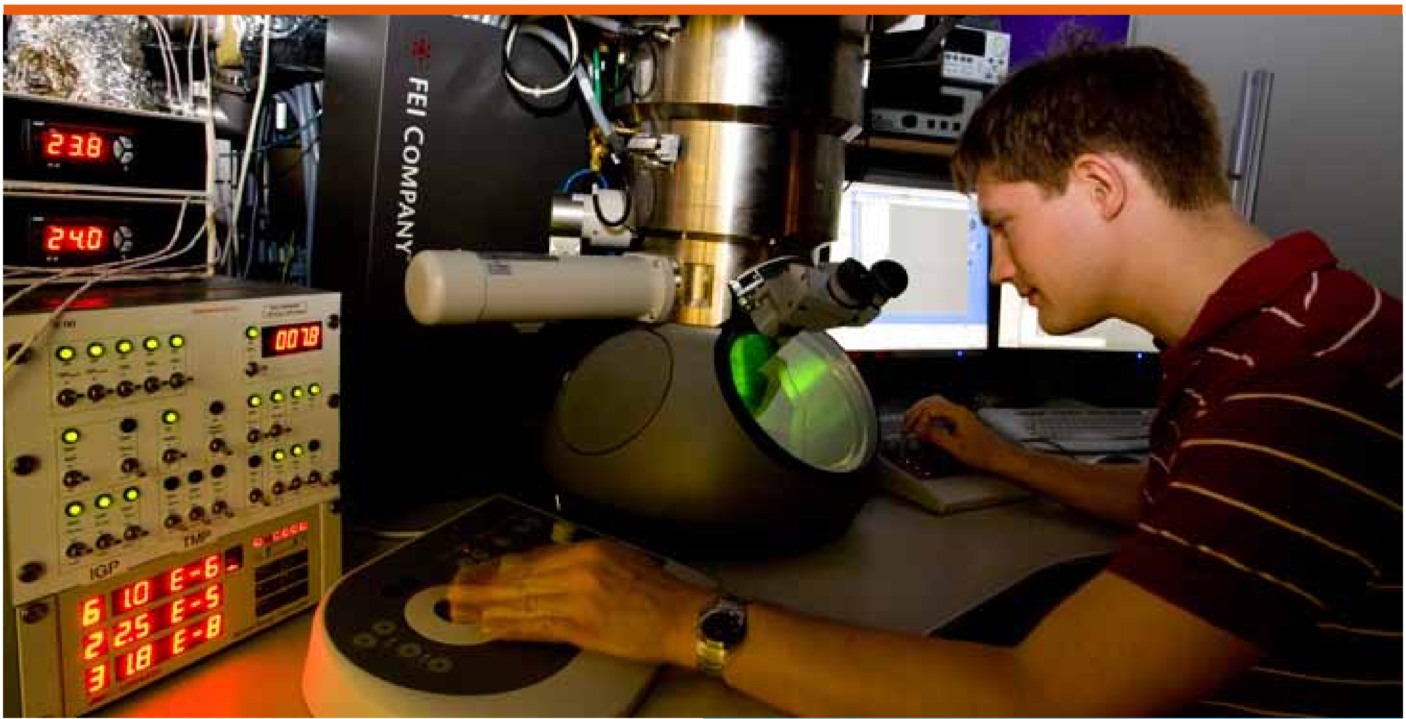
Annual Report



2009

DTU Cen
Center for Electron Nanoscopy

Center for Electron Nanoscopy (Building 314) at DTU.

Preface

Note From the Director
This is the second annual report from the Center for Electron Nanoscopy in the Technical University of Denmark (DTU Cen), which was established in December 2007 following a generous donation of 102,6M DKr by the A.P. Møller and Chastine Mc-Kinney Møller Foundation.

The facility contains seven new electron microscopes in a purpose-built high-specification building. Two of the microscopes are state-of-the-art monochromated aberration-corrected instruments. The remaining instruments include two dual-beam focused-ion-beam microscopes and three smaller microscopes.

Two years after its inauguration, DTU Cen is an active, operational unit at DTU, in which a team of core staff

- perform research in collaboration with scientists from DTU and external partners
- assist and train users of the electron microscopes
- supervise students and scientists
- deliver lecture courses and organize workshops
- secure external grant funding
- host national and international visitors.

Close working relationships with other departments at DTU and with national and international partners have been established and a program for training new users is in place.

Research topics center on studies of nanoscale materials at the highest spatial resolution and in three dimensions using techniques that include aberration-corrected electron microscopy, electron tomography, environmental (gas reaction) electron microscopy, electron holography, FIB SEM slice and view imaging and 3D electron backscatter diffraction.

I am very grateful to DTU Cen's staff, colleagues and equipment manufacturers for creating a successful and dynamic facility in such a short time, as well as to DTU's management and the A.P. Møller Foundation for their foresight and hard work in the establishment of the Center.

Enquiries about both scientific collaborations and commercial partnerships are warmly encouraged.

June 2010
Rafal E. Dunin-Borkowski
Center Director

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DTU Cen, Center for Electron Nanoscopy, 2010 · Editor, Anna Rink Baagøe · Scientific Editor, Shima Kadkhodazadeh
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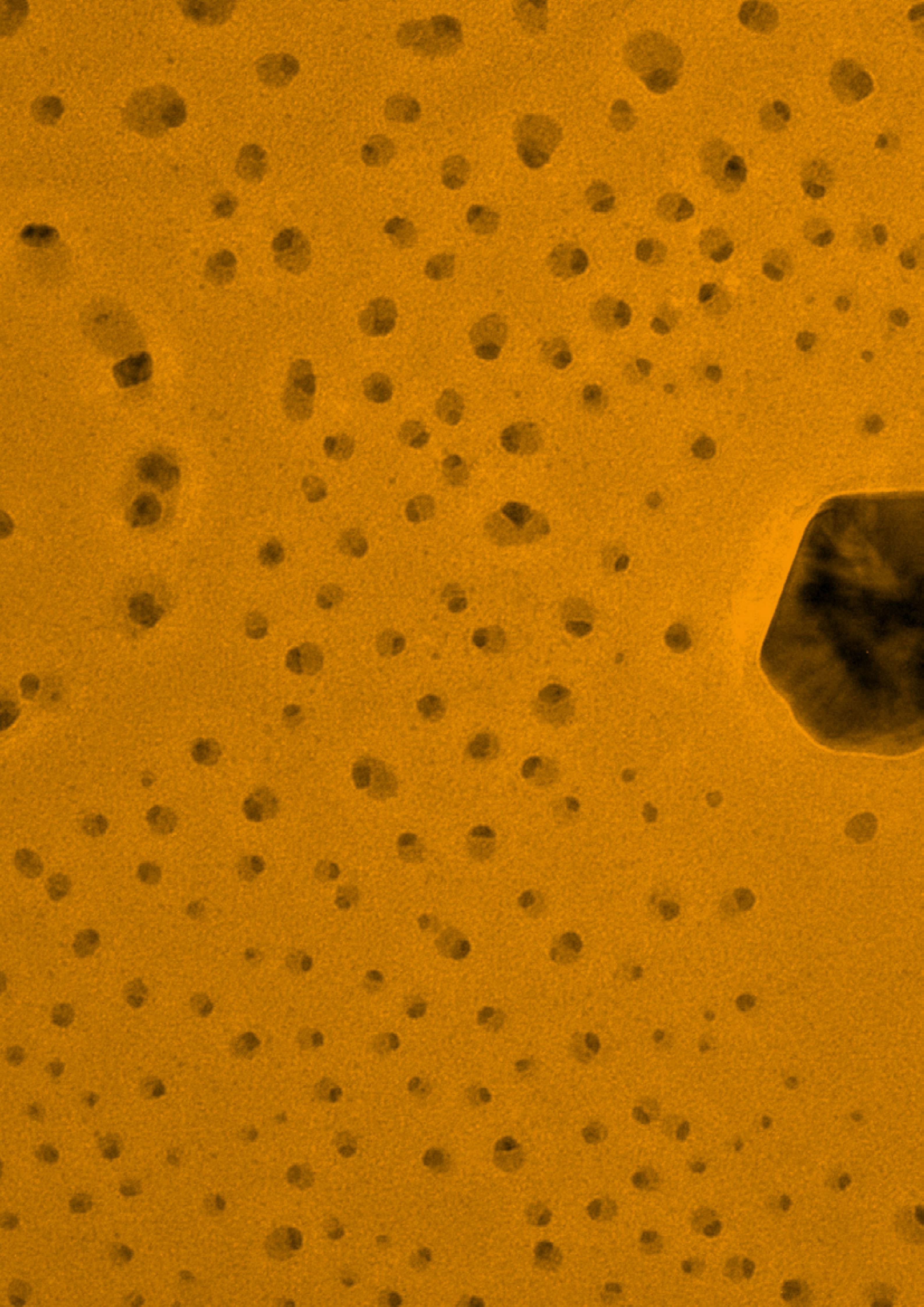


Executive Summary

In the second year after its inauguration, the Center for Electron Nanoscopy in the Technical University of Denmark has grown to become a fully operational unit at DTU in the areas of research, teaching, service and outreach.

The hiring of a core team of experienced scientific and technical support staff has been completed. The Center's seven new electron microscopes are in daily use, while the microscope building provides an excellent environment for research and teaching.

By working together with other departments at DTU and with other universities and industry, both in Denmark and abroad, the Center has established itself rapidly as a state-of-the-art electron microscopy facility.

The rapid development of the Center's national and international profile is reflected by:

- the collaborative research and service activities that take place with academic and industrial partners
- the research articles that are published in scientific and popular journals
- the frequent invitations to speak at international conferences that are received by the Center's staff
- the frequent visits to the Center by visiting scientists and industrialists
- the additional European and national grant funding that has been secured for specific projects.

This report contains descriptions of the Center's seven electron microscopes, examples of recent research and teaching activities and a summary of events, seminars and research output for 2009. Profiles of the Center's research scientists and technical and administrative staff are also presented.

We hope that this report is useful and informative.

Microscopes

Inspect 'S'

What is it?
The Inspect 'S' is a scanning electron microscope (SEM). It is the simplest microscope at DTU Cen and is the first microscope to which new SEM users are introduced. The simplicity of its operation allows quick preliminary inspection of samples.

Main features
- tungsten filament electron source capable of imaging samples in high vacuum, low vacuum (up to 200 Pa) and environmental (up to 4000 Pa) modes
- equipped with a secondary electron detector and X-ray detectors

What can it do?
- secondary electron imaging giving topographical images of samples
- X-ray analysis using both energy and wavelength dispersive spectrometers

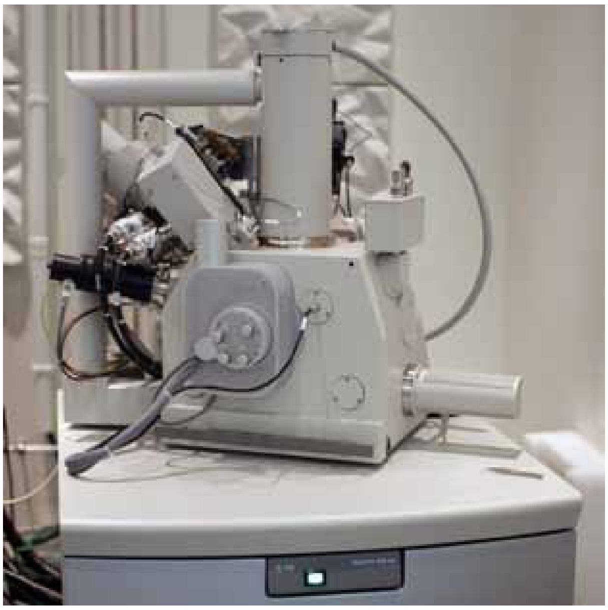
Quanta 200 F

What is it?
The Quanta 200F is a scanning electron microscope fitted with a field emission gun (FEG) electron source to provide high imaging resolution. The Quanta 200F can operate in high-vacuum, low-vacuum and environmental modes, allowing samples to be examined under high vacuum or in gas or water vapour conditions.

Main features
- high spatial resolution (~ 2 nm)
- energy dispersive x-ray analysis
- Cryo-SEM: The Quanta 200 F is fitted with a cryo transfer system, giving the possibility of freezing liquids, particles in liquids or delicate and beam sensitive samples. Frozen samples can be fractured, cut and/or coated and transferred to a cold stage in the microscope where all imaging and analysis techniques can be performed.

What can it do?
- high resolution secondary or backscattered electron imaging
- the three vacuum modes mentioned above give the possibility of examining samples under high vacuum or in gas or water vapour conditions
- chemical analysis using the X-ray detector

Quanta 200 3D



What is it?
The Quanta 3D is a dual-beam scanning electron microscope and focused ion beam (FIB) instrument. The focused ion beam (typically Ga^+ ions) is used to remove material from a bulk sample and the electron beam is used for imaging. The electron source is a tungsten filament. The Quanta 200 3D is equipped with a long, fine tungsten needle, for in-situ micro manipulation of samples.

Main features
- tungsten filament electron source: imaging resolution ~ 50 nm
- FIB milling at 30 kV, 15 kV, 5 kV and 500 V beam energies
- Dual-beam (electron and ion beams) imaging
- secondary electron detector, backscattered electron detector and X-ray detector
- tungsten and platinum deposition from organic precursor gases to provide extra strength to thin specimens, create a milling mask or weld materials together
- in-situ manipulation of specimens using the manipulator

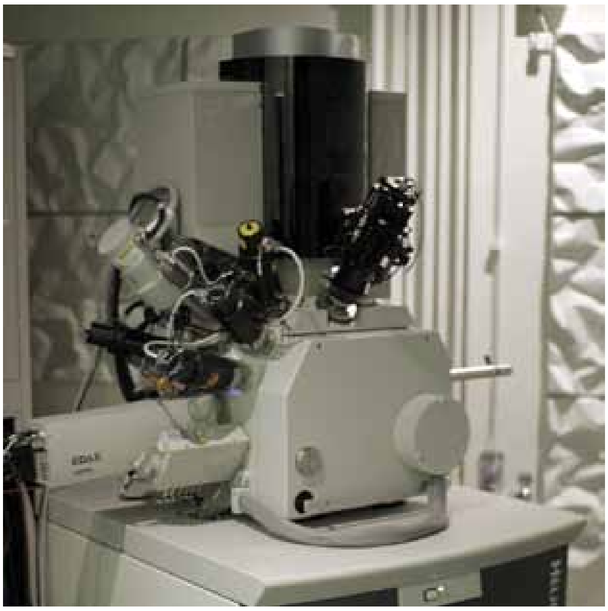
What can it do?
- FIB milling can be used to reveal structure under top layers, cut cross-sections for SEM, prepare site-specific specimens for transmission electron microscopy (TEM) or modify microstructures
- the dual-beam system makes it possible to switch between the ion and electron beam for accurate milling while reducing sample erosion and ion implantation
- three dimensional material imaging and characterization are possible using combined high precision FIB milling and SEM imaging

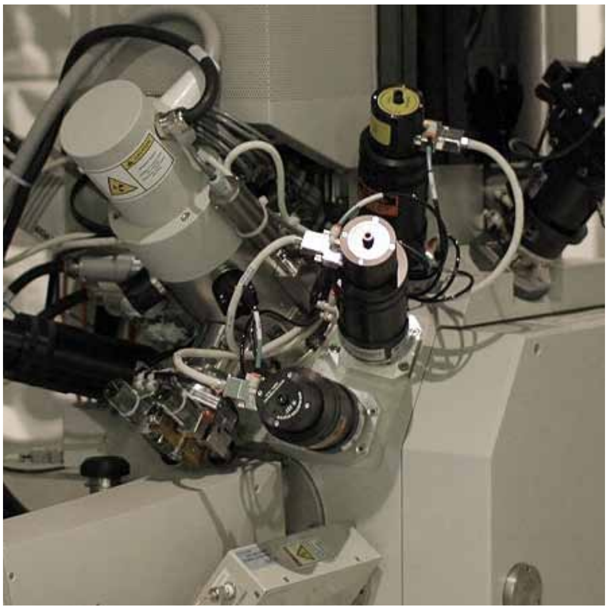
Helios NanoLab600

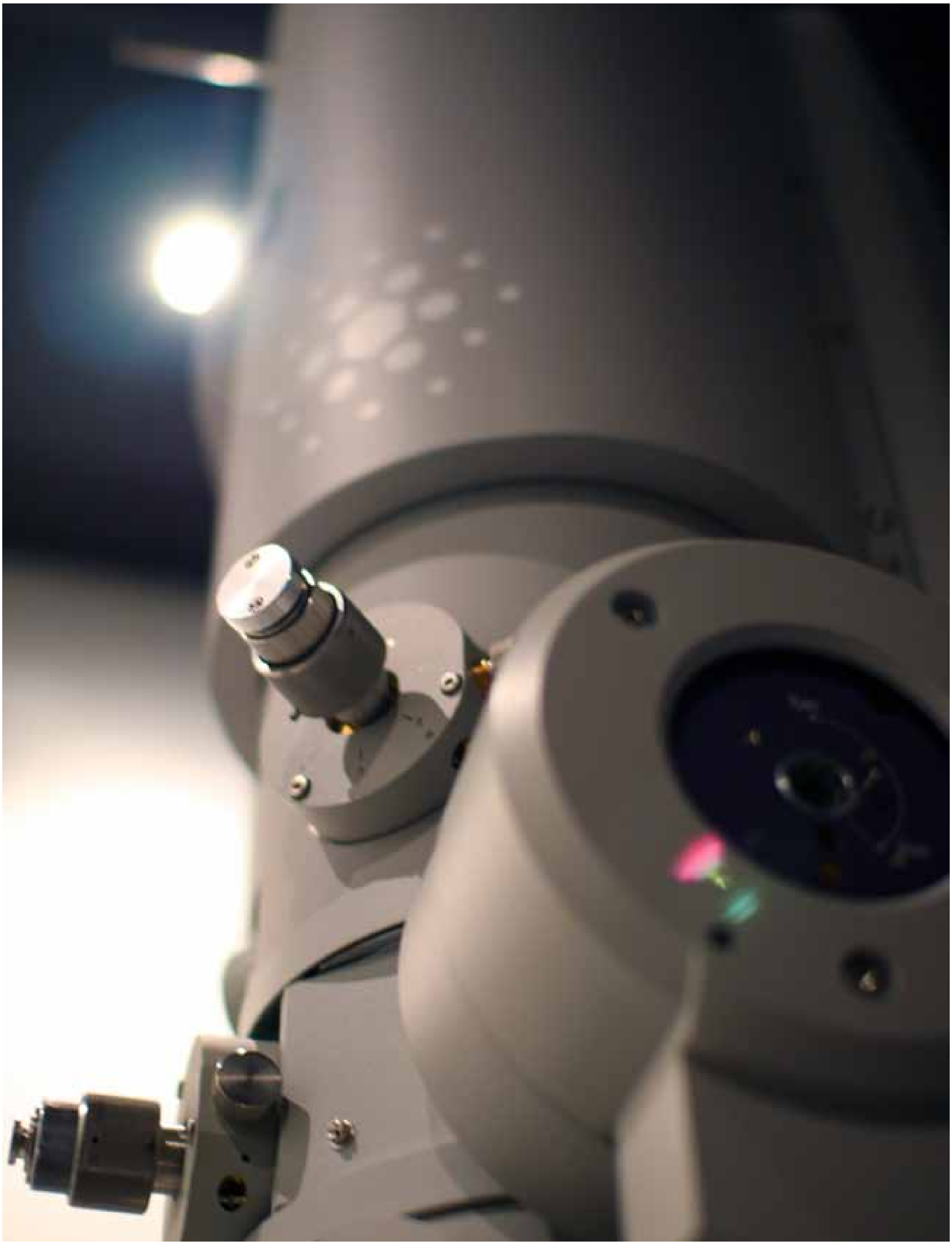
What is it?
The Helios is a dual beam scanning electron microscope and focused ion beam instrument. The Helios is fitted with a FEG electron source, giving this instrument improved resolution compared to the Quanta 200 3D. Furthermore, the Helios NanoLab has ultra-high-resolution optics, providing SEM resolution in the sub-nanometer range.

Main features
- FEG electron source giving high resolution SEM imaging with sub-nanometer resolution at 15 kV and better than 1.5 nm at 1 kV.
- FIB milling at 30kV, 15kV, 5kV and 500V energies.
- electron backscatter detector (HKL EBSD system) and energy dispersive X-ray.
- tungsten and platinum deposition from organic precursor gases to provide extra strength to thin specimens, create a milling mask or weld materials together.

