δ690-695) is the first known multi-codon deletion causing autosomal dominant AD [1]. The mutation alters APP processing, leading to an increased production of the pathogenic AβUpp1-42$_{\Delta 19\text{-}24}$ peptide. Moreover, the amyloid fibril formation and their deposition into amyloid plaques in the brain is increased. A symptom onset in the early forties and a rapidly progressing disease course have been observed in affected individuals. Here, we applied cryo-EM (Fig.1) to characterize fibrils that were assembled in vitro from synthetic AβUpp1-42$_{\Delta 19\text{-}24}$. For the two main polymorphs, density maps at resolutions of 3.5 and 3.9Å, respectively, reveal two novel filament structures (Fig. 2). The first polymorph adopts an extended S-shaped fold with a large inter-protofilament interface, while the second polymorph shows a G-shape fold with a rather small interface involving only three amino acids. For both polymorphs, the C-terminal part forms a C-shaped fold, that was also observed in known structures of wildtype (WT) Aβ(1-42) fibrils [2,3]. However, the overall structures differ from observed WT Aβ(1-42) fibrils prepared under the same conditions [2], suggesting a possible link between the altered structure and the observed faster fibril formation.

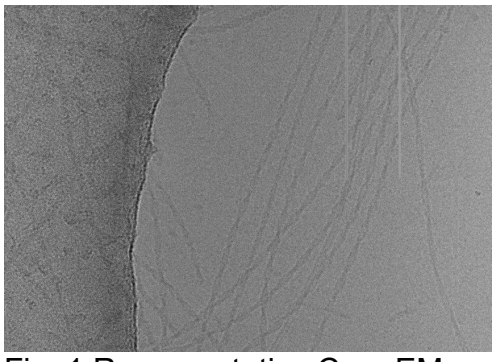

Fig. 1 Representative Cryo-EM micrograph

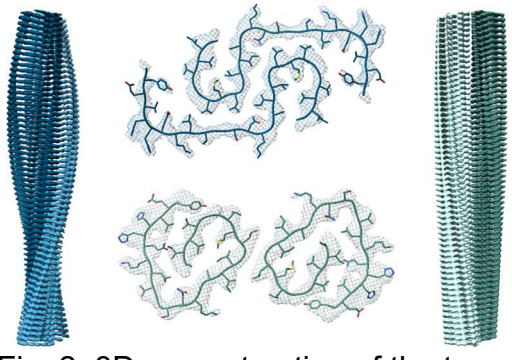

Fig. 2. 3D reconstruction of the two main Aβ Upp1-42$_{\Delta 19\text{-}24}$ polymorphs (PM1: blue, PM2: green) from cryo-EM images

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KEEP IT CLEAN: AN IMPROVED CRYO-FIB WORKFLOW

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Electron tomography at cryogenic temperatures (cryo-ET) offers the unique possibility to structurally analyse biological macromolecules in their native cellular environment at high resolution [1, 2, 3]. A prerequisite for such investigations is a sample thickness of approximately 200-300 nm. In the recent years, numerous studies demonstrated that cryo focused ion beam milling is a suitable technique for preparing biological sample for cryo-ET [4]. Current best-practice protocols for cryo-FIB milling include: i) vitrification of the sample, ii) identification of regions-of-interest, iii) rough ablation of the surrounding sample material and iv) polishing of the target region, leaving a thin lamella ready for cryo-ET. The entire procedure involves multiple handling, transfer, milling and imaging steps. During these steps, samples need to be handled with forceps and it is difficult even for highly experienced practitioners not to destroy the thin and fragile lamellae. Moreover, since the vitrified sample must be constantly cooled to avoid devitrification, it is particularly prone to any kind of ice contamination. Here, we present an integrated workflow (Fig. 1) that addresses and overcomes the aforementioned bottlenecks, thereby increasing the overall throughput [5].