What can it do?
- Dual beam (electron and FIB) imaging
- TEM specimen preparation
- three dimensional material characterization using FIB milling and SEM imaging
- crystallographic characterization by electron backscatter diffraction (EBSD) in two and three dimensions
- chemical characterization by energy-dispersive X-ray spectroscopy in two and three dimensions
- advanced integrated solutions for nanoprototyping.
- Nano-writing and machining







Tecnai

What is it?
The Tecnai is a transmission electron microscope (TEM) fitted with a LaB_6 filament electron source. Techniques such as bright-field, dark-field and high resolution TEM imaging as well as energy dispersive X-ray and electron energy-loss spectroscopy can be carried out on this instrument, allowing material characterization with nanometer resolution.

Main features

- fitted with a LaB_6 filament electron source. The instrument can be operated with an electron beam accelerating voltage in the range 80 – 200 kV.
- TEM imaging point resolution of 0.24 nm and line resolution of 0.15 nm (Supertwin objective lens, CS = 1.2 mm).
- fitted with a 2K CCD camera
- fitted with an EDX detector (Oxford Instruments INCA) and electron energy-loss spectroscopy (GIF Tridiem), allowing chemical analysis of samples.

What can it do?

- imaging all types of organic and inorganic materials TEM images can be used to measure sizes, shapes, and distances in samples.
- diffraction patterns can be used to assess structures and orientations of the crystalline areas present in samples.
- images of interfaces or surfaces can be obtained at atomic-resolution and studied for the presence of defects.
- analysis can be performed to determine the chemical composition of samples as well as their spatial distributions and relative amounts.

Titan Analytical

What is it?

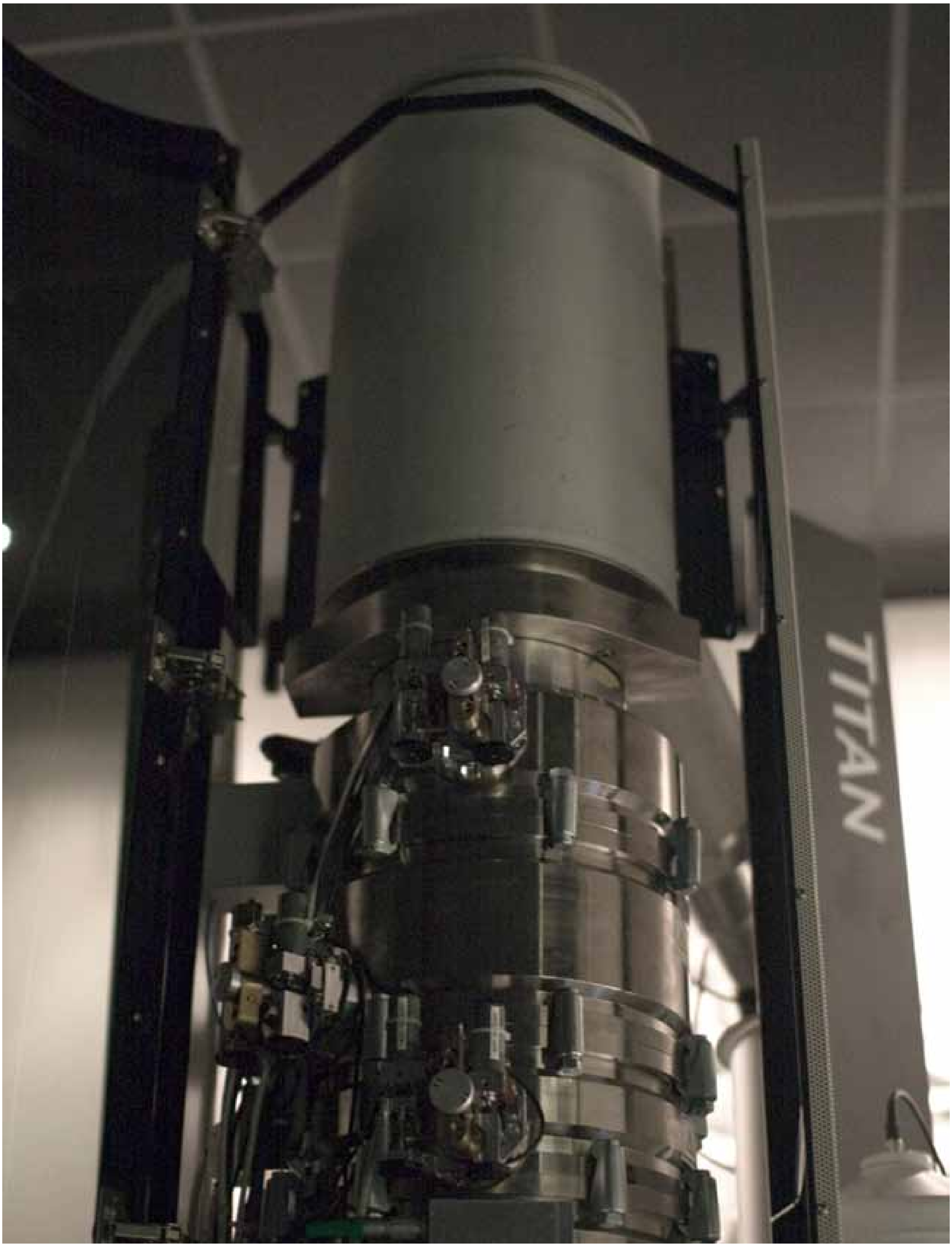
The Titan Analytical is a 120-300 kV transmission electron microscope with a field-emission gun (FEG) electron source. It has a probe C_S-corrector for atomic resolution scanning transmission imaging (STEM), a monochromator, an energy filter, a Lorentz lens, a biprism, an EDX detector and electron tomography capabilities. It is especially suited to studying the structure and chemistry of materials with the highest spatial resolution, as well as electromagnetic properties of materials.

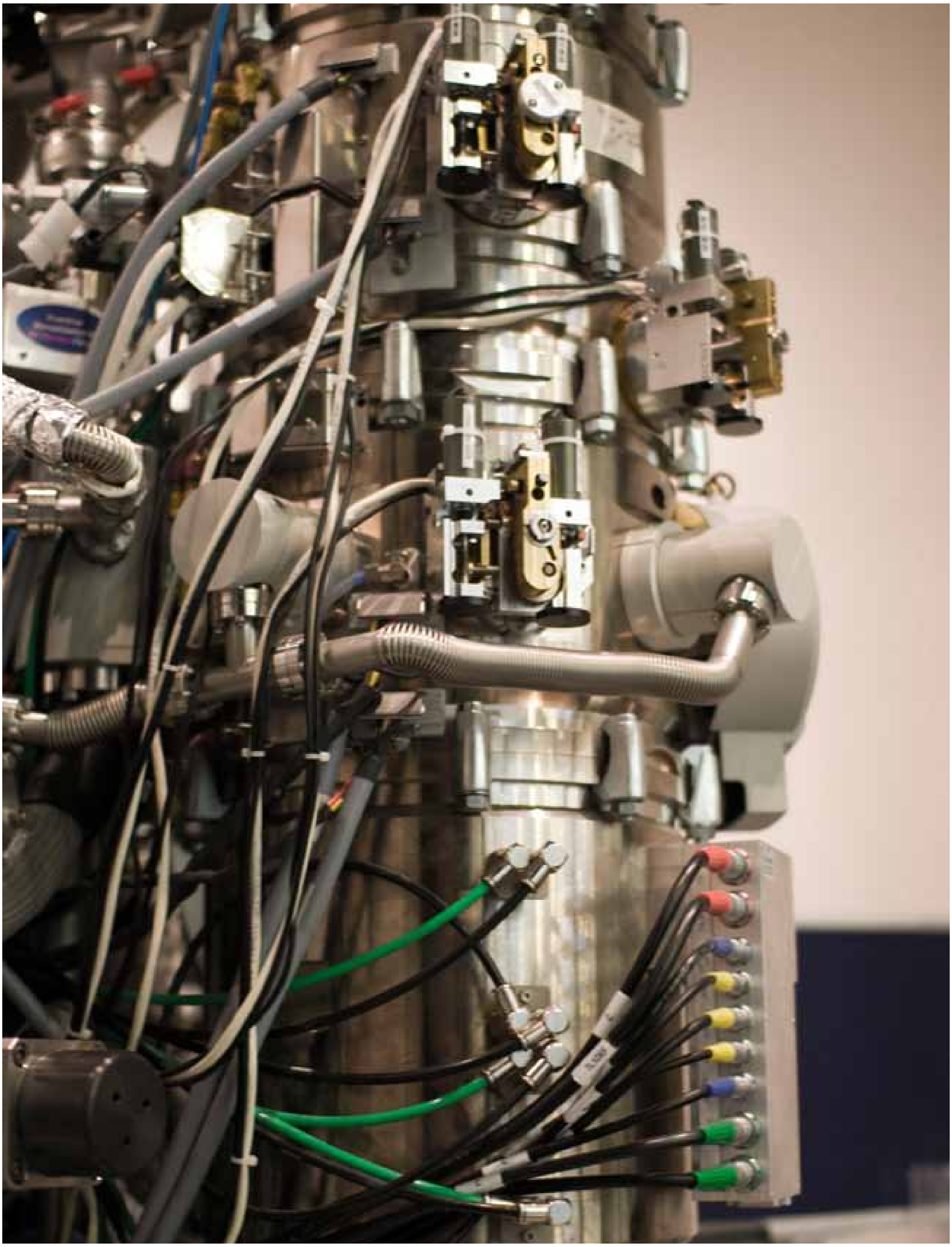
Main features

- FEG electron source
- electron beam accelerating voltage in the range 120 – 300 kV
- monochromator – 150 meV energy resolution
- probe C_S corrector – sub-Ångström STEM image resolution
- GIF Tridiem spectrometer
- high-angle annular-dark-field detectors
- EDX detector
- Lorentz lens – for Lorentz microscopy of magnetic materials
- biprism – for electron holography.

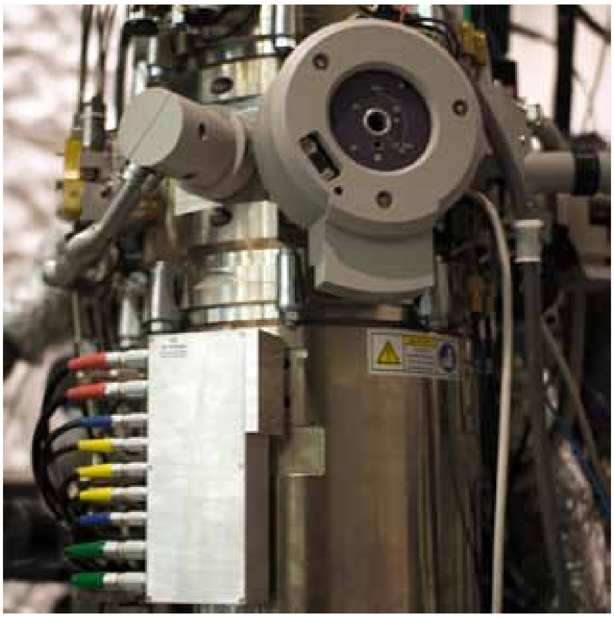
What can it do?

- atomic resolution studies of structures and chemical compositions of materials
- tomography for determining the 3D structure of materials
- Lorentz microscopy for studying magnetic and electro-static fields in materials
- electron holography measuring the magnetic and electric properties of materials, for example, magnetic interactions in arrays of nanoparticles or dopant distributions in semiconductor devices
- high energy resolution EELS electron energy-loss spectroscopy studies
- Nano e-beam lithography.


TITAN



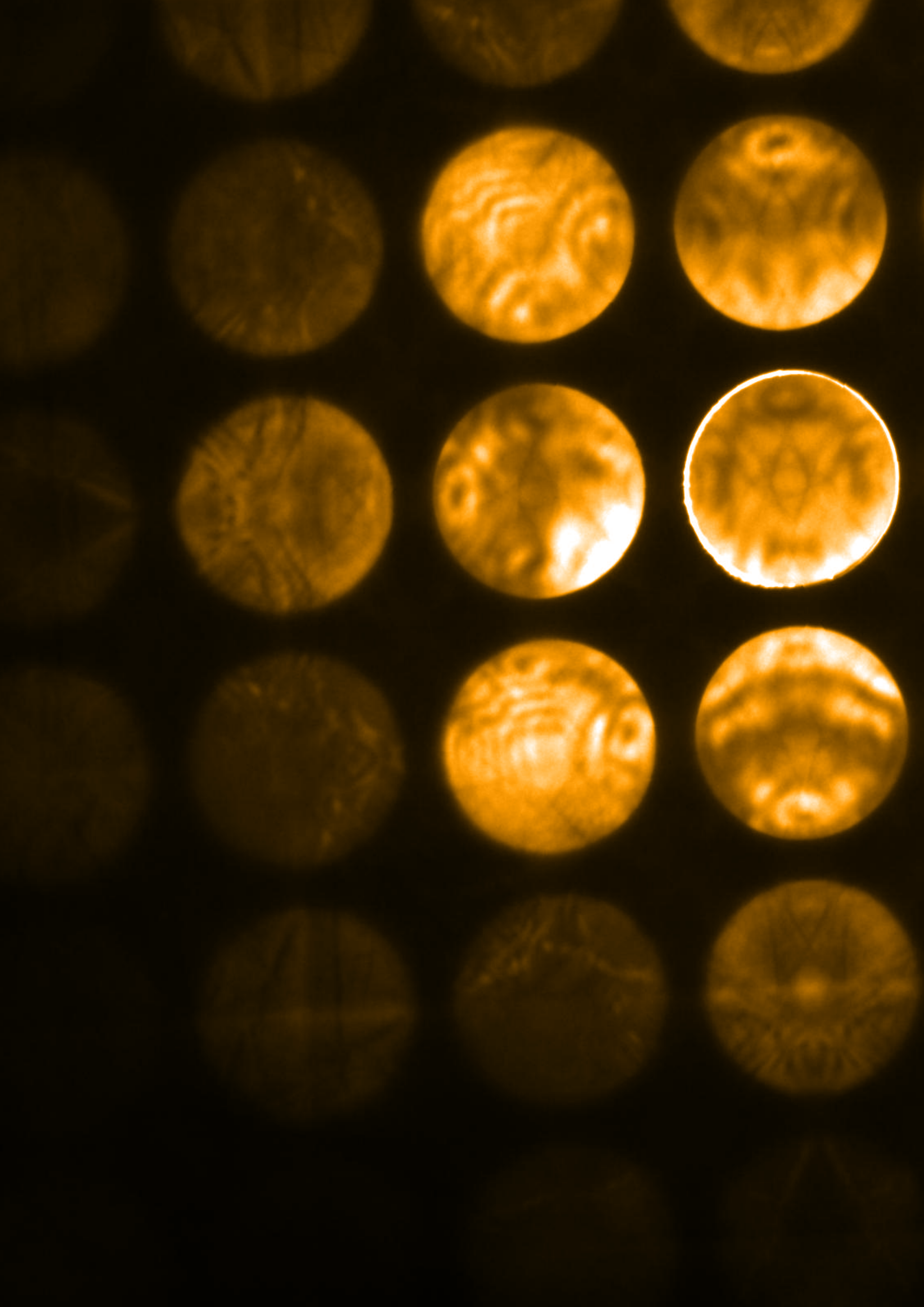
Titan E-Cell



What is it?
The Titan E-cell is a state-of-the-art transmission electron microscope (TEM) equipped with an environmental cell to allow studies of gas-solid interactions under controlled gas flow and at elevated temperature at the atomic scale.

Main features
- FEG electron source
- electron beam accelerating voltage in the range 80 – 300 kV
- image C_S corrector
- environmental cell – for in-situ studies of gas–solid interactions
- monochromator–energy resolution
- high-angle annular dark-field (HAADF) detectors 120 meV
- EDX detector
- GIF Tridiem spectrometer
- in-situ plasma cleaner

What can it do?
- atomic resolution studies of structures
- electron Tomography
- high energy resolution electron energy-loss spectroscopy (EELS)
- in-situ gas–solid interaction studies under controlled gas flow and at elevated temperatures
- in-situ plasma cleaning of specimens and the interior of the microscope for removing hydrocarbons



Funding

The following projects, on which DTU Cen staff are principal or named investigators, were funded in 2008 and 2009:

"SILICON-Light - Improved material quality and light trapping in thin film silicon solar cells".
EU FP7-ENERGY-2009-1 grant with Dr W Soppe, Dr D Fischer, Dr W Schipper, Prof. M Topič, Dr G Sánchez Plaza and Dr K Leitner.

"AMON-RA - Architectures, Materials and Onedimensional Nanowires for Photovoltaics - Research and Applications".
EU FP7-NMP-2007-SMALL-1 grant with Prof. K Deppert, Dr F Dimroth, Prof. B Witzigmann, Dr M Magnusson and Dr J Stangl.

"FunDMS - Functionalisation of Diluted Magnetic Semiconductors".
EU ERC-2008-AdG grant with Prof. T Dietl, Dr A Bonnani, Prof. J Gaj and Prof. J Majewski.

"CASE - The Catalysis Discovery Initiative: Sustainable Energy Solutions".
UNIK grant with Prof. J Norskov, Prof. I Chorkendorff, Prof. J Grunwaldt, Prof. C Christensen and Prof. M Mogensen.

Associated staff from other departments at DTU are principal or named investigators on several projects, which involve research using DTU Cen's electron microscopes. A selection of these projects is listed here:

"Nanolyse - Nanoparticles in food: analytical methods for detection and characterisation".
EU FP7 grant with Dr S Weigel, Prof. E Huusfeldt Larsen (DTU Food) and Dr K Mølhave (DTU Nanotech).

"Understanding interactions between cells and nano-patterned surfaces".
EU FP7-NMP-2007 grant with Dr K Mølhave (DTU Nanotech).

"Nanostructured toughened hybrid nanocomposites for high performance applications".
EU FP7-NMP-2007 grant with Dr A Horsewell (DTU Mekanik).

NATEC - NAnophotonics for TErabit Communications".
Villum Kann Rasmussen Center of Excellence headed by Prof. J Mørk (DTU Fotonik). **"**

"Nanoengineered graphene devices".
FTP grant with Dr P Bøggild (DTU Nanotech), Prof. A-P Jauho, Prof. T G Pedersen and Dr M Brandbyge.

"Grain boundary engineering of functional thin films".
FTP grant with Dr K Pantleon and Prof. M Somers (DTU Mekanik).

"The influence of stress on growth and transformation kinetics of interstitial alloys"
FTP grant. (DTU Mekanik).

"Micro alloyed high strength net shape components"
FTP grant (DTU Mekanik).

"Omega-3 food emulsions. Control and investigations of molecular structure in relation to lipid oxidation"
Fødevareforsknings-Programmet (DTU Aqua and DTU Mekanik).

"Fysiske principper for selvorganiserede materialer" (DTU Fysik).

Advanced Techniques

This section contains descriptions of a selection of the advanced electron microscopy techniques that are developed and applied at DTU Cen.

Electron tomography

Lionel Cervera-Gontard, DTU Cen

Electron tomography has been used in the biological sciences for more than 30 years. It allows three-dimensional information to be obtained from two-dimensional projections of a structure. The mathematical background to the recovery of three-dimensional information from two-dimensional images dates back to the work of Radon in 1917. The basis of tomography is the 'central slice theorem': *A projection of an object at a given angle is a two-dimensional central section through the three-dimensional Fourier transform of that object.*

Reconstruction of an object can then be achieved by acquiring enough projections to fill three-dimensional Fourier space. The real space object can be recovered using a three-dimensional inverse Fourier transform. In practice, limited experimental data usually means that some Fourier space information is missing, and the back-transform of the object becomes degraded.

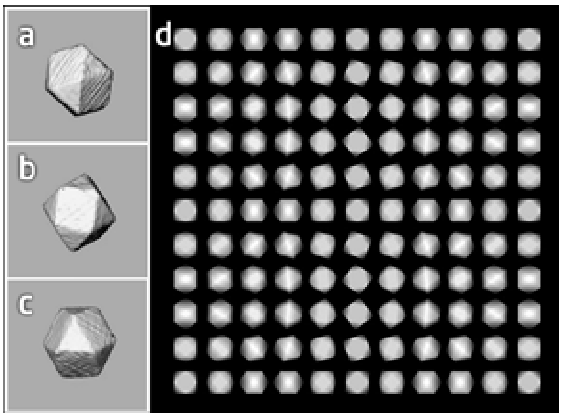
Practical electron tomography is based on four principles:

- the acquisition of a series of projections of the specimen.
- reconstruction of the three-dimensional volume using a chosen algorithm.
- tomogram enhancement.
- processing and visualization of the three-dimensional data.

The two-dimensional projections, or *tomograms*, that are used for reconstruction must meet the '*projection requirement*', that the signal collected should be an integral through the structure of some physical property. This condition implies that conventional bright-field and dark-field TEM images, which may be suitable for biological tomography, rarely provide useful signals for acquiring tomograms of crystalline specimens because they can be dominated by diffraction contrast, in addition to contributions from Fresnel contrast.

If an image is formed by using the signal collected using a STEM HAADF detector then most of the artefacts due to diffraction contrast that are present in CTEM images will be absent. Electrons that have been scattered to high angles are incoherent, and hence STEM images that have been acquired at medium resolution using an HAADF detector vary monotonically with composition and specimen thickness, satisfying the projection requirement for tomography.

STEM HAADF electron tomography has been applied to a wide range of problems in the physical sciences, including characterization of nanocrystals and catalysts. Unlike biological electron tomography, where there is a constant trade-off between beam damage, signal and resolution, HAADF STEM images of inorganic specimens usually have strong contrast and little noise, and there are fewer damage problems due to damage by the high-energy electron beam.

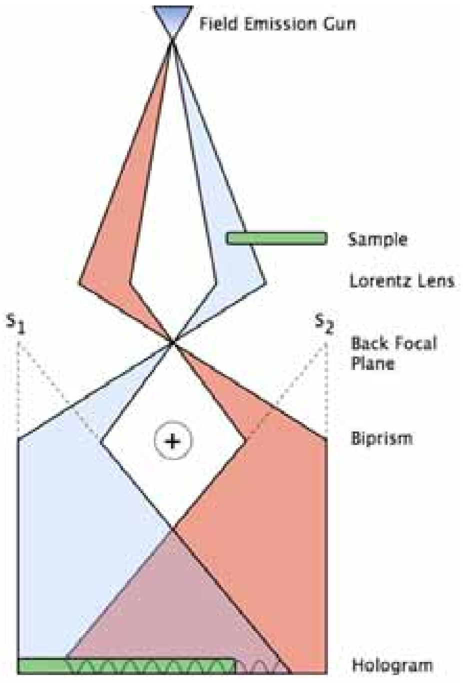


(A-C) Three views of the three-dimensional reconstructed cuboctahedral particle obtained in (d), inwhich the model is tilted about two orthogonal axes.

Electron holography

Takeshi Kasama, DTU Cen

a



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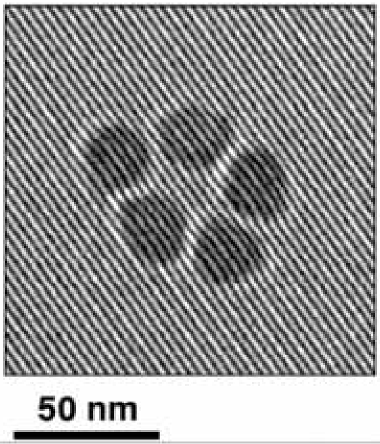


(a) Schematic diagram illustrating the application of a voltage to an electrostatic biprism in the TEM, in order to overlap a 'reference' electron wave that has passed through vacuum with the 'object' electron wave that has passed through the specimen.

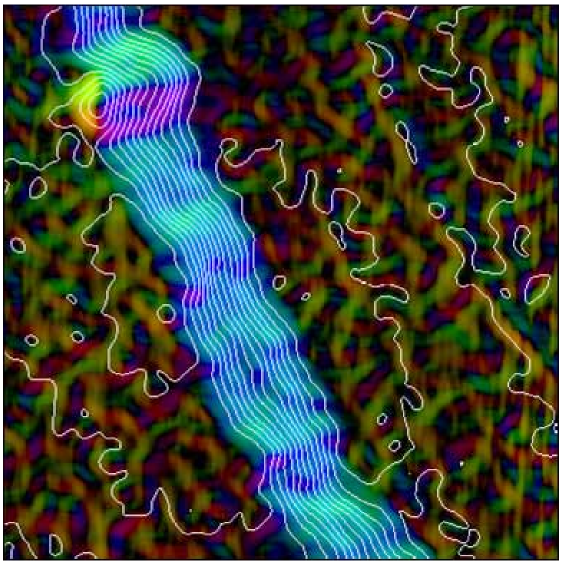
(b) Representative off-axis electron hologram of 20-30 nm cobalt nanoparticles. The fringe spacing is 2.9 nm.

Since Denis Gabor first proposed electron holography in 1948 to improve the resolution of TEM images, electron holography has been used in various fields. At DTU Cen, we have expertise in using medium-resolution off-axis electron holography to study magnetic and electrostatic fields in nanostructured materials. We are interested in improving the technique, as well as understanding the fundamental quantum phenomena.

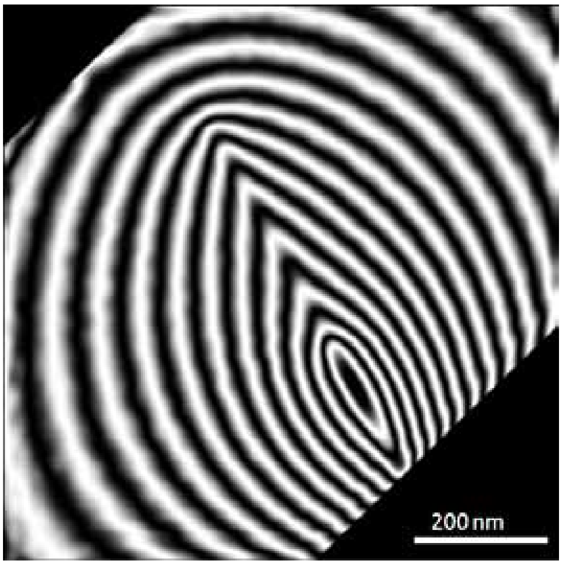
A schematic ray diagram of the experimental set-up for off-axis electron holography is shown below. In addition to a field emission gun, which provides a bright and coherent electron beam, two important hardware modifications are required: the installation of a Lorentz lens and an electrostatic biprism. In conventional TEMs, the current in an electromagnetic objective lens is used to produce high-magnification images of a sample. However, the objective lens is unsuitable for imaging in off-axis electron holography of magnetic materials because it produces a large magnetic field that can alter the magnetic structure in the sample.

Instead, a Lorentz lens is installed below the conventional objective lens to allow for work at high-magnification while keeping the sample in magnetic-field-free conditions. The objective lens is turned off, or is turned on temporarily to apply magnetic fields of up to 2 T to the sample. An electrostatic biprism, typically a gold-coated quartz wire with a thickness of ~1 µm, is installed in one of the selected-area apertures of the TEM.

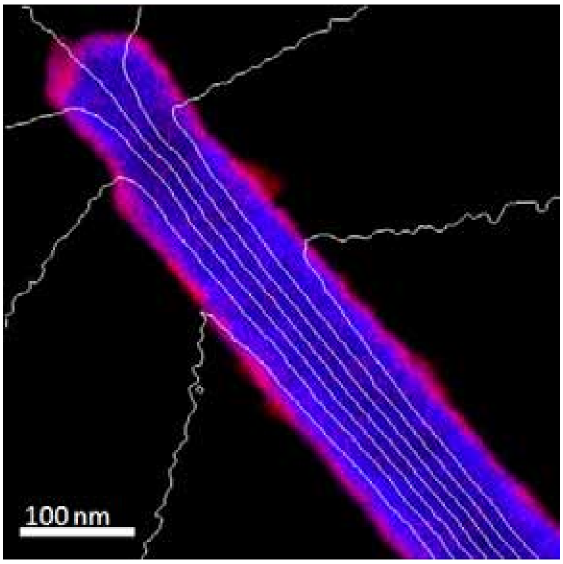
When a voltage is applied to the biprism, different parts of the electron beam are deflected so that they overlap. This overlap produces an interference pattern (a hologram), which has an appearance of a bright-field TEM image overlaid by interference fringes. The phase shift, which is recorded in the interference fringes, is sensitive to both the in-plane component of magnetic induction and the electrostatic potential in the sample. In this way electron holography allows magnetic and electric fields in the sample to be imaged quantitatively at a spatial resolution that can approach the nanometer scale. As electron holography is a TEM technique, one can determine the crystallographic features, chemical composition and three-dimensional shape of the sample examined using electron holography.



Magnetic induction map of self-assembled 15 nm-sized Co nano-particles showing local magnetic arrangements of vortex and zigzag states. The local magnetic structures are associated with the particle size, shape, crystallography, interparticle spacing and three-dimensional distribution of the particles. Collaboration with M. Varón and C. Frandsen.



Contoured phase image of the electric field in the vicinity of a bundle of single-walled carbon nanotubes that has a voltage applied to it in-situ in the TEM. The electric field is strongest close to the end of the nanotube bundle. The voltage was applied using three-contact electrical biasing TEM specimen holder 4 times phase amplification. Collaboration with G. Pozzi.



Magnetic induction map of an amorphous CoFeB nanowire over-laid onto an elemental map measured with energy-filtered TEM. In the elemental map, blue and red represent cobalt and oxygen, respectively. The surface layer is cobalt oxides. The magnetic signal is detected in the CoFeB phase but not in the oxide layer. The phase spacing is 2 radians. Collaboration with K. Kavanagh.

Environmental transmission electron microscopy

Jakob Birkedal Wagner, DTU Cen

The study of gas-solid interactions at the atomic scale is important for understanding several material problems such as the working state of catalysts, growth mechanisms, functionalized surface coatings and failure analysis (for example deterioration of stainless steels and solid oxide fuel cells). An understanding of such materials and processes on the microscopic level provides invaluable information for tailoring and tuning materials properties on the macroscopic level for optimized industrial performance.

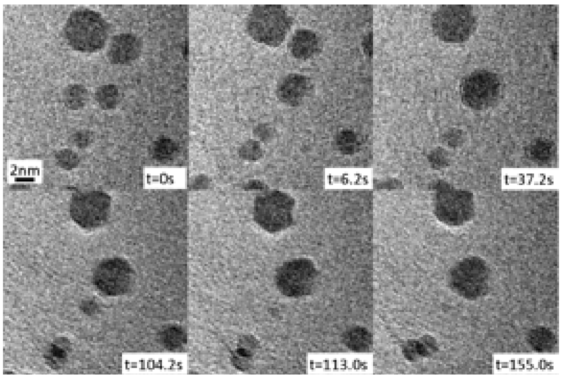
Few facilities in the world provide capabilities for investigating gas-solid interactions on the atomic scale. The Titan E-cell (ETEM) at DTU Cen, is a unique state-of-the-art monochromated and image C_s-corrected TEM instrument equipped with an environmental cell, making it possible to study gas-solid interactions at the atomic level under controlled gaseous atmospheres and at elevated temperatures. Furthermore, gas-solid interactions can be investigated by the acquisition of high frame-rate movies. This capability provides an insight into reaction kinetics, for instance by tracking particles over time or directly monitoring the growth and morphology of nanostructures.

Conventional electron microscopes require high vacuum around the specimen in order to maintain high resolution and optimal operation. The idea of the environmental cell is to confine the gas to a small region around the sample. This is accomplished by limiting gas diffusion through the column by inserting extra apertures and pumps. In this way, the volume with high gas pressure is minimized along the electron beam path.

Sintering of supported metal particles:
Active sites on supported metal catalysts are found on the surfaces of the metal particles. Sintering is the process by which the particles increase their diameters resulting in a lower surface area/volume ratio.
Two schemes for sintering are generally presumed:

- Ostwald ripening, where the particles are immobile and net mass transport from smaller to larger particles is accounted for by single atoms or molecular species
- particle migration and coalescence, where the particles themselves migrate over the surface of the support. When two particles are in close proximity, they can coalesce into a single particle.

In order to elucidate this phenomenon, a model catalyst was made by sputter-coating gold onto boron nitride. The model catalyst was exposed to 1.3 mbar of hydrogen at 400°C in the environmental TEM.



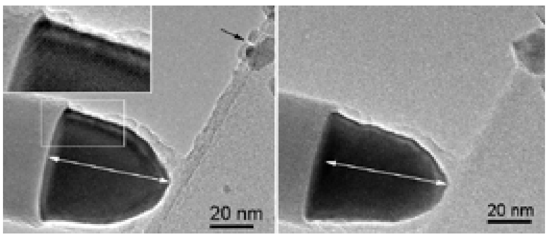
*6 frames from a movie are shown. From frame a to frame b, particles 1 and 2 coalesce into a single particle. Between frames b and c, the same phenomenon is observed for particle 3.
From frame c to frame f, particle 4 gradually becomes smaller indicating that Ostwald ripening is taking place.
Such studies show that both sintering mechanisms have to be taken into account when studying catalyst deactivation.*

Examples of ETEM

Reverse growth of semiconductor nanowires:
The growth and characterization of semiconductor nanowires is of considerable interest. One unresolved question concerns the growth mechanism of nanowires grown with catalyst particles. Some nanowires are grown at temperatures below the eutectic temperature for the catalyst-semiconductor alloy, at which it is not clear that the catalyst-semiconductor alloy is liquid. The InAs nanowire system has an abrupt reduction in growth for temperatures above 500°C. Therefore, examination of these nanowires in and around this temperature is useful for further illumination of the growth model and dynamics.

In this work we heat InAs/InP nanowire hetero-structures *in situ* in the TEM, resulting in nanowire dissolution both at and away from the catalyst-nanowire interface.

In an attempt to remove the native oxide formed on the nanowires, the heating experiment was carried out in a reducing atmosphere of 1.3 mbar of molecular hydrogen. Heating the sample to 520°C in H_2 resulted in the Au-containing seed particle changing shape while it dissolved the wire and new crystallites of indium oxide were formed nearby.

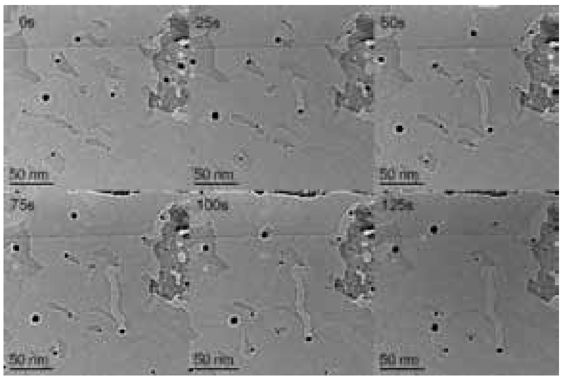


Snapshots from a movie during reverse growth of an InAs nanowire at 520°C in 1.3 mbar H_2. The white arrows, which span the same length, show the elongation of the Au particle over time. The black arrow in the left image indicates new particle formation. The images are acquired 3 min. apart.

Although the seed particle changes shape dramatically, it remains crystalline at this temperature, as can be seen from the lattice fringes. The formation of indium oxide particles is observed on the carbon film nearby.

As the temperature at which these movies were acquired is well above the growth temperature, the crystallinity of the particle in these movies is a strong indication that the particle also remains crystalline during growth at lower temperatures.

Ag-assisted layer by layer decomposition of graphite:
Ag nanoparticles are observed to decompose graphite layer by layer in an oxidizing atmosphere at 500°C. This fundamental study of chemical reactions and catalytic processes involving few layer graphite or graphene is carried out in the ETEM in real time at close to atomic resolution to give valuable insight into the driving forces of the reactions.

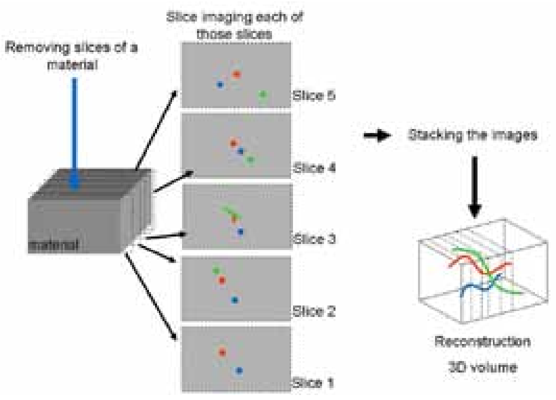


Frames from a movie acquired in real time under reaction conditions in the ETEM. The darker particles are Ag and the grey background is few-layer of graphite. The contrast of the graphite can be related to the number of carbon layers. By following the Ag particles, it is observed that a brighter trail is left behind, indicating the removal of carbon layers. Single layers of carbon appear to be decomposed by each Ag particle. Furthermore, the direction of movement of the Ag particles is discretely controlled by the crystal structure of the graphite.

SEM Techniques

Alice Bastos da Silva Fanta, DTU Cen

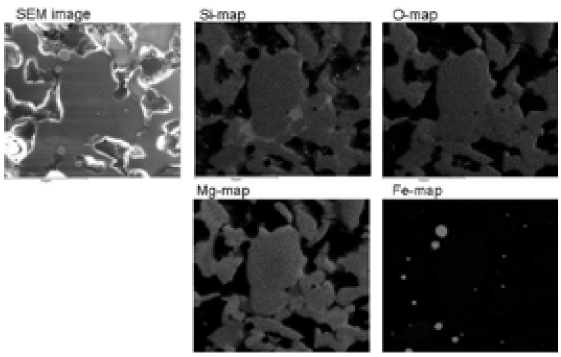
3D characterization by serial sectioning
Tomography by serial sectioning is based on removing slices of a material, imaging each of those slices, and stacking the images together to reconstruct the 3D volume. Material removal can be performed in different ways. The FIB-SEM tomography technique, uses the combination of the two beams to perform 3D characterization inside the microscope chamber. This allows accurate material removal (on the order of 10 nm) and precise re-localization of the area of interest. This technique has the advantage of precise reconstruction of the 3D volume with high 3D resolution. Although the FIB-SEM tomography technique expands the characterization possibilities of the SEM the combination of this technique with EDX and EBSD greatly increases the power of the system.



Schematic representation of 3D tomography by serial sectioning.

Energy-dispersive X-ray spectroscopy
Energy-dispersive X-ray spectroscopy is a standard analytical technique used for chemical microanalysis. In a conventional SEM, the chemical information can only be collected from the surface region of a bulk specimen. However, in combination with material removal (serial sectioning in a FIB-SEM) elemental analysis can be extended to the third dimension. This is, for example, used for studies of crack formation in multiphase materials.

Electron backscatter diffraction (EBSD)
Electron backscatter diffraction (EBSD) allows spatially resolved quantification of crystallographic phases and orientations and the reconstruction of microstructure and texture. EBSD is based on the fully automated analysis of Kikuchi patterns from the scanned area on a crystalline sample. These patterns are captured by a detector positioned close (usually between 20 and 40 mm) to a strongly tilted sample (approximately 70°) inside the SEM chamber. Although the EBSD technique can be applied with success using a standard tungsten-filament SEM, the spatial resolution and orientation accuracy improves when using a field emission gun instrument with a high beam current. The spatial resolution of EBSD-based orientation microscopy is on the order of 20 to 50 nm depending on the material under consideration. EBSD can also be performed in combination with EDX in an automated manner allowing simultaneous crystallographic and chemical characterization.



EDX analysis of olivine.

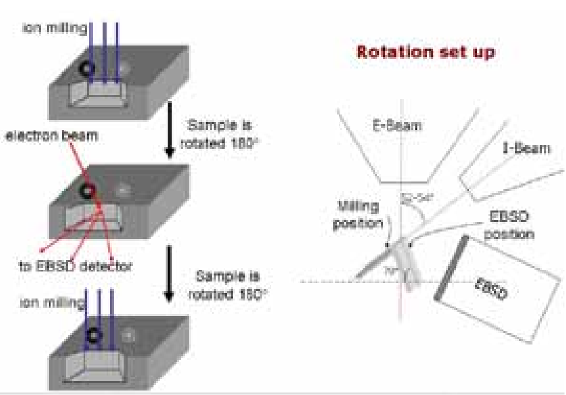
EBS3 (3D orientation microscopy)
In the last 15 years, the 2D EBSD technique has been developed into a powerful tool for the crystallographic study of materials in the SEM. Today 3-dimensional EBSD characterization can be used to study the size and distribution of pores, inclusions, and cracks in a sample by FIB-SEM tomography. However, when the feature to be studied can only be described by its crystallographic characteristics, the combination of FIB tomography and EBSD must be applied.

Grain boundaries are three dimensional crystallographic defects. The natures of grain boundaries can only be studied by serial sectioning in combination with EBSD.