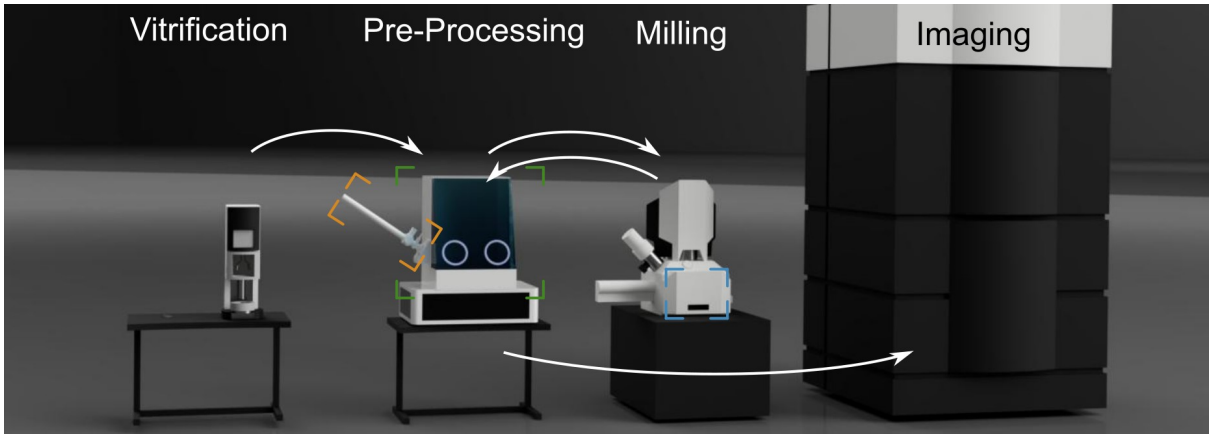

Fig. 1: Streamlined workflow to optimize grid handling and minimize ice contamination during sample preparation. Three main hardware extensions: cryo high-vacuum transfer (orange), glove box (green) and cryo-shutter (blue).

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HYBRID-PIXEL DETECTORS FOR TEM BY DECTRIS

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25 years of advances in aberration correction have clearly shifted the TEM characterization limits from the electron optics to other factors, among them the electron detectors. Recent improvements on electron detection technology clearly impact the TEM characterization on both Materials Sciences and Life Sciences, particularly when beam-sensitive samples are involved [1]. Hybrid-pixel detector (HPD) [2] is one of the approaches to directly detect and count electrons, with the distinctive advantage of flexible design with respect to the sensor material and electronics for optimization to different applications. DECTRIS is a pioneer in the development of HPD for X-rays, which became a reference for protein structure determination to the synchrotron community. Recently DECTRIS optimized its HPD design to enable the most precise detection of electrons. The QUADRO, ELA, and SINGLA detectors feature optimal threshold values for counting electrons with zero read-out noise within a wide energy range, and proprietary retrigger technology that allows counting to 10^7 counts/pixel/second [3]. These features make DECTRIS HPDs adapted to a variety of TEM applications, including low-dose imaging with single-electron sensitivity, 4D-STEM acquisition with 32-bit dynamic range and high frame rate (2,250 fps with full read-out and 18,000 fps with ROI), and unmatched EELS performance – from zero-loss peak (ZLP) to zero-noise core-loss (CL) region included in the same acquisition range (Figures 1 and 2 – adapted from reference [3]).

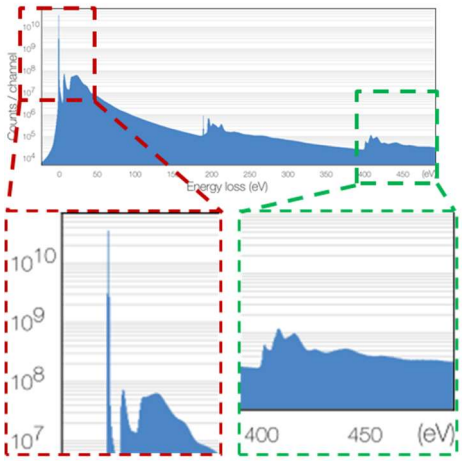

Fig. 1. EELS spectrum of h-BN at 60kV. Simultaneous ZLP and CL collection without saturation across 6 orders of magnitude.

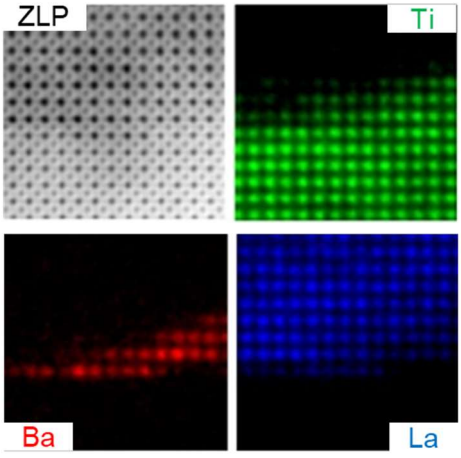

Fig. 2. Spectrum imaging of STO/BTO/LSMO. Flexible elemental mapping with multipass EELS allowed by zero-readout noise.