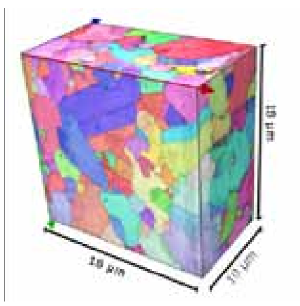
EBS3 is based on 3D tomography in a dual beam system, whereby an EBSD map is performed at every slice of the study. The FIB is applied for the preparation of smooth surfaces by milling the material at grazing incidence. This sectioning process allows controlled removal of thin layers of material with a minimum slice thickness of approximately 20-50 nm. The resulting smooth surface is in most cases very well suited for the formation of electron backscatter diffraction (EBSD) patterns.

After the milling process, the sample is positioned for the acquisition of these patterns. To this end, the sample is rotated by 180° inside the SEM chamber.

At this position, the microstructure of this surface can be analyzed by conventional 2D EBSD-based orientation microscopy. By successive sputtering and EBSD measurements, the 3-dimensional microstructure can be explored. Each of the sections obtained is then used to reconstruct the original microstructure in three dimensions. This process is controlled by software, and runs automatically. By using this technique, the three dimensional crystallographic characteristics of a sample can be explored with a spatial resolution of ~ 50 x 50 x 50 nm^3.

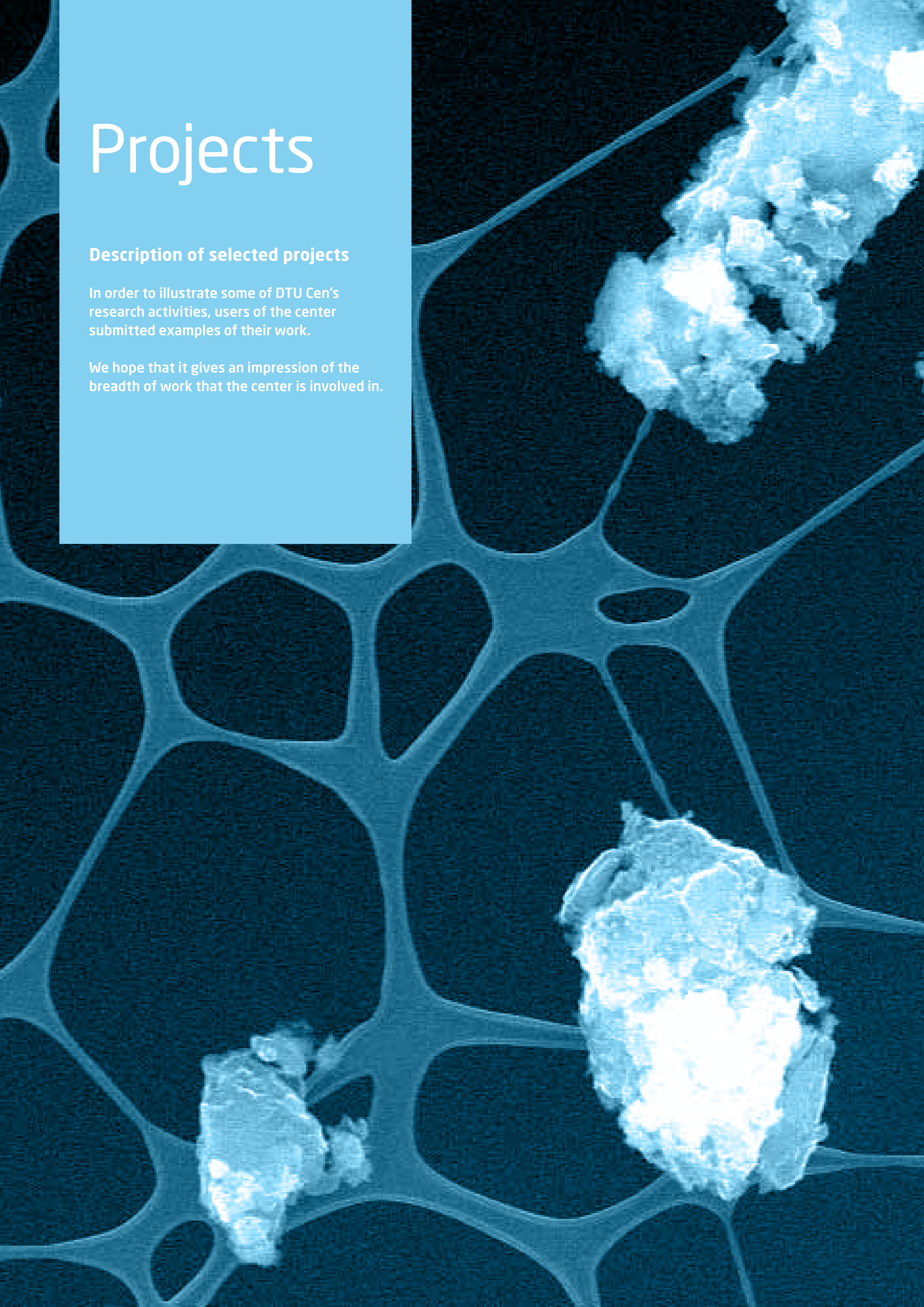


Scematic view of experimental setup for 3D EBSD.



3D EBSD map of an Ni-based superalloy, reconstructed from 100 slices, spatial resolution of 100 nm x 100 nm x 100 nm.

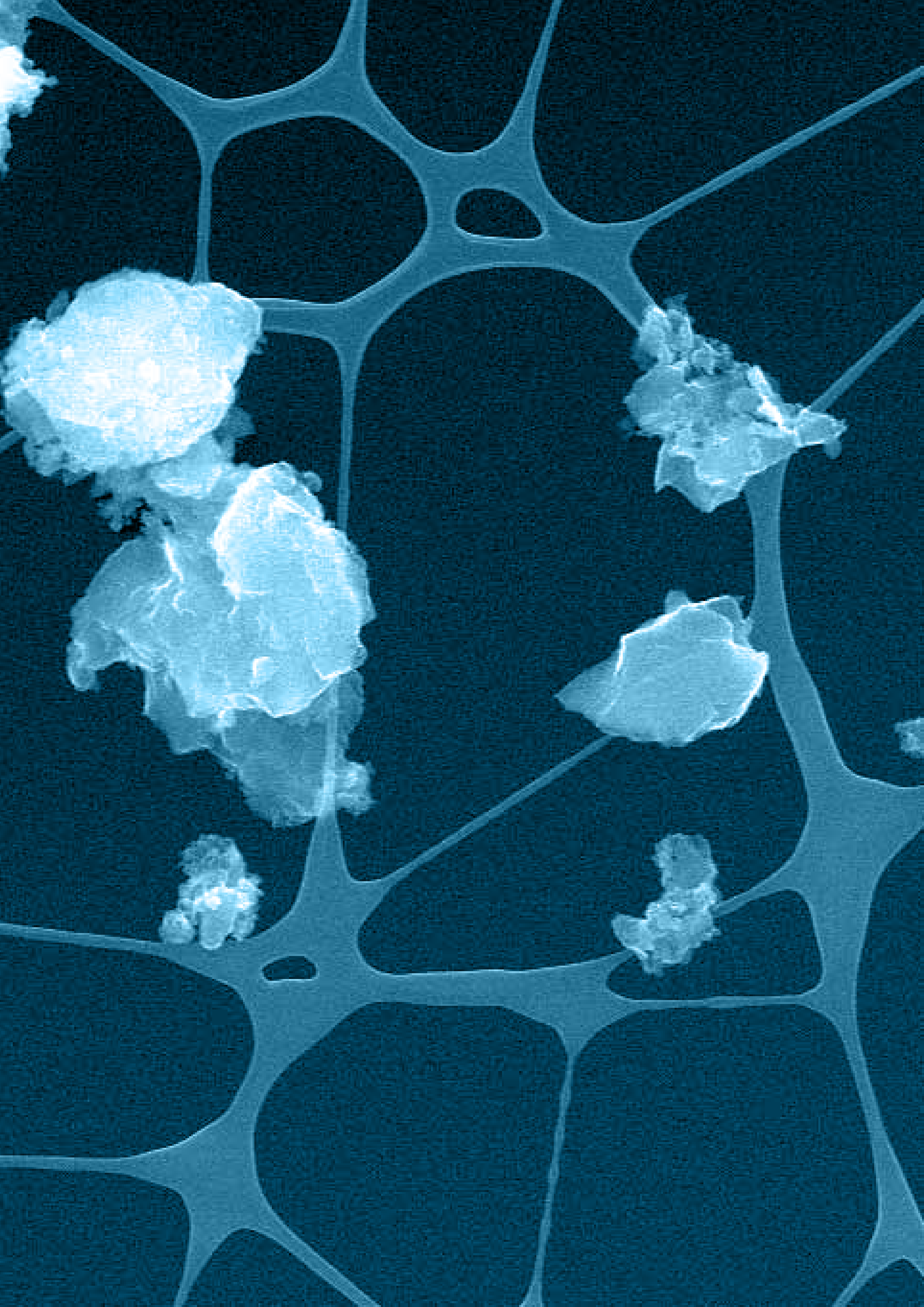
Projects



Description of selected projects

In order to illustrate some of DTU Cen's research activities, users of the center submitted examples of their work.

We hope that it gives an impression of the breadth of work that the center is involved in.



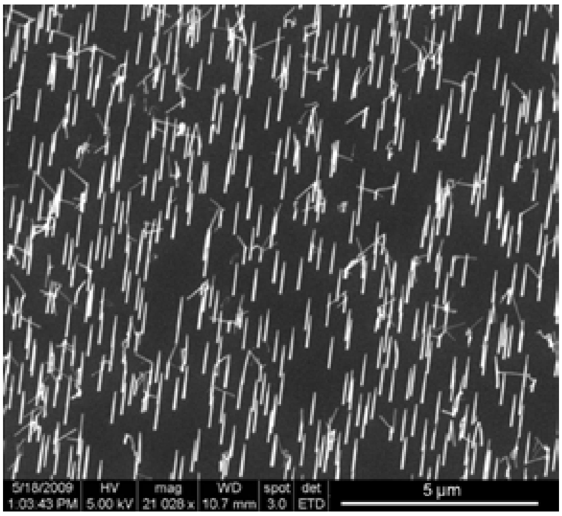
Architecture, materials and one-dimensional nanowires for photovoltaics (AMON-RA)

Johan Persson, DTU Cen

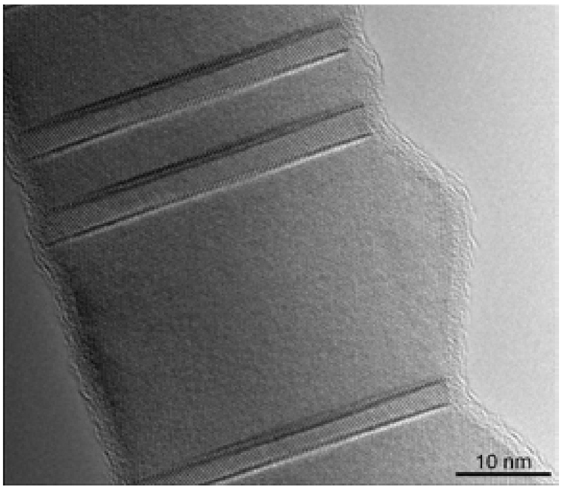
Solar cells are considered an integral part of building future sustainable energy production. Current solar cells are only able to use a fifth of the solar energy that arrives at the earth's surface. One way of increasing this efficiency is to construct what is called a multi-junction solar cell. This is much more efficient than a normal solar cell simply because it consists of several layers that are specialized for a certain part of the sun's spectrum.

AMON-RA is a four year EU 7^{th} framework project which aims to realize a working nanowire-based multi-juction solar cell (www.amon-ra.eu). It is a joint project between the Technical University of Denmark, Lund University, the Fraunhofer Institute for Solar Energy Systems, the University of Kassel, Johannes Kepler University in Linz and the Lund-based company Sol Voltaics AB.

The semiconductor nanowires investigated in this project are grown at Lund University. Characterization of the nanowires is carried out using the electron microscopes at DTU Cen. SEM and TEM imaging techniques are used to examine the morphologies of the nanowires, while EDS and EELS are employed to characterize their chemical compositions. In addition, the electrical properties of the nanowires, such as their electrical potential profiles are investigated using electron holography.



SEM image of a nanowire forest.



HRTEM image of a nanowire.

The catalysis discovery initiative: sustainable energy solutions (CASE)

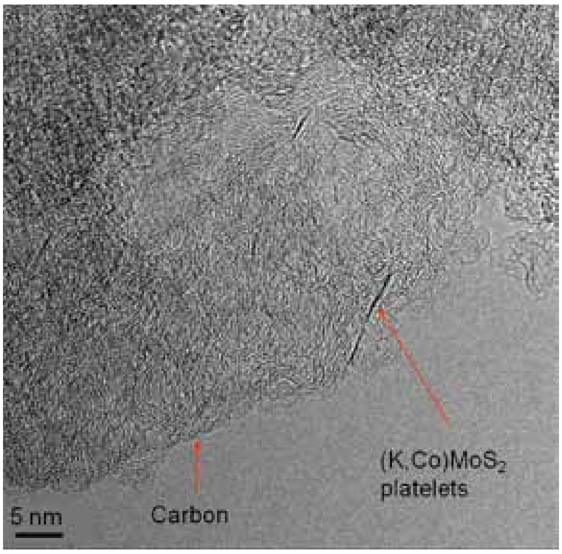
Thomas Willum Hansen, DTU Cen

The wealth created in the world over the last century is based on easy access to inexpensive fossil fuels. This era is coming to an end. Resources are limited. At the same time, it is becoming increasingly clear that the emission of CO2 that follows the use of fossil fuels is threatening climate. Arguably, this makes the development of sustainable energy solutions the most important scientific and technical challenge of our time.

It is the aim of the present proposal to contribute to the solution of these problems in selected areas, by addressing the underlying scientific questions and using the insight to point to solutions to some of the technical problems.

Efficient harvesting of solar energy is the only technology that has the potential to eventually supply the entire population of the Earth with sufficient energy in a sustainable way as a standalone long-term solution. The full potential of harvesting solar energy is emphasized by the fact that covering 0.1 % of the surface of the planet with a device that converts solar energy into a useable form at 10% efficiency would give more than the present worldwide consumption of fossil energy.

2 PhD scholarships have been allocated to DTU Cen within the CASE project. Both projects revolve around investigating materials in an environment close to that in which they operate. The first project focuses on in situ light exposure of samples inside the TEM. Such studies can elucidate the working states of solar cell materials and photo-catalysts. The second project focuses on high-resolution aberration-corrected imaging of catalysts for alcohol synthesis under a simulated working environment with respect to gaseous atmosphere and temperature.



High-resolution image of (K,Co)MoS$_2$ platelets supported on carbon. The sample is used as a catalyst for higher alcohols synthesis.

TEM holder with in-situ light emission capability.

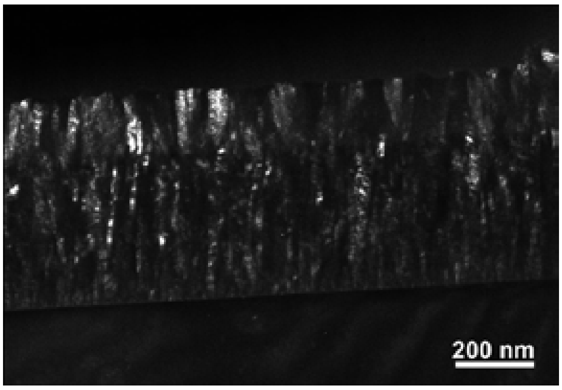
Functionalisation of diluted magnetic semiconductors (FunDMS)

András Kovács, DTU Cen

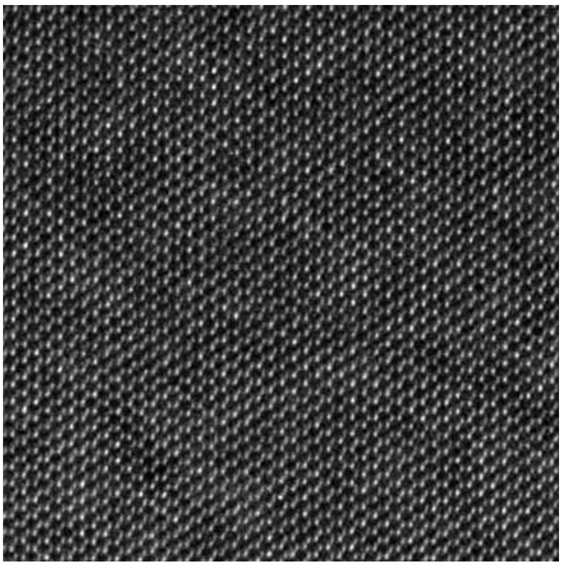
The manipulation of magnetic spins in semiconductor layers and nanostructures is a central theme in the development of spin-based information technologies - "spintronics." Wide bandgap semiconductors and oxides doped with transition metals, such as GaN doped with Fe, ZnO doped with Co and GaAs doped with Mn, constitute a new class of materials, whose magnetic properties are challenging to control and (yet) still poorly understood.

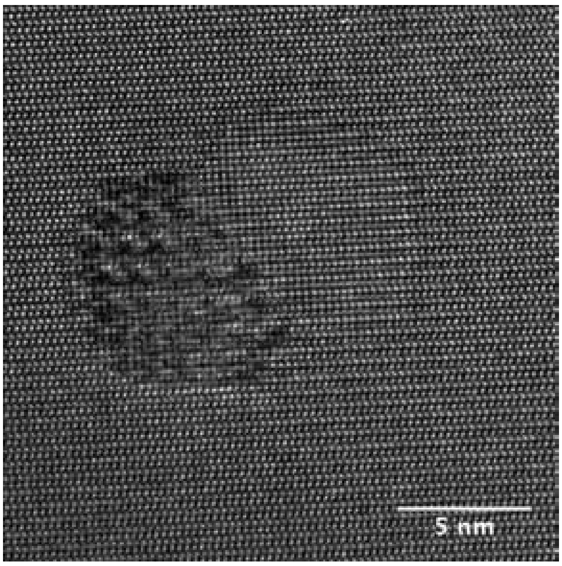
FunDMS is a European Union ERC Advanced Research Grant co-ordinated by Professor Tomasz Dietl (IFPAN, Warsaw, Poland), in which DTU Cen is a partner.

DTU Cen will be working on the FunDMS project, carrying out high spatial resolution quantitative chemical, structural and magnetic characterization of a wide range of diluted magnetic semiconductors using advanced electron microscopy techniques. A key aim of the work will be to understand the local origin of the ferromagnetic signatures in these materials, through the characterization of both embedded nanocrystals and diluted distributions of magnetic dopant atoms.



Dark-field TEM image of columnar grain structure in a Co-doped ZnO layer grown on a silicon substrate.





Aberration-adjusted high-resolution lattice image of Fe-rich precipitate adjacent to a void in GaN.

Grain boundary engineering of functional thin films

Hossein Alimadadi, DTU Mek

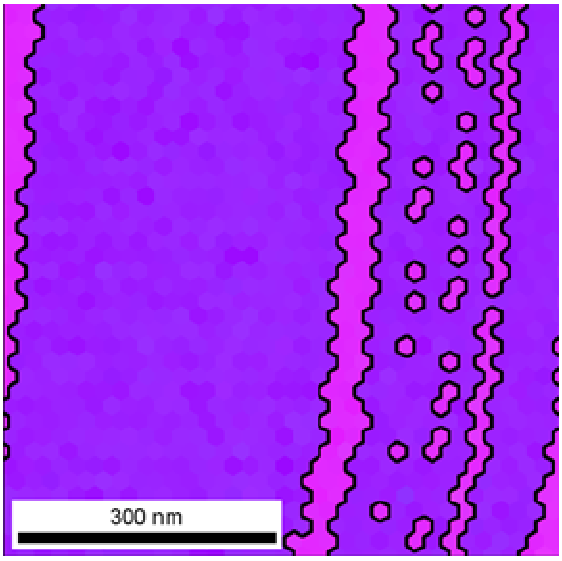
The deliberate manipulation of grain boundary characteristics - so-called grain boundary engineering - is considered the key for designing and controlling the functional properties of certain materials. Some special boundaries, like the coherent Σ3 twin boundary, have particularly low boundary energies and, accordingly, in comparison to random high angle grain boundaries have special properties. For example some boundaries in nanostructured materials result in improved mechanical properties even at elevated temperatures, because the boundaries are rather immobile and thermally stable and, hence, prevent grain growth and hinder dislocation movement. Furthermore, such boundaries contribute much less to electrical resistivity and, hence, result in higher electrical conductivity.

The fabrication of materials with a high density of special boundaries is of particular interest for improving mechanical properties without deteriorating the physical properties. The key technology to fabricate such materials is electrochemical deposition. Active cross-linking between targeted materials processing and microstructure characterization is required.

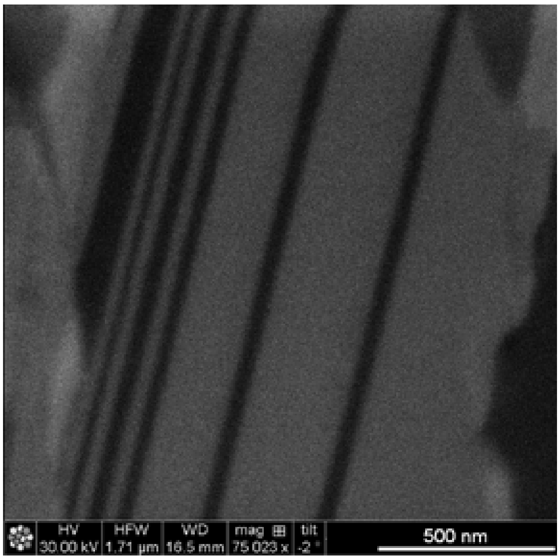
This project aims to tailor the internal structures of thin electrochemically deposited films for advanced applications in technological areas, based on a close collaboration between DTU-Mek (where targeted materials processing is performed) and DTU-Cen (where electron backscatter diffraction (EBSD) is applied onthe FEI dual-beam FIB-FEG SEM Helios).

EBSD analysis supplemented by ion imaging is a useful tool for characterizing special (twin) boundaries in nanostructured films. In addition, complete three-dimensional (3D) quantitative information on the internal structures of the films is essential for well directed grain boundary engineering. Conventional 2D EBSD mapping can be extended into the third dimension by successive serial sectioning using FIB milling.

Complete microstructural characterization by 3D EBSD, including local orientation relations between grains, spatial distributions of the grain sizes, shapes and crystallographic orientations and boundary types, will allow tailoring of the functional properties of the films.



High-resolution EBSD map showing twins in an as-deposited nickel thin film.



High resolution FIB image showing twin boundaries in an as-deposited nickel thin film

Microstructure evolution during friction stir welding

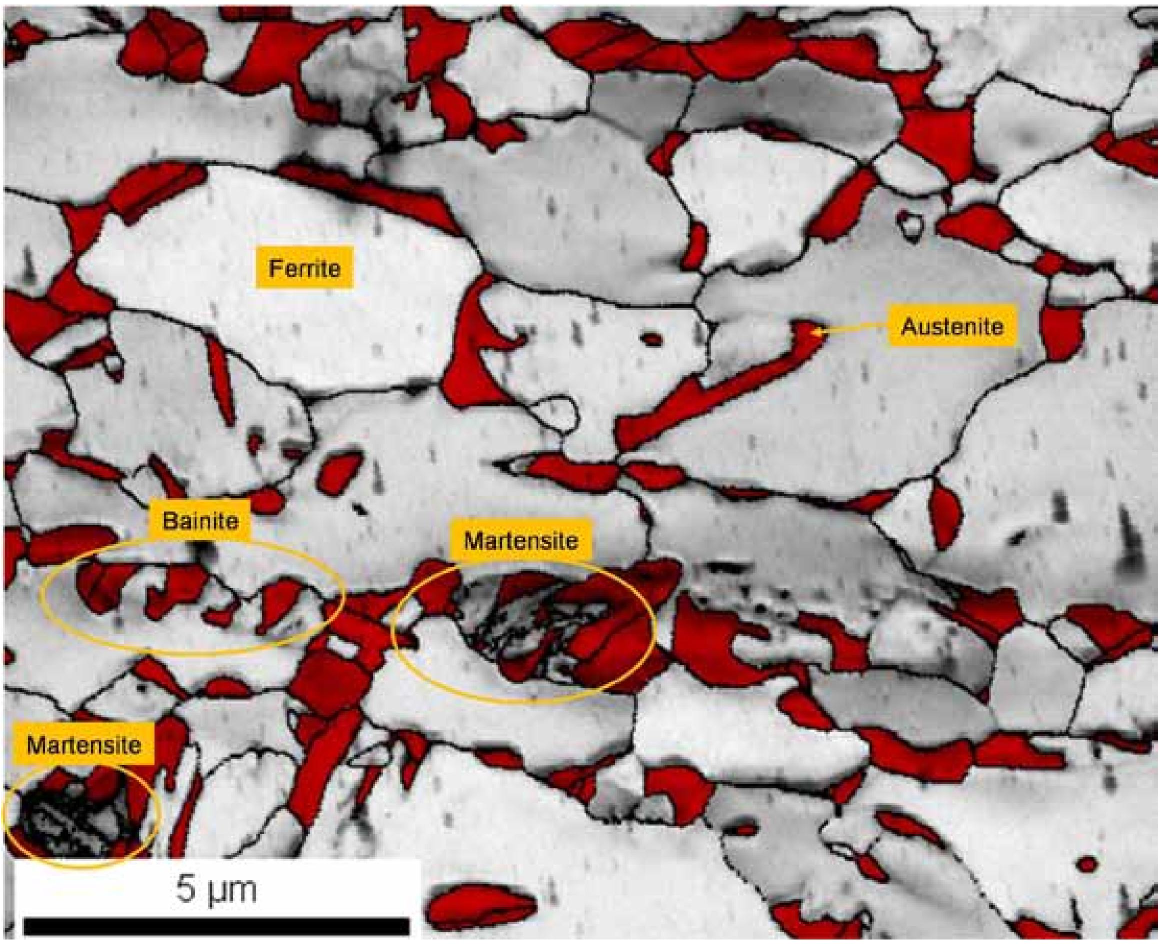
Trine Colding Lomholt, DTU Mek

In August 2008, a PhD project was started on the characterization of microstructure evolution during friction stir spot welding of TRIP steel. Material characterization involves investigating the micro-structure and associated properties and is performed on the welds, the heat affected zones and their immediate surroundings. Reflected light microscopy, SEM and TEM, are applied for the determination of grain size and grain shape distributions and identification of morphology.

The equipment for reflected light microscopy and electron microscopy is available at DTU Mechanical Engineering, Risø DTU and DTU Cen.

In particular, 3D characterisation of welds with the Helios Dual Beam FIB-SEM at DTU Cen is expected to yield unique results. X-ray diffraction and electron diffraction techniques will be applied for identification of the phases constituting the microstructure.

Moreover, texture development in and around the welds will be determined, primarily with EBSD techniques. Stress distributions (both micro and macro stress) in and around the welds will be determined with X-ray diffraction. The microstructural details will be compared to micro-hardness distributions, which are a measure of the distribution of mechanical properties around the weld.



Nanostructured toughened hybrid nanocomposites

Alessandra Mosca, DTU Mek

This project is a part of the EU 7th Framework NanoTough project, which aims to produce cheaper and lighter materials with tailor-made properties based on the incorporation of inorganic nanoparticles into a polymer matrix.

The material system studied here is polyolefin (mainly polypropylene (PP)) containing clay (layered silicate) nanoparticles. The main idea is to replace metals, heavily filled polymers and expensive engineering polymers currently used in the automotive industry with PP in order to reduce weight and obtain the same or better properties at lower cost.

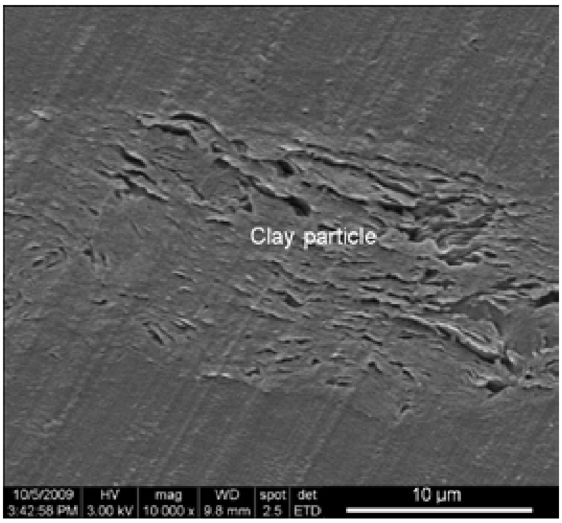
The introduction of silicate nanoparticles in PP results in considerable improvement in stiffness at the expense of toughness. However, through carefully designing multicomponent systems, toughness may also be increased.

A primary objective of this project is to obtain a better understanding of the structural and interfacial properties of polyolefin based nanocomposites. Electron microscopy techniques (SEM and TEM) are carried out at DTU Cen to study silicate nanoparticles in PP, particularly related to their interfaces.

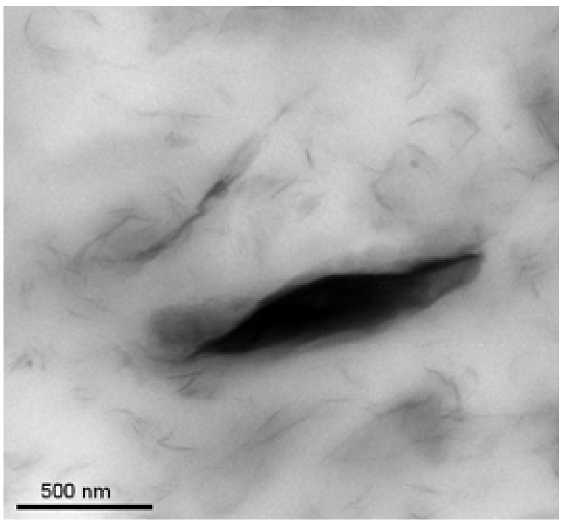
The SEM image of the clay – PP nanocomposite, shows that the surface is relatively smooth, with some indication of scratches on the surface from the microtoming sample preparation. A large (30 µm x 15 µm) clay particle is observed, as well as some pull-outs or porosity. The presence of the clay particle was confirmed by back scattered electron (BSE) images.

In the bright field TEM micrograph of the clay – PP nanocomposite, the dark region most likely represents part of a larger clay particle, which was not fully exfoliated during preparation. The average size is on the order of a few microns, hence many times smaller than the clay particle observed in the SEM image.

The lighter regions in the TEM image are variations in the thickness of the sample (not voids), and are likely to be a microtoming artefact. The sample contains dispersed layers of platelets. Stacked clay layers are believed to increase stiffness, and thus represent an advantage for the development of nanocomposites with improved mechanical properties. The spacing between the layers is very regular and is on the order of a few nanometers.



SEM micrograph of clay - PP nanocomposites.



TEM micrograph of clay - PP nanocomposites.

Nanophotonics for terabit communication (NATEC)

Shima Kadkhodazadeh, DTU Fotonik/DTU Cen

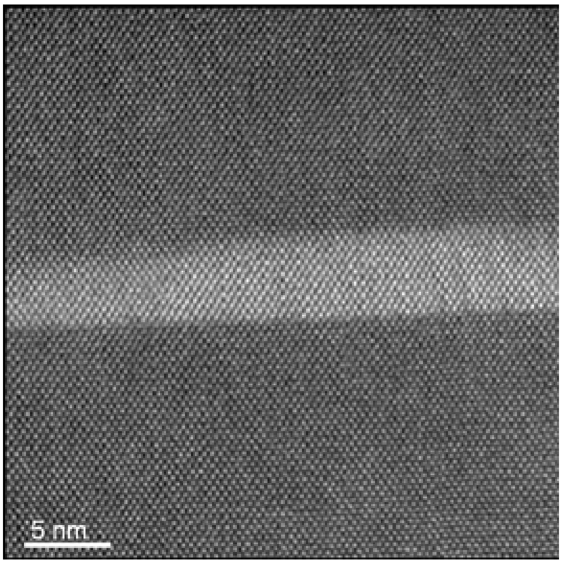
Photonic crystal structures and semiconductor quantum dots provide new possibilities for controlling the properties of photons and electrons at a fundamental level. It is expected that it will eventually be possible to realize chips in which photons play a similar role to that of electrons in present-day electronics chips. The argument for pursuing the use of photons rather than electrons for data processing is mainly one of speed.

In the near future, it will be necessary to communicate and process data in the terabit per second regime. This prospect entails fundamental challenges within basic science and opportunities for introducing new technologies.

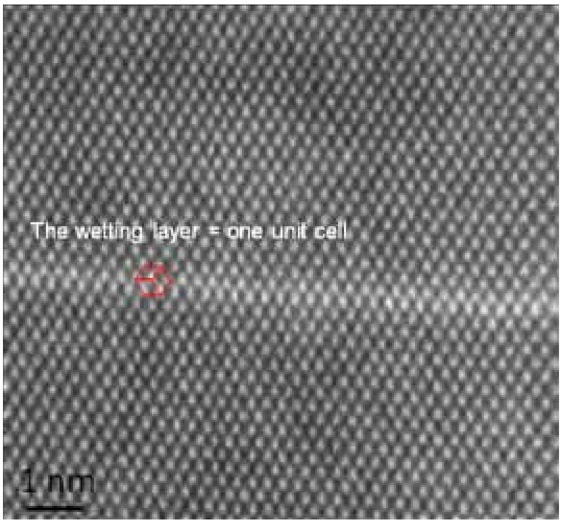
The NATEC project is a collaboration between several departments at DTU and includes nanofabrication, theory and device characterization and optimization.

The work at DTU Cen involves characterization of semiconductor quantum dots. The optical and electronic properties of quantum dots are believed to be largely affected by their structures and chemical compositions. Thus the successful application of the quantum dots as photonic devices requires a good understanding of their structure property relationships.

This project aims to combine electron tomography with other analytical TEM techniques to determine 3-dimensional information about the dots. Such information can then be correlated with the optical and electronic properties of quantum dot devices, as well as providing feedback for the fabrication and modelling of desired structures.



High resolution HAADF STEM image of an InAs-InP quantum dot.

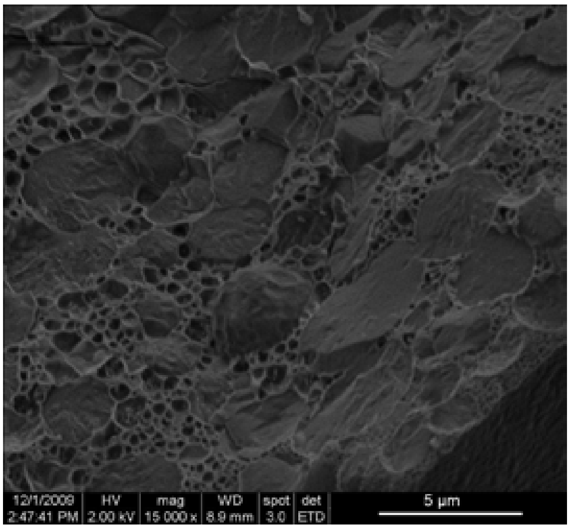


High resolution HAADF STEM image of the wetting layer of InAs - InGaPAs.

Omega-3 food emulsions

Louise Helene Søgaard Jensen, DTU Mek

The addition of fish oil to industrially prepared foods is an attractive option because of the well-documented health effects of omega-3 fatty acids. Polyunsaturated fatty acids (PUFAs) are highly susceptible to lipid oxidation due to their many double bonds. Oxidation causes deterioration of nutritional value and flavour. This project aims to explore the factors that influence lipid oxidation in emulsions of fish oil and water, because emulsions such as mayonnaise and milk products are potential candidates for enrichment with omega-3 PUFAs. It has been suggested that oxidation is initiated at the interface between oil and water and is dependent on the structure of this interface, along with a number of other factors. The overall goal of this project is to develop new strategies for preventing oxidation in food emulsions that are rich in omega-3 fatty acids in order to help the Danish food industry to improve its position on the fast growing market for omega-3 enriched foods.



SEM micrograph of light mayonnaise.

The aims of the microscopy studies are to extend existing capabilities and to develop new techniques for investigating:

- the general characteristics of the emulsion, in particular, volume fractions, size distributions and separations of the droplets
- the width and structure of the oil-water interfacial layer
- the localisation of iron (acts as pro-oxidant) and phosphor lipids that may be used as emulsifiers.

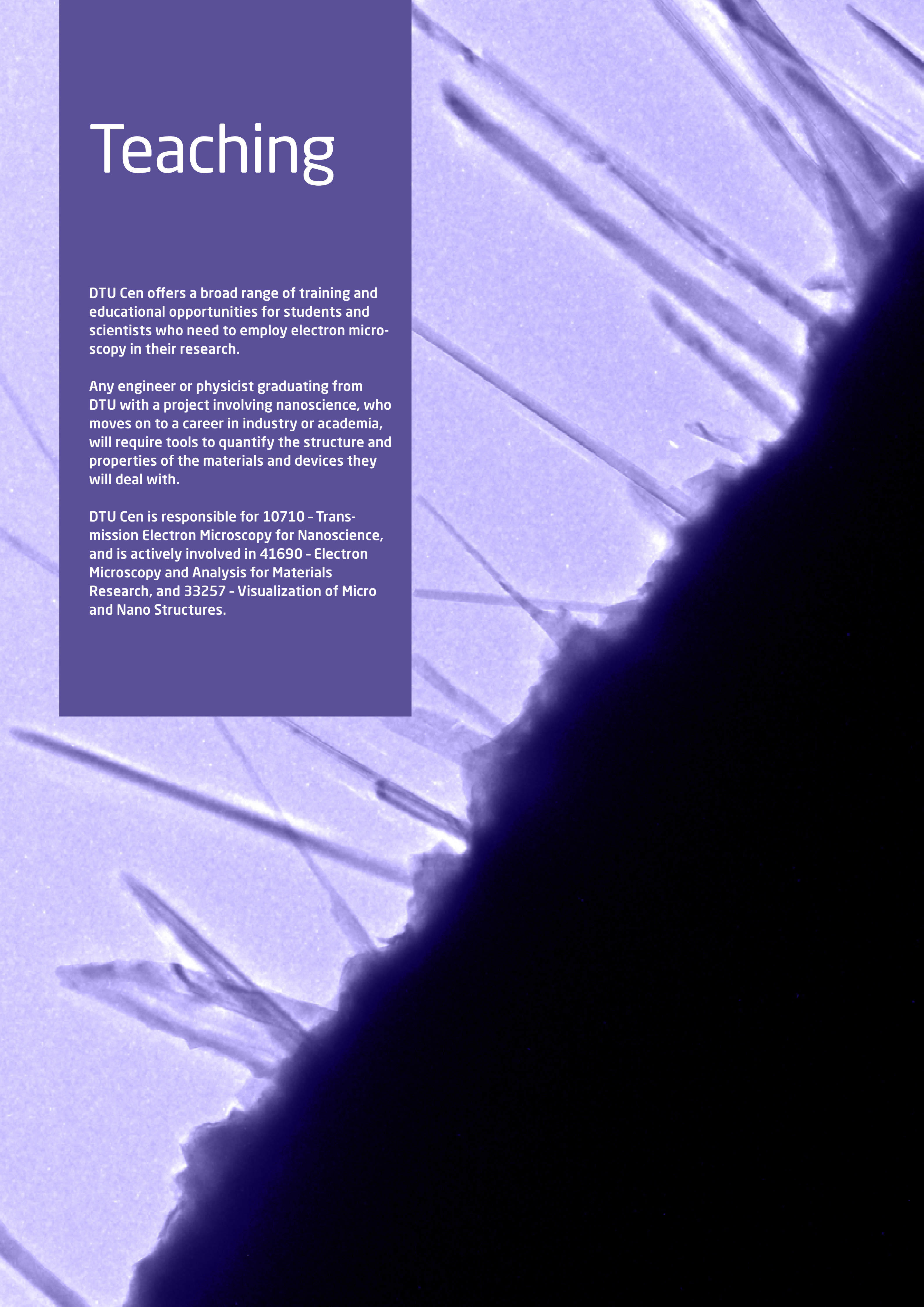
The use of electron microscopy techniques for investigating the location of oxidation in emulsions will be investigated. Environmental SEM (ESEM) is a technique capable of imaging oil-water samples in their natural state without drying out the specimens during observation. ESEM will be used to determine emulsion drop size distributions and to follow coarsening of the droplets with time. Potentially, this technique may reveal details of the structure of the interfacial layer between oil and water. Cryo-SEM can be used to freeze the sample and then fracture it inside the SEM. Freeze-fractured surfaces may be able to reveal variations in the surface microstructures and thicknesses of interfaces around the droplets. Plans are being made to use cryo-TEM to characterize the structure of the interfacial layer.