References:
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PULSED LASER INTEGRATION AND ELECTROSTRATIC SYSTEMS FOR DODE MODULATION AND TEMPORAL RESOLUTION TEM

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In situ TEM is a powerful tool for revealing fundamental nanoscale processes in physics, biology, and materials science. This talk will explore the possibilities for studying laser-driven processes with the help of electrostatic deflection systems that can switch states on the nanosecond scale. In addition, we will highlight new developments involving electrostatic beam blankers, programmable high-speed timing systems, and advanced automation and data analysis. A recently developed laser module, “Luminary Micro” (JEOL/IDES), facilitates direct focused laser-sample interaction. This has previously successfully been used to show dewetting of gold on a surface, sintering of nanoparticles and more [1]. Two electrostatic optical systems have been developed that can be precisely synchronized with the laser. [2] The first of these, a pre specimen electrostatic dose modulator (EDM), works with a timing control system, “Synchrony” (JEOL/IDES), to enable nanosecond precision definition of electron beam temporal profiles. The second, a post specimen camera subframing system, “Relativity”, (JEOL/IDES) has previously been shown to provide kHz scale fame rates with similarly precise timing control [2]. Together the systems described here, a compact sample laser, a pre-specimen EDM, a post specimen camera subframing deflector, and a precision timing controller enable users to craft scriptable experiment definitions to control all timing aspects of a laser driven in situ experiment. From dose rate to heating profiles to camera frame rate, users can decide how their experimental conditions will evolve with nanosecond precision.

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CLIMATE - EXPOSE YOUR SAMPLE TO A CONTROLLED GAS ENVIRONMENT WITH PRESSURES UP TO 2 BAR AND SWITCH GASSES IN SECONDS

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Conventionally, Transmission Electron Microscopy (TEM) studies are limited to work under static conditions by investigating nanomaterials supported on a static grid under high vacuum conditions. With the introduction of environmental TEMs (ETEMs), nanoscale investigations within gas environments became possible, but limited to very low gas pressures. With the use of Micro Electro-Mechanical System (MEMS)-based sample carriers, in situ gaseous TEM experiments at high pressures became feasible, opening up a wide variety of opportunities in fields like catalysis, nanomaterials growth and corrosion studies.

Here, we present our in situ TEM gas and heating system, referred to as the “Climate” System. The Climate System converts high-vacuum (S)TEMs from a static imaging tool into a dynamic in situ chemical laboratory, enabling real-time observation and analysis of nanomaterials. We will discuss the different main components of the Climate System and highlight their key features and benefits. Among these components, the Nano-Reactor (Figure 1) is our MEMS-based sample carrier that allows a defined amount of gas to be flowed through the Nano-Reactor chamber where the sample temperature is controlled by an integrated microheater.[1] The Nano-Reactor is very unique as it has been heavily optimized to enable real nano-calorimetry.[2] Next to the advanced Nano-Reactor, the Gas Supply System (GSS) is a key component of the Climate System (Figure 2). The Climate GSS enables the possibility of performing live gas mixing, i.e. allowing you to achieve any desired gas composition instantaneously. This unique feature enables you to safely work with explosive mixtures, even at extreme conditions like high temperatures and pressures.

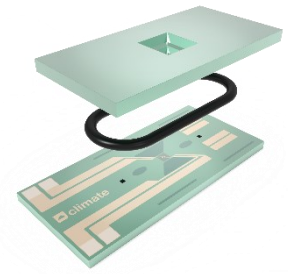

Fig. 1. DENSsolutions Climate Nano-Reactor. Fig. 2. DENSsolutions Climate GSS

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MERLIN EM, HYBRID PIXEL ARRAY COUNTING DETECTOR FOR TRANSMISSION ELECTRON MICROSCOPY

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At the University of Glasgow, experiments with hybrid pixel array counting detectors started with a Medipix2 detector [1]. It was clear that direct electron detection and hardware-based electron counting can offer advantageous imaging capabilities. Subsequently, a Medipix3 detector with a Merlin readout system was commercialised as a MerlinEM detector by a collaboration between the University of Glasgow and Quantum Detectors Ltd. With more than 50 systems around the world, the detector has been applied in multiple experimental configurations.

The detector has been mainly applied in scanning transmission electron microscopy (STEM), however, diffraction and EELS applications are also wide-ranging. [2]

In STEM, the ability to collect a full distribution of electrons for each probe position with a millisecond and better timescales can be readily used in DPC [3] and ptychography [4] but it also enables a more quantitative approach to standard techniques and potential for development of new ones.

In diffraction, MerlinEM can be used to image direct probes while still retaining the ability to count single electrons for the weaker diffraction spots. This makes the detector an attractive solution for low dose techniques in the microscopy of bio-molecules, microED and scanning precession techniques.

In EELS, the 4x1 MerlinEELS detector version, offers low noise, large dynamic range and fast framing. These characteristics are can be used to push core-loss imaging to high kV losses [5] and allow live drift corrections.

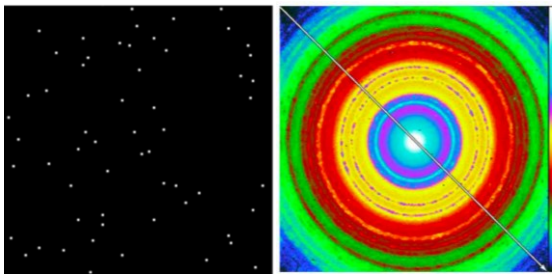

Fig. 1. Single-electron sensitivity and full diffraction pattern images collected with the same MerlinEM settings (albeit dwell time). Obtained under CC BY 4.0 from [2].

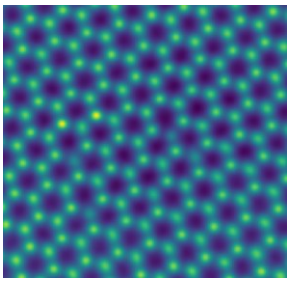

Fig. 2. Ptychography with MerlinEM. Image of single gold atoms in $MoSe_2$. Image courtesy Dr Shoucong Ning, NUS Singapore.

References:

[1] Mac Raighne, A., et al., Journal of Instrumentation 6.01 (2011): C01047.
[2] Mir, J. A., et al. Ultramicroscopy 182 (2017): 44-53.
[3] Krajnak, Matus, et al., Ultramicroscopy 165 (2016): 42-50.
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DYNAMICAL STUDIES OF ADVANCED MATERIALS COMBINING *IN SITU* 4D-sPED MAPPING AND PIXELATED DETECTORS

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Automatic crystal orientation & phase mapping (ASTAR technique) can be applied in a variety of materials, is known also as 4D-STEM technique [1, 2]. We apply a dedicated acquisition process (named as 4D-SPED "scanning Precession Electron Diffraction") where beam precession (to improve ED patterns quality [3]) in TEM is used in combination with beam scanning. ASTAR technique is very similar to the traditional EBSD-SEM method, where the major advantage is the significant improved spatial resolution that can be down to 1-3 nm (in case of FEG-TEM). In the standard configuration, a special CCD camera is used (able to collect a 100 fps) with dedicated scanning 4D-SPED DigiSTAR hardware. Local crystallographic orientation/phases are identified through an original algorithm that compares experimental ED spot patterns with pre-calculated (simulated) templates for all possible orientations and/or phases [4]. We have studied many advanced materials using 4D –SPED ASTAR, to investigate local properties and/or study of nanosized samples. In addition, *in situ* methods have been extensively applied towards dynamical studies. The combination of the 4D-SPED ASTAR with the new generation of pixelated detectors, allows the use of extremely low electron dose (up to 0.001 e/Å2/sec) with significantly fast acquisition times (500-1000fps). The use of pixelated detectors has critical advantages over conventional CCD, due to the absence of electronic detection noise (allowing to work at extremely low dose). Combining pixelated detectors (like QD & ASI Medipix,Dectris,PnCCD etc…) with NanoMEGAS 4D-SPED ASTAR system, not only acquisition time is greatly reduced, but more critically, data quality is enhanced, allowing in situ dynamical studies by sequential mapping even for beam sensitive samples.

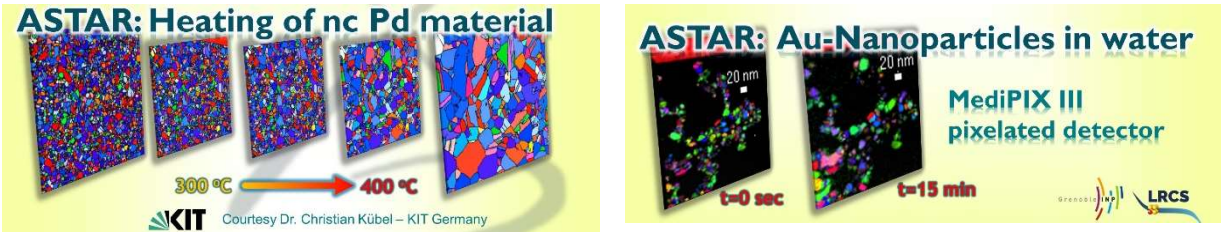

Fig. 1 *In situ* ASTAR applications using pixelated detector

References:

[1] E.F. Rauch *et al*, Zeitschrift für Kristallographie **225** 103- (2010),.
[2] J. Portillo, E. F. Rauch, S. Nicolopoulos, M. Gemmi, and D. Bultreys. *Materials Science Forum*, **644**, 1–7 (2010)
[3] R. Vincent and P. Midgley, Ultramicroscopy **53**, 271- (1994)
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AXON DOSE: A MACHINE VISION SOLUTION FOR ACCURATE, QUANTIFIABLE DOSE MANAGEMENT IN THE TRANSMISSION ELECTRON MICROSCOPE

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Until now, a robust solution for managing the electron dose during TEM experiments, one that enables an operator to track and manage beam effects, has been hampered by several key issues. First, the calibration a TEM's beam current can be a tedious and difficult process that requires many steps to accurately account for all the variables [1]. Furthermore, once calibrated, a typical workflow requires the operator to either remain at a specific magnification or work within other severe limitations to keep dose rates constant and simplify dose calculations. Thus, freely operating the instrument through various spot sizes or magnifications while keeping track of the total accumulated dose and/or dose rate over the sample area isn't practical without some form of assisting technology. Here, we review the development and utilization of AXON Dose, a machine-vision solution designed to address and eliminate the specific pain points with calibrating, managing, and tracking a sample's electron dose exposure throughout the course of a TEM experiment. To achieve this goal, a platform was required that could account for both dose and dose rate on a per-pixel basis while compensating for sample drift and mechanical slop. Integration of that dose information into the image metadata enables robust analysis and visualization through heat maps from which accurate, quantifiable information on cumulative dose and fluence can be obtained from any point on the experimental timeline (Figure 1). Experimental methodology and scientific examples of dose analysis will be presented.

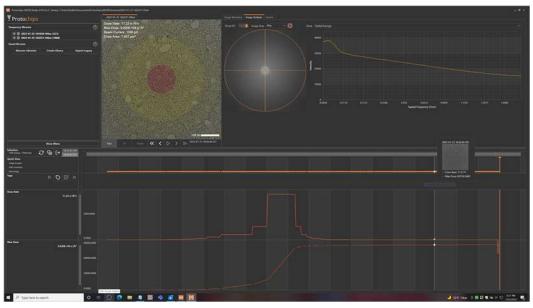

Fig. 1 *The AXON Dose User Interface*

References:

[1] Żak, A. Guide to Controlling the Electron Dose to Improve Low-Dose Imaging of Sensitive Samples. *Micron* **2021**, *145*, 103058.

IMPROVING SAMPLE DISPERSION WITH AN ADDITIONAL ULTRATHIN CARBON LAYER

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Sample preparation is a major roadblock in the cryo-EM workflow; identifying conditions in which the sample is well-distributed across the holes in random orientations can often seem like a black art to the novice cryo-electron microscopist. Grid choice and grid surface modifications can make a big difference to sample quality; but identifying the best support for a new project can be challenging. One additional option which can often improve particle distribution is an additional ultrathin carbon (UTC) layer [1], and Quantifoil offers additional UTC layers in a range of thicknesses for most holey supports. The layer acts in several ways to improve particle distribution:

- Increasing the number of particles due to adsorption of biomolecules onto carbon prior to blotting.
- Improving particle distribution due to interaction with a continuous carbon surface across the hole as well as the support.
- Reducing the number of particles adopting a preferred orientation.

Ultrathin carbon layers have been used with a wide range of biomolecules including: SARS-CoV2 proteins [2, 3] and membrane-embedded complexes, such as *Helicobacter pylori* outer membrane complex (OMC) [4] as well as a wide range of other biological macromolecules.

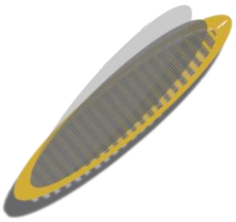

Fig. 1 Sample support with extra layer of ultrathin carbon

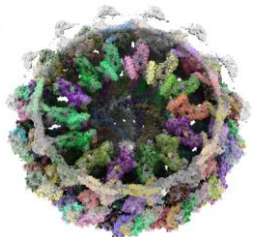

Fig. 2. *Helicobacter pylori* OMC from data collected on a sample support with a UTC [4]

References:
[1] Drulyte, I et al. Acta Cryst D**74**: 560-571 (2018).
[2] Thomes, M et al. Science **369**: 1249-1255 (2020)
[3] Schubert, K et al. Nat Struct Mol Biol **27**: 955-966 (2020).
[4] Sheedlo, MJ et al. eLife **9**: e59495 (2020).