Teaching

DTU Cen offers a broad range of training and educational opportunities for students and scientists who need to employ electron microscopy in their research.

Any engineer or physicist graduating from DTU with a project involving nanoscience, who moves on to a career in industry or academia, will require tools to quantify the structure and properties of the materials and devices they will deal with.

DTU Cen is responsible for 10710 - Transmission Electron Microscopy for Nanoscience, and is actively involved in 41690 - Electron Microscopy and Analysis for Materials Research, and 33257 - Visualization of Micro and Nano Structures.



10710 – Transmission Electron Microscopy for Nanoscience

PhD course (open to final year MSc), 5 ECTS

Course responsible: Marco Beleggia (DTU Cen)
DTU Cen staff involved: Chris Boothroyd, Jakob B. Wagner.

General course objectives: This course will enable participants to interpret electron micrographs of various types in order to extract quantitative physical information on the materials or structures they are interested in. The course will not train electron microscope operators. The course aims at providing a theoretical understanding of the principles of electron optics, image formation, data acquisition, and image processing as related to the TEM. The course will enable the participants to understand and interpret TEM images, diffraction patterns, and phase contrast images. Problems and examples will be taken from current research in nanoscience, materials science and engineering at DTU and will focus on nanostructured materials. The course participants will be PhD and Master students who need to employ TEM methods in their own research.

41690 – Electron Microscopy and Analysis for Materials Research

PhD course (open to final year MSc students), 5 ECTS points

Course responsible: Andy Horsewell (DTU Mechanical Engineering and DTU Cen).

DTU Cen staff involved: Chris Boothroyd, Jakob B. Wagner, Nicole MacDonald and Berit Wenzell.

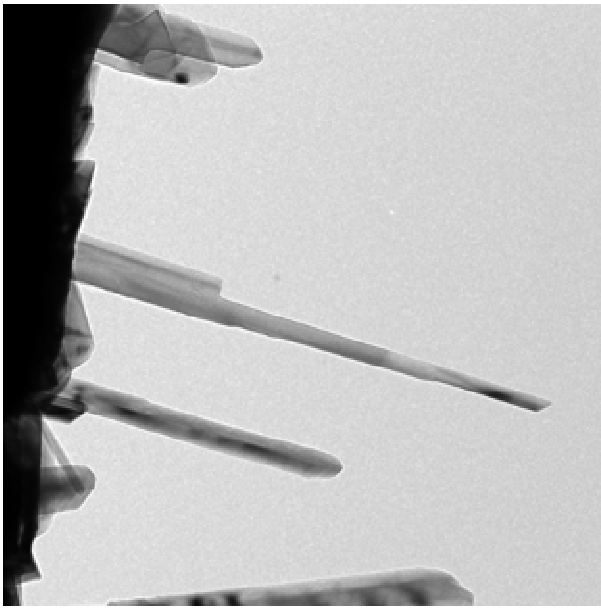
General course objectives: The course aims at providing a basic theoretical understanding of the principles of electron microscopy. The course will enable the participants to understand and interpret images, diffraction patterns and spectra obtained from both scanning and transmission electron microscopes. Specimen types and problems will be taken from current research in Materials Science and Engineering at DTU and will cover metals and alloys, ceramics, polymers, composites and nanomaterials. The course participants will exclusively be PhD and final year Master project students. An active learning approach will be adopted with considerable input from the students' own research projects.

33257 – Visualization of Micro and Nano structures

PhD course (open to final year MSc students), 5 ECTS points

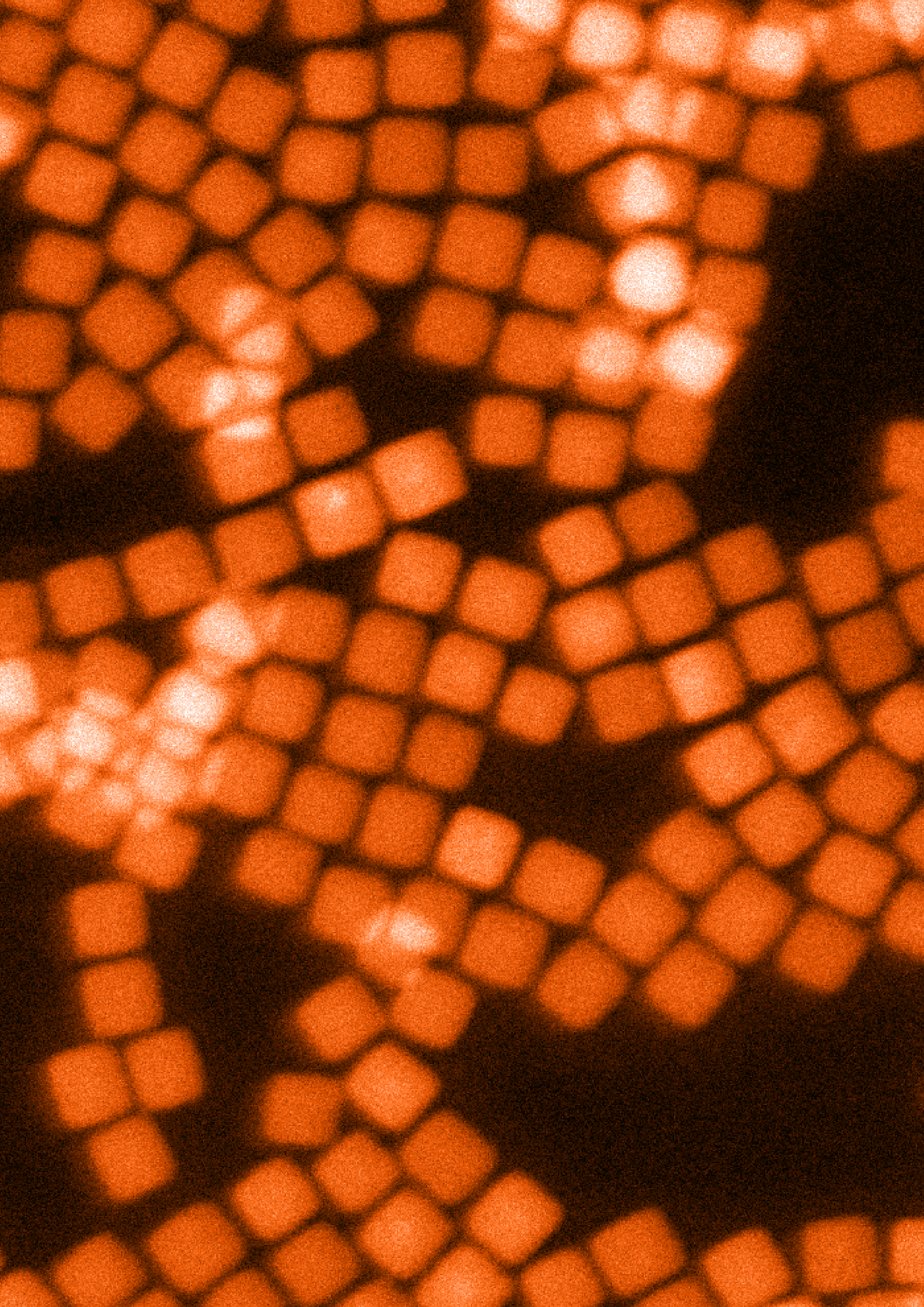
Course responsibles: Rodolphe Marie (DTU Nanotech), Sebatian Horch (DTU Physics) and Jakob B. Wagner (DTU Cen).

Micro and nano structures are central elements in a large range of research and industrial projects and products. The goal of this course is to give provide an overview of methods for visualisation of these structures and to understand the underlying physics. With this knowledge the student will be able to choose the best suited techniques for a given task and to diagnose and further develop these visualisation techniques. Furthermore, the student will be able to interpret the information provided by these techniques. This can be in the form of the shape of the sample (topography) and its material properties.



Training

DTU Cen technical and scientific staff also trains students and scientists in the operation of all instruments available in the facility.

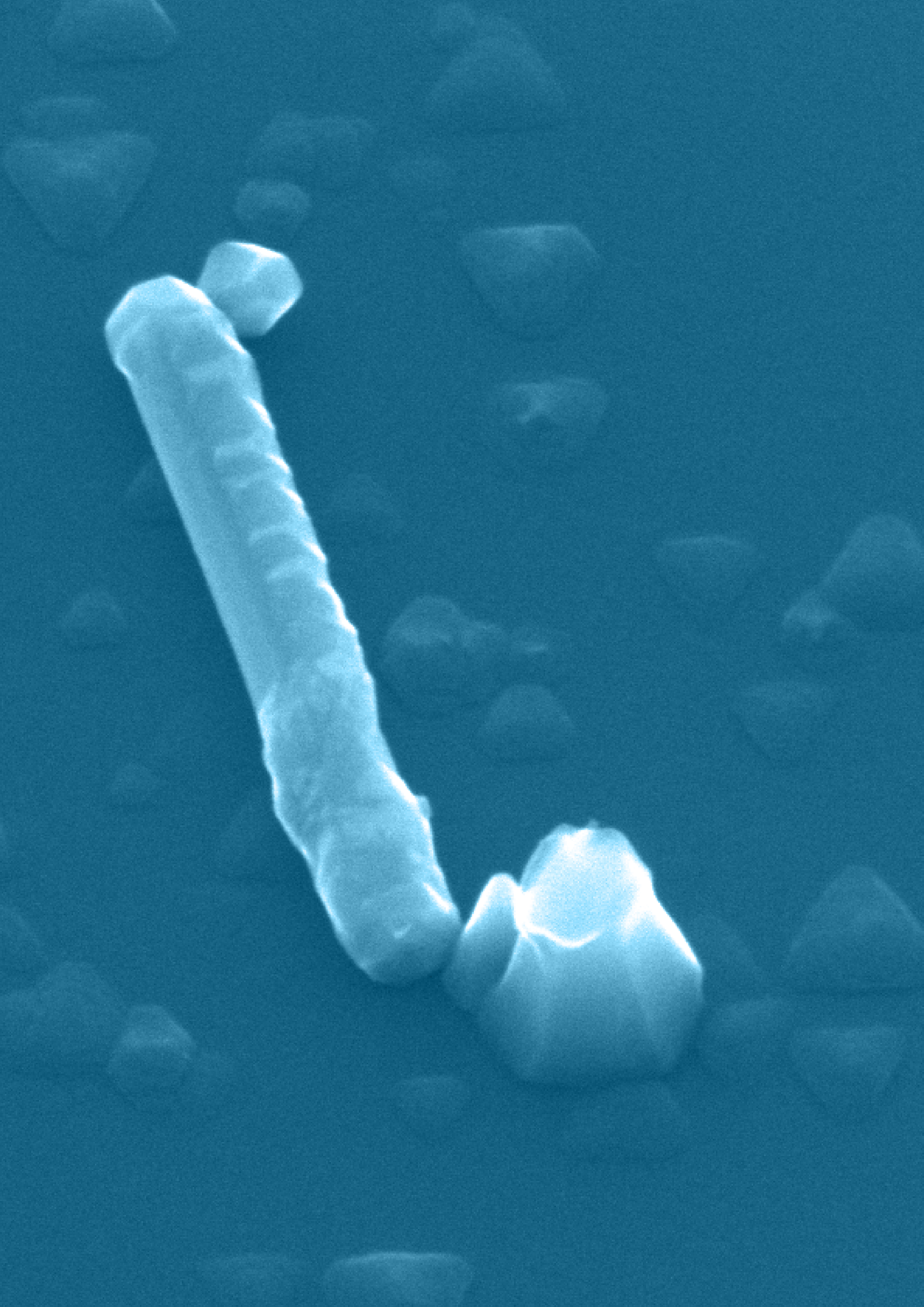


Events

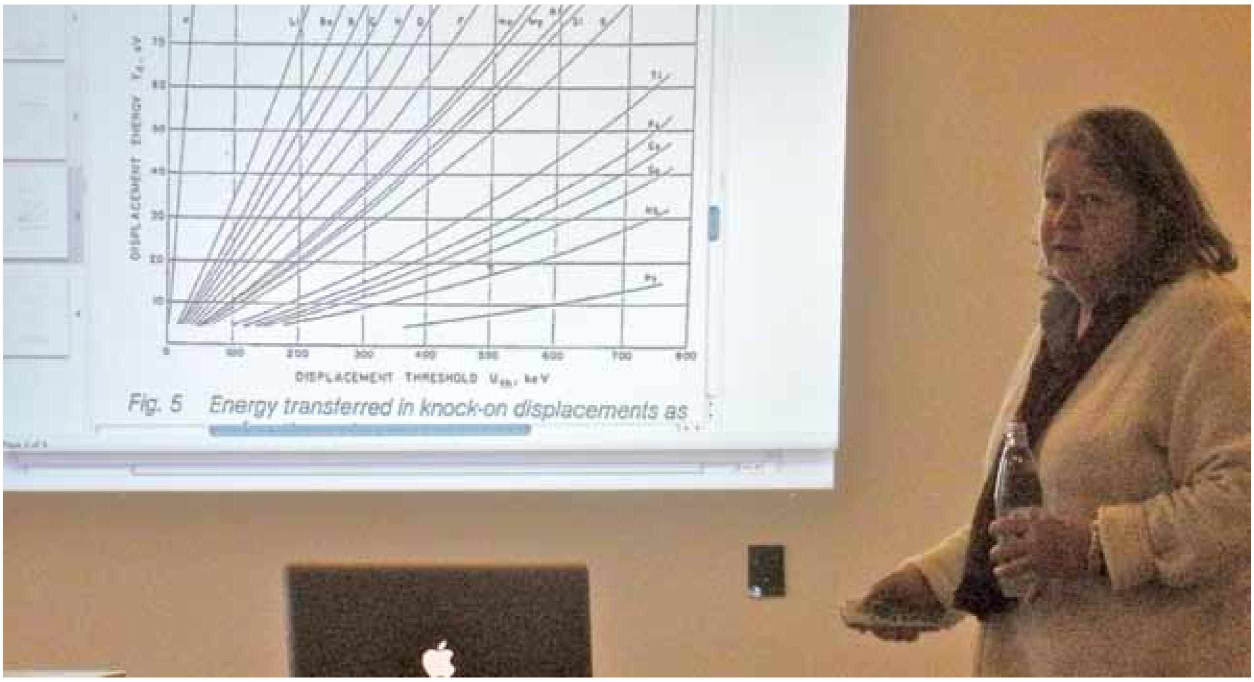
DTU Cen organizes summer schools and workshops in basic and advanced electron microscopy.

From 21 June to 4 July 2009, together with Chalmers University and FEI, DTU Cen organized a "Port to Port" Advanced Electron Microscopy School, at which lectures were given by 20 leading international experts in electron microscopy.

The lectures were given by:
Archie Howie
Joachim Mayer
Max Haider
Harald Rose
Adreas Thust
Juri Barthel
Molly McCartney
David Wall
Stig Helveg
Hannes Lichte
Anrew Bleeloch
Ondrej Krivanek
Crispin Hetherington
Dave McComb
Jean-Paul Morniroli
Paul Midgley
Christian Kuebel
Peter Tiemeijer
Bert Freitag
Eva Olsson
Rafal E. Dunin-Borkowski



Seminars



Lecture on radiation damage by Prof. M. R. McCartney (Arizona State University).

Friday, 9 January, 2009
Recent progress in vector field electron tomography
Marc de Graef
Carnegie Mellon University, Pittsburgh, USA

Wednesday, 29 April, 2009
An overview of the TEAM project in its scientific framework
Uli Damen
National Center for Electron Microscopy, Berkeley, USA

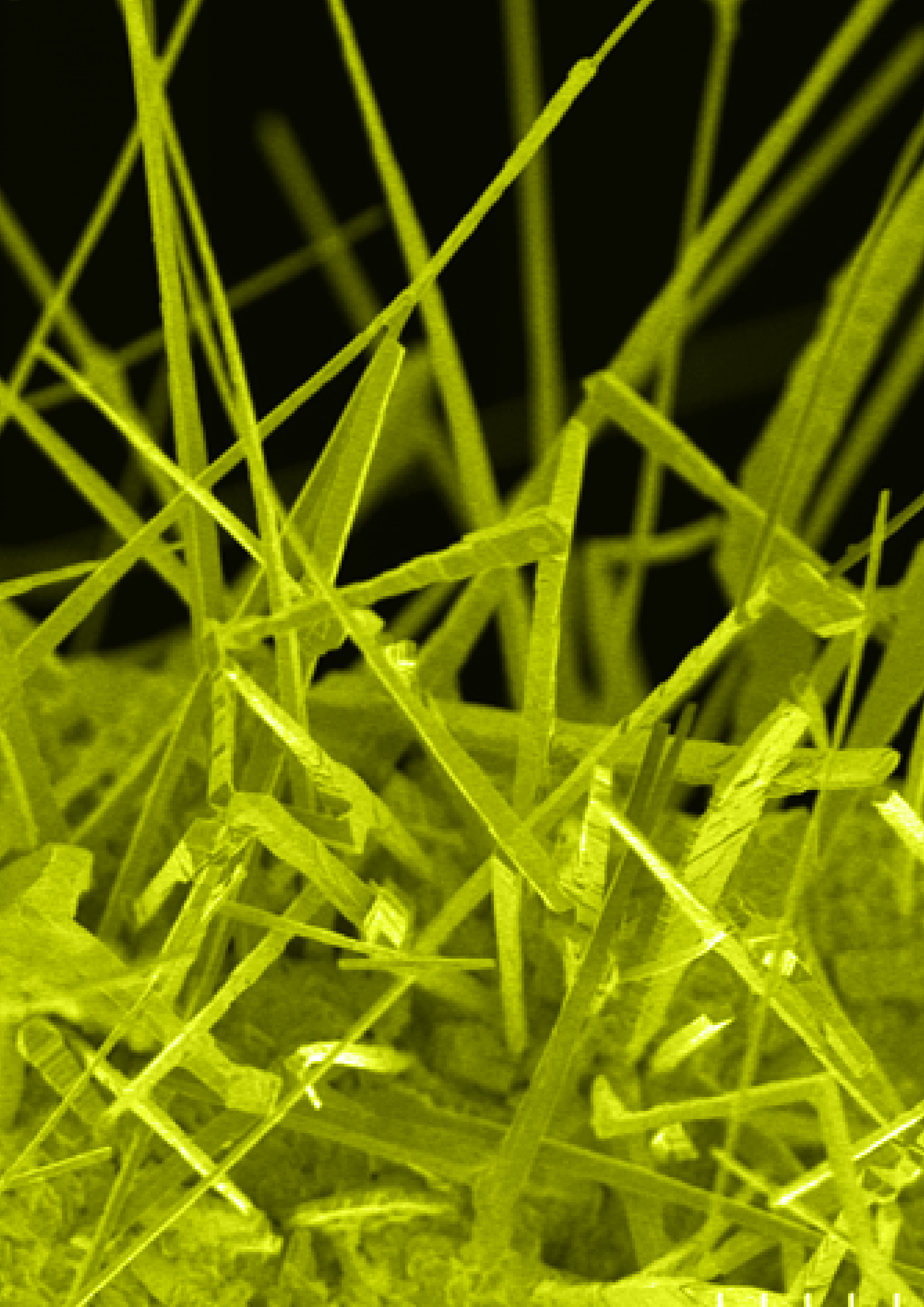
Tuesday, 12 May, 2009
Solid oxide fuel cell (SOFC) redox instability characterized by in situ transmission electron microscopy
Quentin Jeangros
Ecole Polytechnique Federal de Lausanne, Switzerland

Monday, 18 May, 2009
Electron microscopy studies of exchange-biased magnetic thin layers
Andras Kovacs
Oxford University, United Kingdom

Tuesday, 2 June, 2009
What can we learn from inner shell EELS fine structure?
Peter Rez
Department of Physics, Arizona State University, USA

Thursday, 1 October, 2009
Power of dynamic observations in understanding the role of catalyst in the synthesis of carbon nanotubes and nanowires
Renu Sharma
Arizona State University and NIST, USA

Thursday, 19 November, 2009
Electronmicroscopy study of Nb_2Co_7 with layer defects
Wilder Carrillo-Cabrera
Max-Planck-Institute CPtS, Dresden, Germany



Publications & Presentations

1. Refereed and invited journal papers

"Quantitative off-axis electron holography of GaAs p-n junctions prepared by focused ion beam milling" D. Cooper, R. Truche, A. Twitchett-Harrison, R.E. Dunin-Borkowski, and P. Midgley, Journal of Microscopy-Oxford, vol. 233, Jan.2009, pp. 102-113.