DEDICATED WORKFLOWS FOR ATOMIC STEM BASED ANALYSIS ON LITHIUM BASED BATTERY MATERIALS

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In the past decade, the rapid growth of electric vehicles and consumer electronics market leads Li-ion batteries to attract significant attention. In order to further advance their performance for higher energy and better safety, fundamental understanding of battery materials structures and chemistry is essential. Nowadays, in order to pursue higher battery performance, more and more materials used in battery are beam sensitive and inherently air sensitive. This brings challenges to preserve their native structure and properties via routine electron microscope practices. Low-dose EELS STEM analysis carried out at cryogenic temperature on a Krios (S)TEM under cryogenic conditions has proven to be a very successful approach to study the solid electrolyte interface (SEI) in the Li metal anode region. The main challenges, that until recently have limited the accurate investigation of SEI samples, are the extreme sensitivity to air and probing sources such as the electron beam. However, by combining the cryo transfer approaches developed for life science tools and the extra stability of the dedicated cryo-stage in the Krios, it was possible to successfully carry out, morphological, chemical and tomography studies of the solid electrolyte interface region in Li-anodes [1]. The Liquid Nitrogen used during the transfer of the sample into the microscope acts as protection against exposure to air and the extra stability of the cryo-stage in the Krios ensures successful high resolution data collection that otherwise would be greatly limited by the large drift typical of side-entry cryo holders. This paper will discuss the applications and challenges of cryo-EM in Li-ion battery research in general.

References:

[1] B. Han, X. Li, S. Bai, Y. Zhou, B. Lu, M. Zhang, Z. Ma, Z. Chang, Y. S. Meng and M. Gu, Matter **4**, 1-12 (2021).

QUANTITATIVE MAPPING OF LITHIUM IN THE SEM USING COMPOSITION BY DIFFERENCE METHOD

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Developments in Lithium-ion batteries and lightweight structural lithium alloys has been remarkable given the lack of a method to determine lithium content at the microscale. Energy dispersive X-ray spectroscopy (EDS) in the scanning electron microscope (SEM) is commonly employed for microanalysis, however, this is not suitable for elements with atomic number (Z) < 4 as the characteristic X-rays are easily attenuated by the sample or contamination and require specialized detectors. Even so, a limit of detection of ~20 wt. % Li and inability to perform quantitative analysis are significant issues [1]. However, quantification of Li in the SEM was demonstrated recently using a composition by difference method based on EDS and quantitative backscattered electron imaging (qBEI) [2]. EDS was used to quantify elements Z = 4 – 94, whilst qBEI was used to determine the mean atomic mass (the qBEI signal being a function of atomic number for Z = 1 – 94). The fraction of light elements (Z = 1 – 3) was calculated enabling detection of <5 wt. % Li in a MgLi alloy. We extend the composition by difference method to generate quantitative, spatially resolved elemental maps of a Mg-Li-Al alloy. The sample was prepared by argon milling using an Ilion™ polisher (Gatan Inc.). EDS and qBEI maps were recorded at 3 and 5 kV, respectively, determined prior to analysis by Monte Carlo modelling to achieve equal sampling volumes. Quantified EDS maps were captured using an Octane Elite EDS System and APEX software (EDAX LLC) and qBEI was performed using an OnPoint™ backscattered electron detector (Gatan Inc.). Image analysis was performed using DigitalMicrograph® software (Gatan Inc.). The results demonstrate that single-digit mass percentages of Li can be mapped quantitatively in the SEM using the composition by difference method. Limitations are known to include surface topography as well as by the presence of unknown quantities of H, He or voids. Nevertheless, the method offers distinct advantages compared specialized “Li” EDS detectors.

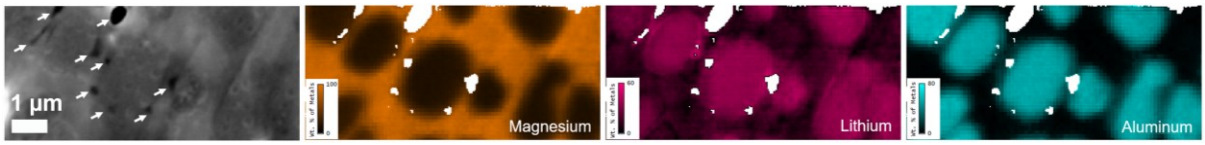

Fig. 1. Secondary electron image (left) and elemental metal fraction maps; white pixels excluded from the analysis due to topographic influence (arrowed in the secondary electron image).

References:
[1] P. Hovington *et al.*, Scanning **38** (2016) p571–578
[2] JA. Österreicher *et al.*, Scripta Materialia **194** (2021) 113664

LIQUID HELIUM CRYO-TEM

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The company condenZero develops and builds cryo-TEM solutions at liquid helium temperatures. The cryo-TEM sample holders feature heating and electrical biasing capabilities and provide temperature stability over long periods of time. For more information, please visit condenzero.com.

Fig. 1 Liquid helium sample holder

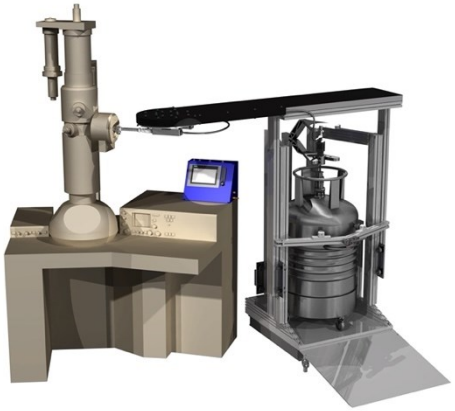

Fig. 2. Setup of the condenZero system.

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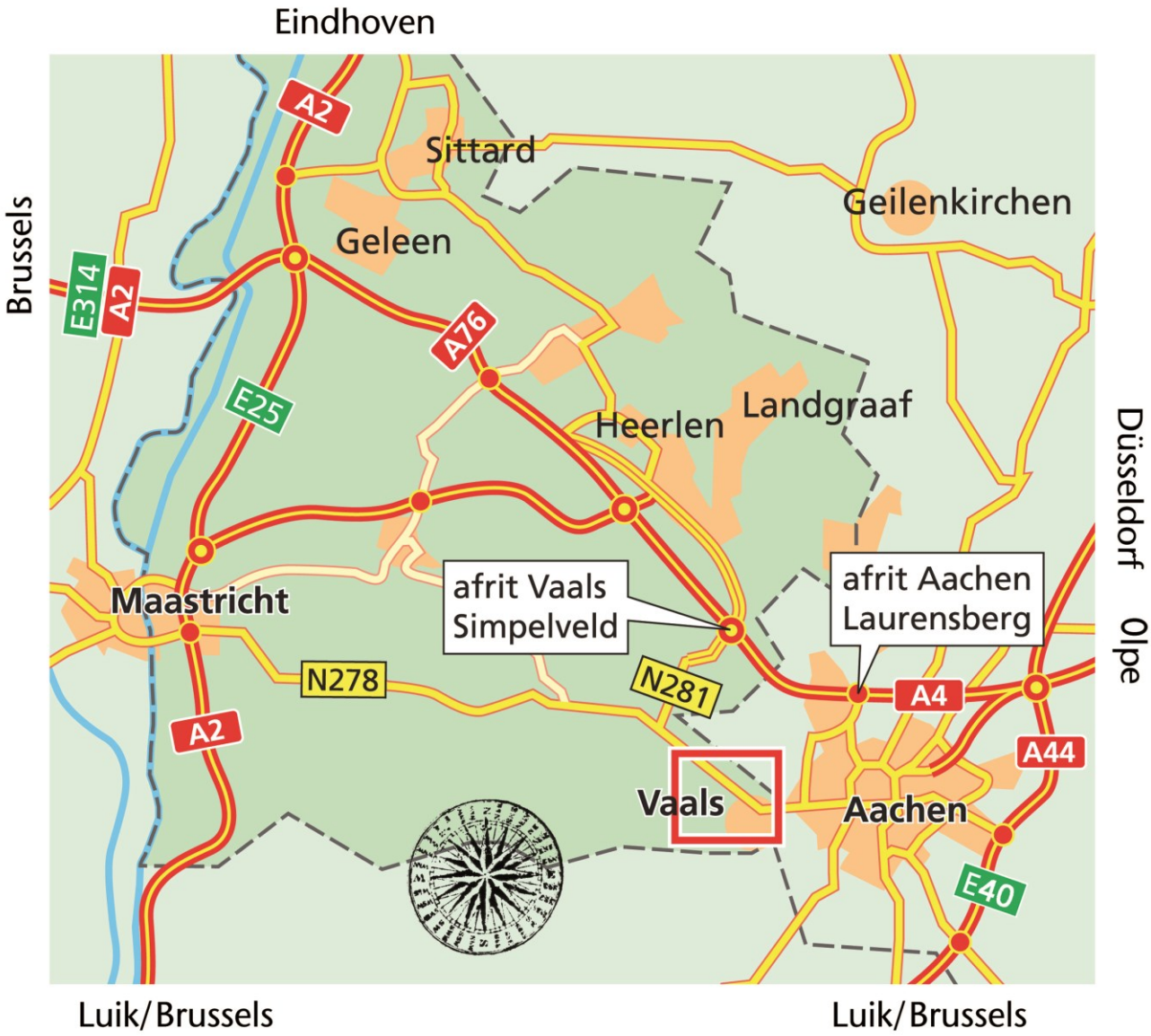
ROAD MAPS & FLOOR PLANS