"Off-axis electron holographic potential mapping across AlGaAs/AlAs/GaAs heterostructures" S. Chung, S. Johnson, Y. Zhang, D. Smith, and M. McCartney, Journal of Applied Physics, vol. 105, Jan. 2009, p. 014910.

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"Calixarene-stabilised cobalt nanoparticle rings: Self-assembly and collective magnetic properties" A. Wei, S. Tripp, J. Liu, T. Kasama, and R.E. Dunin-Borkowski, Supramolecular Chemistry, vol. 21, 2009, pp. 189-195.

2. Conference papers published in proceedings

"Kinked superconducting vortices in the transmission electron microscope," M. Beleggia and G. Pozzi, Presented at: Aharonov-Bohm and Berry Phase 50/25 Anniversary meeting, Bristol, UK: 2009.

"Aharonov-Bohm phase shift analysis: transmission electron microscopy of multiply connected magnetized particles," M. Beleggia, Y. Millev, and M. De Graef, Presented at: Aharonov-Bohm and Berry Phase 50/25 Anniversary meeting, Bristol, UK: 2009.

"Measuring the magnetic moments of Co nanoparticles rings from their Aharonov-Bohm phase shift", M. Beleggia, T. Kasama, R.E. Dunin-Borkowski, J. Liu, and A. Wei, Presented at: Aharonov-Bohm and Berry Phase 50/25 Anniversary meeting, Bristol, UK: 2009.

"Mineral magnetism of dusty olivine: a potential carrier of pre-accretionary remanence?" N. Church, R. Harrison, T. Kasama, and R.E. Dunin-Borkowski, Eos Trans. AGU, Presented at: American Geophysical Union Fall Meeting, San Francisco, USA: 2009, 90 (52) Fall Meet. Suppl., Abstract GP11A-0746.

"Prospects and challenges for electron holography of doped semiconductors," D. Cooper and R.E. Dunin-Borkowski, Presented at: Microscopy of Semiconducting Materials XVI, Oxford, UK: 2009.

"Theoretical and experimental factors affecting measurements of semiconductor mean inner potentials," R.S. Pennington, J. Mortensen, T. Kasama, C.B. Boothroyd, and R.E. Dunin-Borkowski, Presented at: Microscopy of Semiconducting Materials XVI, Oxford, UK: 2009.

"Atomic resolution imaging of in situ InAs nanowire dissolution at elevated temperature," R.S. Pennington, J.R. Jinschek, J.B. Wagner, C.B. Boothroyd, and R.E. Dunin-Borkowski, Presented at: Microscopy of Semiconducting Materials XVI, Oxford, UK: 2009.

"Biomineralization and magnetism in magnetotactic bacteria," M. Pósfai, R.E. Dunin-Borkowski, T. Kasama, R. Chong, E. Simpson, and P. Buseck, Microscopy and Microanalysis 15 Suppl. 2, Presented at: Microscopy and Microanalysis 2009, Richmond, USA: 2009, pp. 90-91.

"Quantitative electron holography of magnetic fields in nanoscale materials and devices," M. McCartney, T. Kasama, and R.E. Dunin-Borkowski, Presented at: 9th Multinational Congress on Microscopy, Graz, Austria: 2009.

"Interpretation of electron holographic phase images and defocused bright-field images of nanocarbon field emitters," F. Ubaldi, T. Kasama, G. Pozzi, and R.E. Dunin-Borkowski, Presented at: 9th Multinational Congress on Microscopy, Graz, Austria: 2009.

"The titan environmental transmission electron microscope: specifications, considerations and first results," T.W. Hansen, J.B. Wagner, J.R. Jinschek, and R.E. Dunin-Borkowski, Microscopy and Microanalysis Vol. 15 (Suppl. 2), 2009, pp. 714-715. Presented at: Microscopy and Microanalysis, Richmond, 26-30 July 2009

"In-situ reduction and oxidation of nickel from solid oxide fuel cells in a transmission electron microscope", A. Faes, Q. Jeangros, J.B. Wagner, T. W. Hansen, J. Van herle, A. Brisse, R.E. DuninBorkowski and A. Hessler-Wyser. Presented at: the 11th International Symposium on Solid Oxide Fuel Cells (SOFC-XI) at the 216th ECS Meeting, Vienna, Austria, 49 October 2009.

3. Other conference papers, short conference abstracts and posters

"EFTEM and EELS at electron-beam sensitive nano glass-ceramics," Th. Höche, S Bhattacharyya, J.R. Jinschek, C. Bocker, R. Wurth, C. Rüssel, and P.A. van Aken. Poster presented at the EDGE 2009, Banff, Alberta Canada, May 2009.

"Transmission electron microscopy of magnetite at low temperature," T. Kasama, R.J. Harrison, M. Nagao, N.S. Church, J.M. Feinberg, Y Matsui, and R.E. Dunin-Borkowski Presented at Scandem, Iceland, June 8, 2009.

"Solid oxide fuel cell redox instability characterized by in situ transmission electron microscopy," Q. Jeangros, A. Faes, J. Van herle, J.B. Wagner, T.W. Hansen, R.E. Dunin-Borkowski, and A. Hessler-Wyser. Presented at the SFmu, Paris, France, June 22, 2009.

"In-situ reduction and oxidation of nickel from solid oxide fuel cells in a Titan ETEM," A. Faes, Q. Jeangros, A. Hessler-Wyser, J. Van herle, J.B. Wagner, and R.E. Dunin-Borkowski. presented at SOFC-XI, Vienna, Austria, September 30, 2009.

"Occurrence and restoration of the vortex state in nanorings for magnetic phase plate development". M.K. Hari, M. Beleggia, R.M.D. Brydson. Presented at: EMAG 2009, Sheffield, UK, September 2009.

"Kinked superconducting vortices in the transmission electron microscope". M. Beleggia, G. Pozzi. Presented at: Aharonov-Bohm and Berry Phase 50/25 Anniversary meeting, Bristol, UK, December 2009.

"Aharonov-Bohm phase shift analysis: transmission electron microscopy of multiply connected magnetized particles". M. Beleggia, Y. Millev, M. De Graef. Presented at: Aharonov-Bohm and Berry Phase 50/25 Anniversary meeting, Bristol, UK, December 2009.

"Measuring the magnetic moments of Co nanoparticles rings from their Aharonov-Bohm phase shift". M. Beleggia, T. Kasama, R.E. Dunin-Borkowski, J. Liu, A. Wei. Presented at: Aharonov-Bohm and Berry Phase 50/25 Anniversary meeting, Bristol, UK, December 2009.

"Advanced electron micro- and nano-scopy", R.E. Dunin-Borkowski and T. Kasama, Presented at: 38th International School and Conference on the Physics of Semiconductors "Jaszowiec" 2009, Krynica-Zdrój, Poland: 2009.

"Mineral magnetism of dusty olivine: a potential carrier of pre-accretionary remanence?" R. Harrison, S. Lappe, J. Feinberg, S. Russel, G. Bromiley, A. Bastos da Silva Fanta, and R.E. Dunin-Borkowski, Eos Trans. AGU, Presented at: American Geophysical Union Fall Meeting, San Francisco, USA: 2009, 90 (52) Fall Meet. Suppl., Abstract GP34A-06.

"Direct observation of the interaction between elastic and magnetic domain walls below the Verwey transition in magnetite using electron holography", T. Kasama, N. Church, J. Feinberg, R.E. Dunin-Borkowski, and R. Harrison, Presented at: Societe Francaise des Micro-scopies, Paris, France: 2009.

"Large scale synthesis of single crystalline iron oxide magnetic nanorings", F. Luo, L. Heyderman, G. Tzvetkov, J. Raabe, C. Jia, L. Sun, Z. Yan, C. Yan, X. Han, K. Zheng, M. Takano, N. Hayashi, M. Eltschka, M. Kläui, U. Rüdiger, T. Kasama, L. Cervera-Gontard, and R.E. Dunin-Borkowski, Presented at: The International Conference on Magnetism - ICM 2009, Karlsruhe, Germany, 2009.

"The mineralogy of magnetosomes," M. Pósfai, I. Nyírõ-Kósa, D. Csákberényi Nagy, D. Faivre, T. Kasama, and R.E Dunin-Borkowski, Presented at: 11th Scientific Assembly of the International Association of Geomagetism and Aeronomy, Sopron, Hungary, 2009.

"Electron tomography and electron holography of dislocations," A. Ramar, T. Kasama, R. Schaublin, G. Winther, N. Hansen, and R.E. Dunin-Borkowski, Presented at: Electron Microscopy and Multiscale Modelling Conference, Zurich, Switzerland, 2009.

"Catalyst free MBE growth of Mn doped GaAs nanowires on silicon substrates" J. Sadowski, P. Dluzewski, T. Wojciechowski, K. Gas, W. Zaleszczyk, W. Szuszkiewicz, J. Morhange, T. Kasama, and R.E. Dunin-Borkowski, Presented at: 38th International School and Conference on the Physics of Semiconductors "Jaszowiec" 2009, Krynica,-Zdrój, Poland, 2009.

"A study of the nucleation of focused electron beam induced deposits: growth behavior on the nanometer scale," W. van Dorp, J.B. Wagner, T.W. Hansen, R.E. Dunin-Borkowski, and C. Hagen, Presented at: American Vacuum Society 56th International Symposium and Exhibition, San Jose, California, USA, 2009.

4. Invited and plenary talks at conferences and workshops

C.B. Boothroyd, "Measurement of the point-spread function of CCD cameras for HREM" presented at the 26th Annual Conference of the Microscopy Society of Thailand, Chiang Mai, Thailand, 28 Jan, 2009.

C.B. Boothroyd, "Measurement of the point-spread function of CCD cameras for HREM" presented at the Workshop on the electron microscopy of ceramic materials Anadolu University, Eskisehir, Turkey, 12 Oct, 2009.

R.E. Dunin-Borkowski, "Prospects and challenges for electron holography of doped semiconductors". Microscopy of Semiconducting Materials. Oxford, U.K., 17-20 Mar 09.

R.E. Dunin-Borkowski, “Challenges and opportunities for electron holography of magnetic materials”. Workshop on “Understanding Materials through Electron Microscopes: Realising the Potential” Imperial College, London, U.K., 22-24 Apr 09.

R.E. Dunin-Borkowski, “Imaging of nanoparticles using aberration correction and electron tomography”. Workshop on “TEM in Nanoscience”. Oslo, Norway, 7-8 May 2009.

R.E. Dunin-Borkowski, “Calibrations and initial results from the aberration corrected Titans at the Center for Electron Nanoscopy at DTU”. Workshop on “TEM in Nanoscience”. Oslo, Norway, 7-8 May 09.

R.E. Dunin-Borkowski, “Towards quantitative 3D electron holography of nano-structures”. Sino-Danish Workshop on Automated Characterization of Metallic Nanostructures. Beijing, China. 15-16 Jun 09

R.E. Dunin-Borkowski, “Electron tomography: current status, roadblocks and future opportunities”. Workshop on “Characterizing materials damage in four dimensions” Annapolis, U.S.A. 16-19 Aug 09.

R.E. Dunin-Borkowski, “Off axis electron holography of magnetic fields in nanostructured materials and devices”. Ernst Ruska Prize 2009 of the Deutsche Gesellschaft für Elektronenmikroskopie (German Society for Electron Microscopy), jointly with Prof. M R McCartney and Dr T Kasama, to recognize achievements in the field of electron microscopy, awarded at the Microscopy Conference 2009 in Graz, Austria. 30 Aug-4 Sep 09.

R.E. Dunin-Borkowski, “Electron holography of functional materials”. EMAG 2009. Sheffield, U.K. 8-11 Sep 09.

R.E. Dunin-Borkowski, “Off-axis electron holography of magnetic nanoparticles, nanostructures and devices”. 11th International Conference on Advanced Materials. Rio de Janeiro, Brazil. 20-25 Sep 09.

R.E. Dunin-Borkowski, “In situ transmission electron microscopy”. School on the Electron Microscopy of Ceramic Materials (EMCM) Eskisehir, Turkey. 8-13 Oct 09.

R. E. Dunin-Borkowski, “Introduction to (medium resolution) off axis electron holography”. School on the Electron Microscopy of Ceramic Materials (EMCM) Eskisehir, Turkey. 8-13 Oct 09.

R.E. Dunin-Borkowski, “Towards in situ electron microscopy of working devices”. 2nd International Workshop on Remote Electron Microscopy and In Situ Studies, Chalmers University of Technology, Gothenburg, Sweden, 16-18 Nov 09.

T.W. Hansen: “Environmental Transmission Electron Microscopy”, European School of Transmission Electron Microscopy of Nanomaterials TEM-UCA 2009, 13-17 July, Cadiz, Spain.

5. Invited seminars

R.E. Dunin-Borkowski, “Advanced transmission electron microscopy of nanoscale materials and devices”. University of Linz, Austria, 15 Jan 09.

R.E. Dunin-Borkowski, “Aberration corrected imaging and electron tomography of industrial catalyst nanoparticles”.
CIME,EPFL, Lausanne, Switzerland, 2 Jul 09.

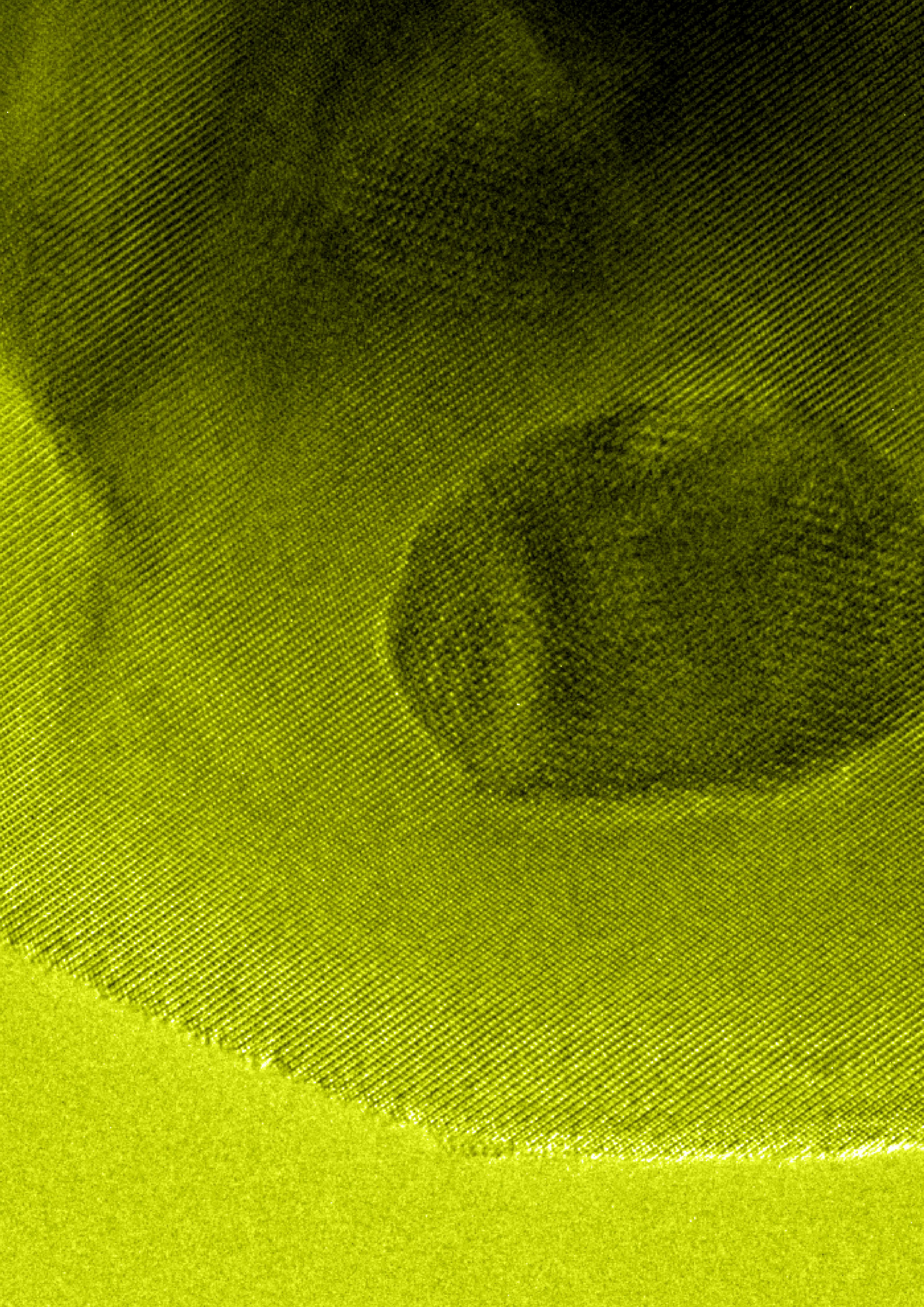
R.E. Dunin-Borkowski, “Center for Electron Nanoscopy at DTU”. Danish Metallurgical Society Meeting, Technical University of Denmark, 21 Oct 09.

R.E. Dunin-Borkowski, “Offaxis electron holography of magnetic nanoparticles, nanostructures and devices”. University of Duisburg-Essen, Germany, 12 Nov 09.

R.E. Dunin-Borkowski, “Quantitative and in situ transmission electron microscopy of functional materials”. Forschungszentrum Jülich, Germany, 24 Nov 09.

R.E. Dunin-Borkowski, “Advanced transmission electron microscopy of nanoscale materials and devices”. Department of Chemical Engineering, Technical University of Denmark, 26 Nov 09.

T. Kasama, “Electron holography of nanostructured magnetic materials.” Invited seminar, Dept. of Materials, Univ. of Oxford, 19 Oct, 2009.



6. Contributed talks at conferences

Boothroyd, Chris B. "Energy dispersive X-ray analysis in TEM scanning transmission electron microscopy (STEM) (lectures at SEM/TEM school)" presented at the School and workshop on the electron microscopy of ceramic materials Anadolu University, Eskisehir, Turkey, October 9, 2009.

Boothroyd, Chris B. "Measurement of the point-spread function of CCD cameras for HREM" presented at MTEC, Thailand, February 3, 2009.

Boothroyd, Chris B. "Measurement of the point-spread function of CCD cameras for HREM" presented at Scandem, Reykjavik, Iceland, June 8, 2009.

Dunin-Borkowski, Rafal E. "Electron holography of magnetic (and electrostatic) fields" Electron Microscopy and Multiscale Modelling Conference, Zurich, Switzerland, 27-30 Oct 09.

Jinschek, Joerg R. "Titan E-TEM." Talk presented at the 2nd International Titan Club meeting, FEI Company (NL), Apr 09.

Wagner, Jakob B. "Center for Electron Nanoscopy - Facility and Research" presented at the 39th Danish Crystallographers Meeting, DTU, Denmark, June 2009.

7. Scientific awards

Ernst Ruska Prize 2009 of the German Society for Electron Microscopy, awarded at the Microscopy Conference 2009 in Graz, Austria to R E Dunin-Borkowski, M R McCartney and T Kasama for their work on: "Electron Holography for Characterisation of Magnetic Fields in sub-100nm-sized Materials and Devices".

Max Hey Medal 2009 of the Mineralogical Society of Great Britain and Ireland was awarded to Takeshi Kasama to recognize research of excellence carried out by young workers in the fields of mineralogy, crystallography, petrology og geomistry.

8. Awards for papers and posters presented at international conferences

30 Aug - 4 Sep 09
Prize at the Microscopy Conference 2009, Graz, Austria for a poster entitled "In situ reduction and oxidation of nickel from solid oxide fuel cells in a Titan ETEM".

9. Conferences and symposia organized or co-organized by Cen staff

21 Jun - 4 Jul 09
Advanced Electron Microscopy School "From Port to Port" Copenhagen and Gothenburg.
Tutorials and demonstrations on electron holography.

19 - 26 Jun 09
38th International School and Conference on the Physics of Semiconductors "Jaszowiec" KrynicaZdrój, Poland.
Tutorial lecture: "Advanced electron micro- and nano-scopy".

17 - 29 May 09
Summer School on Quantitative Electron Microscopy St Aygulf, France. Tutorials and demonstrations on electron holography.