EMS Extension

ROAD MAP – REGIONAL



EMS Extension

ROAD MAP – LOCAL

Bilderberg Kasteel Vaalsbroek

Vaalsbroek 1

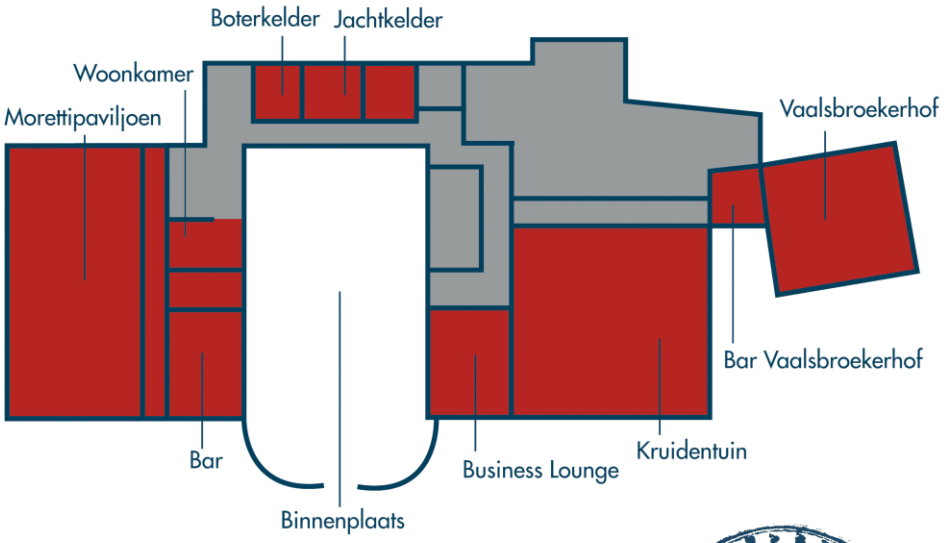
6291 NH Vaals

The Netherlands

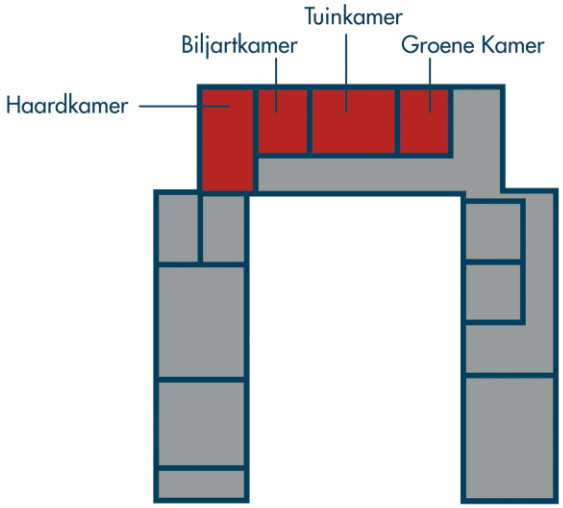
Phone +31 43 308 93 08

FLOOR PLAN – CASTLE

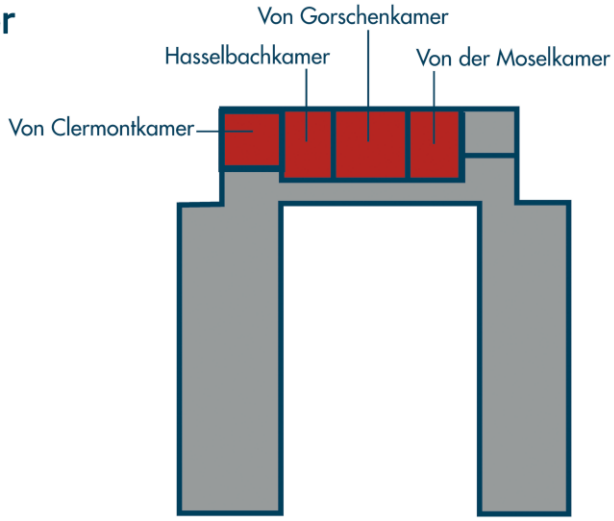
ground floor



first floor



second floor



hotel

EMS Extension