10. Selected tours of DTU Cen

26 Oct 09
Tour for Professors from KAIST (Korean Advanced Institute of Science and Technology).

16 Oct 09
Tour for Divisional Director of Nanyang Technological University.

18 Sep 09
Tour for delegates from 28th Conference of Rectors and Presidents of European Universities of Technology.

24 Aug 09
Tour for Professors from Delft University.

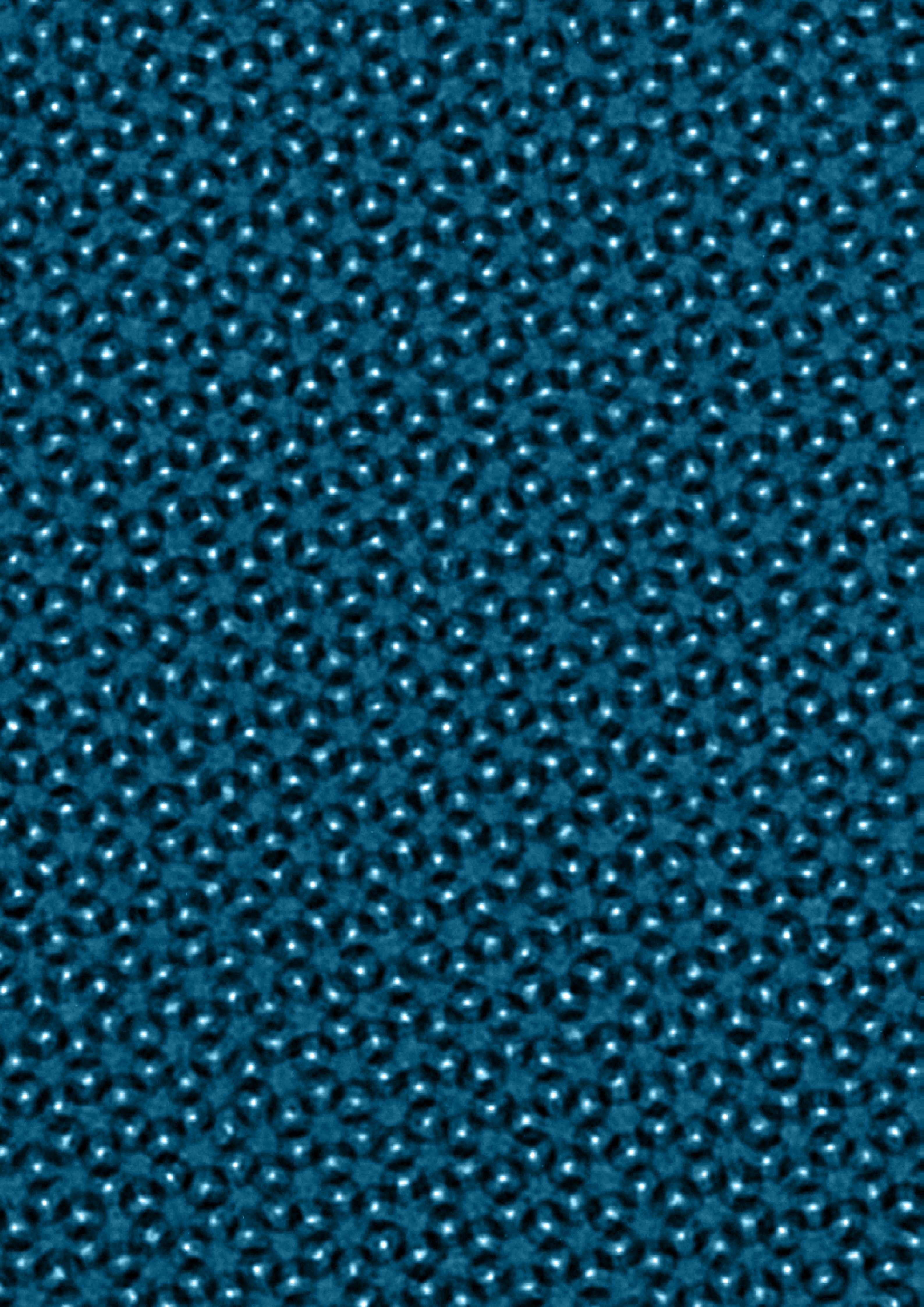
16 Mar 09
Tour for Austrian Federal Ministry of Science.

03 Mar 09
Tour for Henrik Tvarno, Director of the A. P. Møller Foundation.

13 Feb 09
Tour for Sten Scheibye, Chairman of the Board of Governors of DTU.

22 Jan 09
Tour for the President of Pohang University, Korea.

20 Jan 09
Tour for the permanent secretary of the Ministry of Science (VTU).



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Advisory Board

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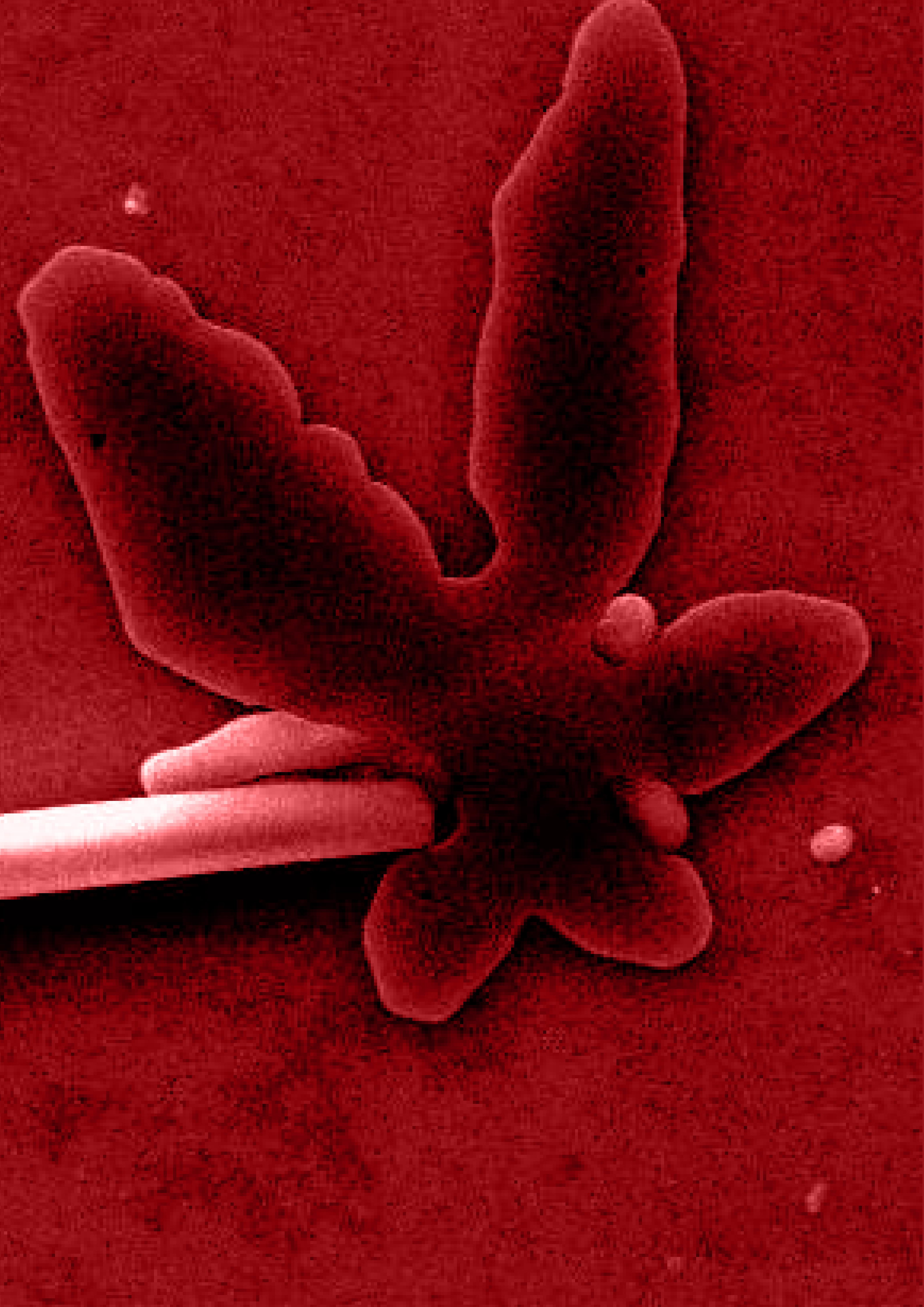


People

Rafal E. Dunin-Borkowski
Director

Rafal Dunin-Borkowski is responsible for directing DTU Cen's operation and activities. His research interests include off-axis holography, electron tomography, quantitative high-resolution electron microscopy, aberration-corrected imaging, image simulation, image processing, instrumentation development and in situ electron microscopy.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 101
2800 Kgs. Lyngby
Denmark

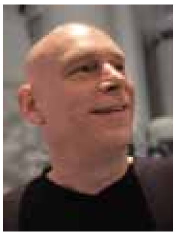
Phone +45 4525 6465
Email rdb@cen.dtu.dk

Anna Rink Baagøe
Center administrator

Anna Rink is responsible for DTU Cen's employees concerning HR, communications and the center's overall administration.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 110
2800 Kgs. Lyngby
Denmark

Phone +45 4525 6474
Email arb@cen.dtu.dk

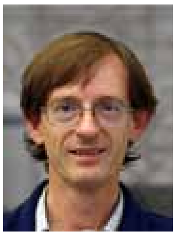


Andy Burrows
Technical manager

Andy Burrows is responsible for ensuring that DTU Cen's instruments are maintained and serviced. His role is to co-ordinate technical support, access to instruments and training, to manage incoming projects and to maintain contact with DTU Cen's equipment manufacturers and other suppliers. He also has primary responsibility for the booking and billing systems at DTU Cen.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 108
2800 Kgs. Lyngby
Denmark

Phone +45 4525 6473
Email andrew.burrows@cen.dtu.dk

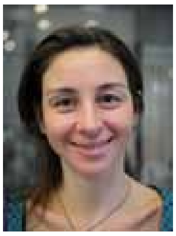


Chris B. Boothroyd
Senior scientist

Chris B. Boothroyd is responsible for the operation of DTU Cen's Analytical Titan TEM as well as the computing facilities in the center. In addition to developing microscopy techniques, particularly in the area of quantitative microscopy, his role at DTU Cen includes teaching at undergraduate and graduate level in conventional and advanced electron microscopy techniques.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 112
2800 Kgs. Lyngby
Denmark

Phone +45 4525 6475
Email chris.boothroyd@cen.dtu.dk

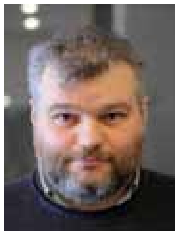


Alice Bastos da Silva Fanta
Researcher

Alice Bastos is responsible for DTU Cen's Helios dual-beam FIB, for the development and application of techniques based on electron backscatter diffraction, for training new users, and for providing assistance in the FIB preparation of TEM specimens. Her research interests include the characterization of electro-deposited materials.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 115
2800 Kgs. Lyngby
Denmark

Phone +45 4525 6468
Email asf@cen.dtu.dk

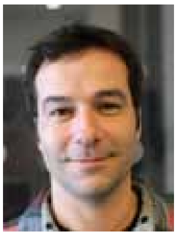


Marco Beleggia
Associate Professor

Marco Beleggia is responsible for DTU Cen's teaching activities. His research interests include electron phase contrast and retrieval techniques, image simulation and processing, microscopy of magnetic materials and the physics of condensed matter at the nanoscale.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 120
2800 Kgs. Lyngby
Denmark

Phone +45 4525 3147
Email mb@cen.dtu.dk



Lionel Cervera Gontard
Researcher

Lionel Cervera Gontard has overall responsibility for the Tecnai TEM at DTU Cen. In this capacity, he ensures that all potential users are adequately trained through practical training sessions. Lionel pursues studies to advance electron tomography and to understand electron beam modification of materials induced by high-energy electrons.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 115
2800 Kgs. Lyngby
Denmark

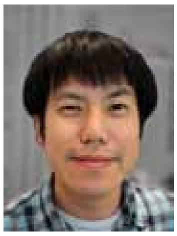
Phone +45 4525 6469
Email lionel.gontard@cen.dtu.dk

Thomas Willum Hansen
Researcher

Thomas Hansen's main research interests are the characterization of catalyst materials using the aberration corrected environmental Titan TEM (ETEM) at DTU Cen. Other tasks include keeping the ETEM running, training new users on DTU Cen is electron microscopes and investigations of other nanostructured materials.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 114
2800 Kgs. Lyngby
Denmark

Phone +45 4525 6476
Email twh@cen.dtu.dk

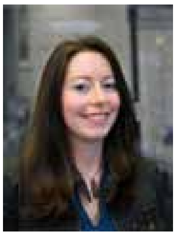


Takeshi Kasama
Senior scientist

Takeshi Kasama's research interests include off-axis electron holography of magnetic and electrostatic fields in nanostructured materials. At DTU Cen, he is responsible for the development and optimization of advanced specimen preparation techniques and specimen holder technologies for the examination of working devices using this technique. He also provides hands-on training, advice and assistance to users of DTU's transmission electron microscopes in conventional and advanced electron microscopy.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 117
2800 Kgs. Lyngby
Denmark

Phone +45 4525 6470
Email tk@cen.dtu.dk.

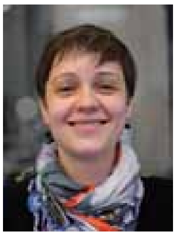


A. Nicole MacDonald
Academic laboratory manager

Nicole MacDonald is the primary user of the Quanta3D FIB SEM, using it to prepare SEM and TEM cross-sections. She also works on the Inspect 'S', Quanta 200 FEG and Helios SEM, collaborating with and training users on all four instruments.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 116
2800 Kgs. Lyngby
Denmark

Phone +45 4525 6477
Email macdonald@cen.dtu.dk

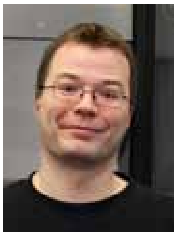


Ramona V. Mateiu
Academic laboratory technician

Ramona Mateiu is the primary user of the Quanta 200 FEG. She also works on the Inspect SEM, Quanta3D FIB SEM and Helios SEM, collaborating with and training users on all four instruments.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 116
2800 Kgs Lyngby
Denmark

Phone +45 4525 6477
Email ramona.matheiu@cen.dtu.dk



Jakob Birkedal Wagner
Senior scientist

Jakob B. Wagner is responsible for DTU Cen's Environmental Titan TEM and X-ray diffractometer. His research interests include high-resolution electron microscopy, energy-filtered imaging and 'in situ' electron microscopy, with a primary emphasis on the characterization and in situ study of semiconductor nanowires and nanoparticle catalysts. He is also involved in DTU Cen's teaching activities.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 114
2800 Kgs. Lyngby
Denmark

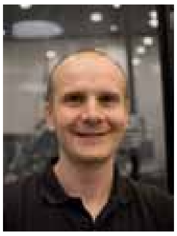
Phone +45 4525 6476
Email jakob.wagner@cen.dtu.dk

Berit Wenzell
Laboratory technician

Berit Wenzell is primarily responsible for X-ray analysis on the three SEMs: energy dispersive spectroscopy (EDS) on the Inspect 'S', the Helios FIB SEM and the Quanta 200 FEG, and wavelength-dispersive spectroscopy on the Inspect 'S'.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 116
2800 Kgs. Lyngby
Denmark

Phone +45 4525 6477
Email bewe@cen.dtu.dk

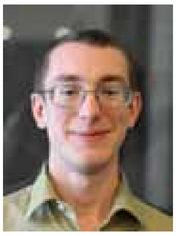


András Kovács
Post. Doc.

András works on the FunDMS - Functionalisation of Diluted Magnetic Semiconductors - project which is a European Union ERC Advanced Research Grant Co-ordinated by Professor Tomasz Dietl (IFPAN, Warsaw, Poland). He carries out high spatial resolution quantitative chemical, structural and magnetic characterization of a wide range of diluted magnetic semiconductors using advanced electron microscopy techniques. He also provides advice and assistance to users in conventional TEM sample preparation methods.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 1172800 Kgs. Lyngby
Denmark

Phone +45 4525 6477
Email macdonald@cen.dtu.dk



Robert S. Pennington
PhD student

Robert Pennington is a PhD student at DTU Cen. His doctoral research is focusing on TEM characterization of nanowires and other nanostructures using a variety of electron microscopy techniques. He uses primarily the Tecnai and the Analytical Titan TEM.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 118
2800 Kgs. Lyngby
Denmark

Phone +45 4525 6468
Email robert.pennington@cen.dtu.dk

Johan Mikael Persson
PhD student

Johan Persson is a PhD student at DTU Cen. His research is primarily on transmission electron microscopy of semiconductor nanowires designed for applications in novel solar cell architectures. The research is funded by a European Commission 7th Framework collaborative project called AMON-RA.

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 118
2800 Kgs. Lyngby
Denmark

Phone +45 4525 6494
Email johmp@cen.dtu.dk

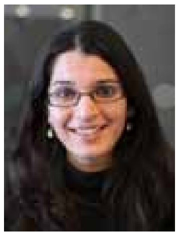
Amuthan Ramar
Ørsted post doctoral fellow

Amuthan Ramar arrived from the Swiss Federal Institute of Technology (EPFL). His research interests are mainly on diffraction contrast imaging, weak beam imaging, high resolution TEM imaging, X-ray microanalysis and scanning electron microscopy of ODS materials. His recent interest is on developing new 'in-situ' techniques in TEM, holog-raphy and tomography of dislocations.

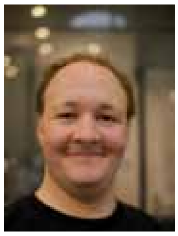
Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307, room 115
2800 Kgs. Lyngby
Denmark

Phone +45 4525 6468
Email aram@cen.dtu.dk

Pradnya Chavan
DTU Cen

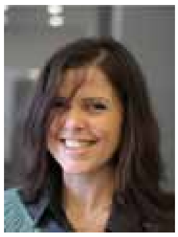


Shima Kadkhodazadeh
Post Doc
DTU Fotonik/
DTU Cen

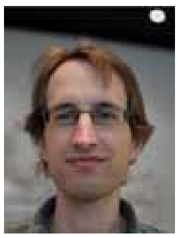


Eric Jensen
PhD student,
DTU Nano/DTU Cen

Louise Helene Søgaard Jensen
PhD student,
DTU Mek/DTU Cen



Alessandra Mosca
Post Doc
DTU Mek/DTU Cen



Lars Schulte
Research Assistant
DTU Mek/DTU Cen

Affiliated Staff

Cathrine Frandsen
Department of Physics

Flemming Bjerg Grumsen
Department of Management Engineering

Andy Horsewell
Department of Management Engineering

Luise Theil Kuhn
Fuel Cells and Solid State Chemistry, Risø DTU

Kristian Mølhave
Department of Micro- and Nano-technology

Kresten Yvind
Department of Fotonik

Former Staff

DTU Cen gratefully acknowledges the contributions of

Anne Marie Rasmussen

Guest Researchers

Crispin Hetherington - Oxford University, United Kingdom

Marc de Graef - Carnegie Mellon University, U.S.A.

Kenta Yoshida - JFCC, Japan

Wyn Williams - Edinburgh University, United Kingdom

Adrian Muxworthy - Imperial College, United Kingdom

Ted Evans - University of Alberta, Canada

Nathan Church - Cambridge University, United Kingdom

Robin Schaublin - Paul Scherrer Institute, Switzerland

Alexandra Porter - Imperial College, United Kingdom

Owen Saxton - Cambridge University, United Kingdom

Willem van Dorp - Delft University of Technology, The Netherlands

Kees Hagen - Delft University of Technology, The Netherlands

Quentin Jeangros - EPFL, Switzerland

Tan Hui Ru - IMRE, Singapore

Joyce Tan Pei Ying - IMRE, Singapore

Molly McCartney - Arizona State University, U.S.A.

Giulio Pozzi - University of Bologna, Italy

Filippo Ubaldi - University of Bologna, Italy

Uli Dahmen - NCEM, Berkeley, U.S.A.

Peter Rez - Arizona State University, U.S.A.

Mathias Wötzel - University of Konstanz, Germany

Renu Sharma - Arizona State University, U.S.A.

John Walmsley - NTNU, Norway

Roya Dehghan - NTNU, Norway

Miriam Varon - Barcelona, Spain

Wilder Carillo-Cabrera - Max-Planck-Institute CPtS, Dresden, Germany

Miroslava Schaffer - SuperSTEM laboratory, United Kingdom

Thomas Weirich - Aachen University, Germany

Center for Electron Nanoscopy
Technical University of Denmark
Fysikvej
Building 307
2800 Kgs. Lyngby
Denmark

Phone +45 4525 6474
www.cen.dtu.dk

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