ANNUAL REPORT 2017

TEM GEMINI CENTRE

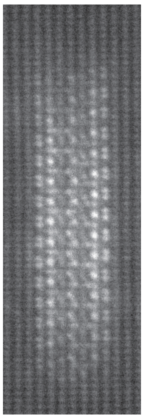
Department of Physics, NTNU; Department of Materials Science and Engineering, NTNU; Materials Physics, Trondheim, SINTEF Materials and Chemistry

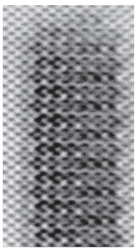
THE TEM GEMINI CENTRE

The TEM (Transmission Electron Microscopy) Gemini Centre was established in 2006, and consisted of professors, postdocs, students and engineers from the Department of Physics (DP), NTNU and researchers from the Material Physics, Trondheim research group in SINTEF Materials and Chemistry (called SINTEF industry from 2018).

In June 2009 the Centre was renominated as a Gemini Centre for a new three years period, and the TEM activities in Department of Materials Science and Engineering (DMSE) at NTNU were included in the Centre. The same constellation was last renominated in November 2015 for another period of 3 years.

The Centre's research groups work within materials physics and materials science, studying a broad range of materials down to the nanometre and atomic level, where the main tool is the transmission electron microscope (TEM). The overall objective of the TEM Gemini Centre is to build and secure a robust scientific environment within TEM with high international profile as a sound basis for growth, not only for the Centre itself, but also for other parts of NTNU and SINTEF and academic and industrial partners. Parallel to and together with this, the large nationally coordinated infrastructure project, NORTEM, has given a national identity to Centre's TEM infrastructure.





Graphic design: Tina Bergh.
Photos of people: Lena Knutli.
Cover image: CBED of GaAs by Maryam Vatanparast and Yu-Tsun Shao.
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THE GEMINI CENTRE CONCEPT

The Gemini collaboration represents a model for strategic research coordination between parallel research groups at SINTEF, NTNU and the University of Oslo. The aim is to develop large-scale technical centres that produce higher quality results collectively than the individual groups would achieve independently. The Gemini Collaboration will enable collaborating groups to grasp new opportunities and bring them to fruition in the form of better value generation and profitability. High-quality technical centres are in great demand internationally from both commercial clients and students. So the Gemini Centres are working with a shared vision:

"Global excellence together".

In order for the collaboration to work, the groups must undertake to adhere to joint strategic processes as the basis for their research planning, technical coordination in connection with large-scale projects, joint fora for concept development and information exchange, the collective presentation of collaborative projects, and shared approaches to investment and the operation of laboratories and equipment. The strategic plan encompasses all aspects of the collaboration model, from teaching and research to commercial research projects, entrepreneurship, recruitment and internationalisation.

CONTENT

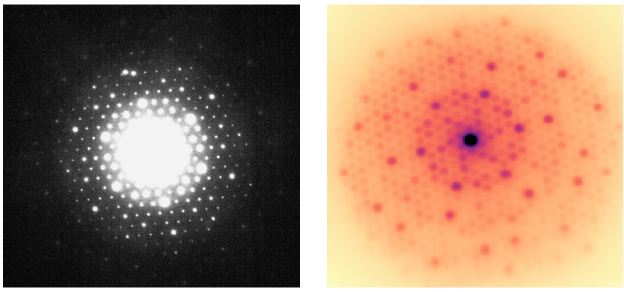
INTRODUCTION

2017 has again been a good year for the TEM Gemini Centre. We have continued to systematically build up routines and extend our competence around the NORTEM Trondheim node instruments that were installed in 2013. It is a big responsibility and long term project to establish and have an effective role within Norway and in the NTNU/SINTEF landscape for such expensive and advanced equipment. It is, therefore very satisfying to see the consistent trend of increasing high levels of use and quality of scientific and educational output achieved during the last years. Furthermore, the JCC (Jeol Competence Centre) agreement with JEOL was renowed in 2017, this secures a close collaboration with JEOL. We will be hosting the 2018 annual ARM user meeting in Trondheim in June.

The total cost model for lab infrastructure introduced by NTNU has been fully implemented for the TEM lab. This required a complex and lengthy debate, ending in a full transfer of ownership to NTNU in 2017. NTNU is now owning and running the infrastructure, with SINTEF as an important user. The next step is to define NORTEMs national role and secure appropriate access for research groups who need TEM. The NORTEM project is a partnership between NTNU, UiO, and SINTEF, financed by the Research Council of Norway and the partners. Looking forward, we are discussing the role of the TEM Gemini Centre in relation to the national NORTEM project and a follow-up project.

The Gemini Centre participates in a range of projects, including national, public, industrial and EU funding ones. The Centre is involved in three long-term SFI projects – Centre of advanced structural studies (CASA), Sustainable innovations for automated manufacturing of multi-material products (SFI-Manufacturing) and Industrial catalysis science and innovation for a competitive and sustainable process industry (iCSI). Furthermore, the TEM Gemini Centre is central in two ongoing KPN projects on aluminium with Norwegian aluminium industry – FICAL and AMPERE. In addition, we have extended the range of research areas and collaborators, which let to an increase in the number of different projects that utilize the infrastructure.

As documented in this report, the Centre had 38 active users, and served over 80 different projects, whose results have contributed to 48 journal publications (plus 8 *in press*) in 2017. Of these, 29 have co-authors from both NTNU and SINTEF. 18 of the publications have international co-authors. They are found in a broad range of journals and cover a spectrum of topics, showing how generic TEM is. In addition, 7 Master candidates were



educated with TEM as a substantial part of their theses in 2017. A positive trend is that the infrastructure gets more NTNU users from outside the Physics department. Three NTNU courses, with a total of approximately 140 students, used the facilities. Jian Min Zuo was awarded NTNU's Lars Onsager professorship for 2017 and stayed in our group for three months. A few of his students could use the facility and concrete collaboration projects were initiated. In connection to his visit we organized an international workshop in electron diffraction in June with 27 participants. The TEM introduction course was organized in September and had 30 participants. We had group meetings with presentations almost every week. Many guided tours for high school students and visitors to the microscopes took place. As seen from the publication list at the end, most members of the TEM group participated in international conferences and meetings in 2017. A very positive trend is that many people in the group now use the scanning precession electron diffraction (SPED) technique for multidimensional data analysis in a broad range of projects. Furthermore, several members contributed to open source code developments within HyperSpy. The Gemini Centre took in the spring initiative to and chaired a proposal to NordForsk for a Nordic University Hub within TEM. Seven partners from Norway, Sweden, Denmark and Finland were in, and a lot of effort was put into making a balanced proposal. Unfortunately, the Hub was not granted, but we hope to use the good ideas generated in future calls. We are also very happy that our group has been asked to participate in the new EU Horizon 2020 initiative ESTEEM3. This is a network of the leading European TEM groups, and the proposal will be submitted in March 2018.

In 2017 a profile in the TEM Gemini Centre, John Walmsley left after 19 years in SINTEF/NTNU. He moved to Cambridge – we want to thank him for many years of hard work and his dedication to the TEM environment in Trondheim!

This annual report gives an overview of people, resources and activities in the group, examples of a few scientific papers, and it lists all publications in the Centre for 2017. For more details, see our home page:
http://www.ntnu.edu/geminicentre/tem
- TEM Gemini Centre management, January 2018.

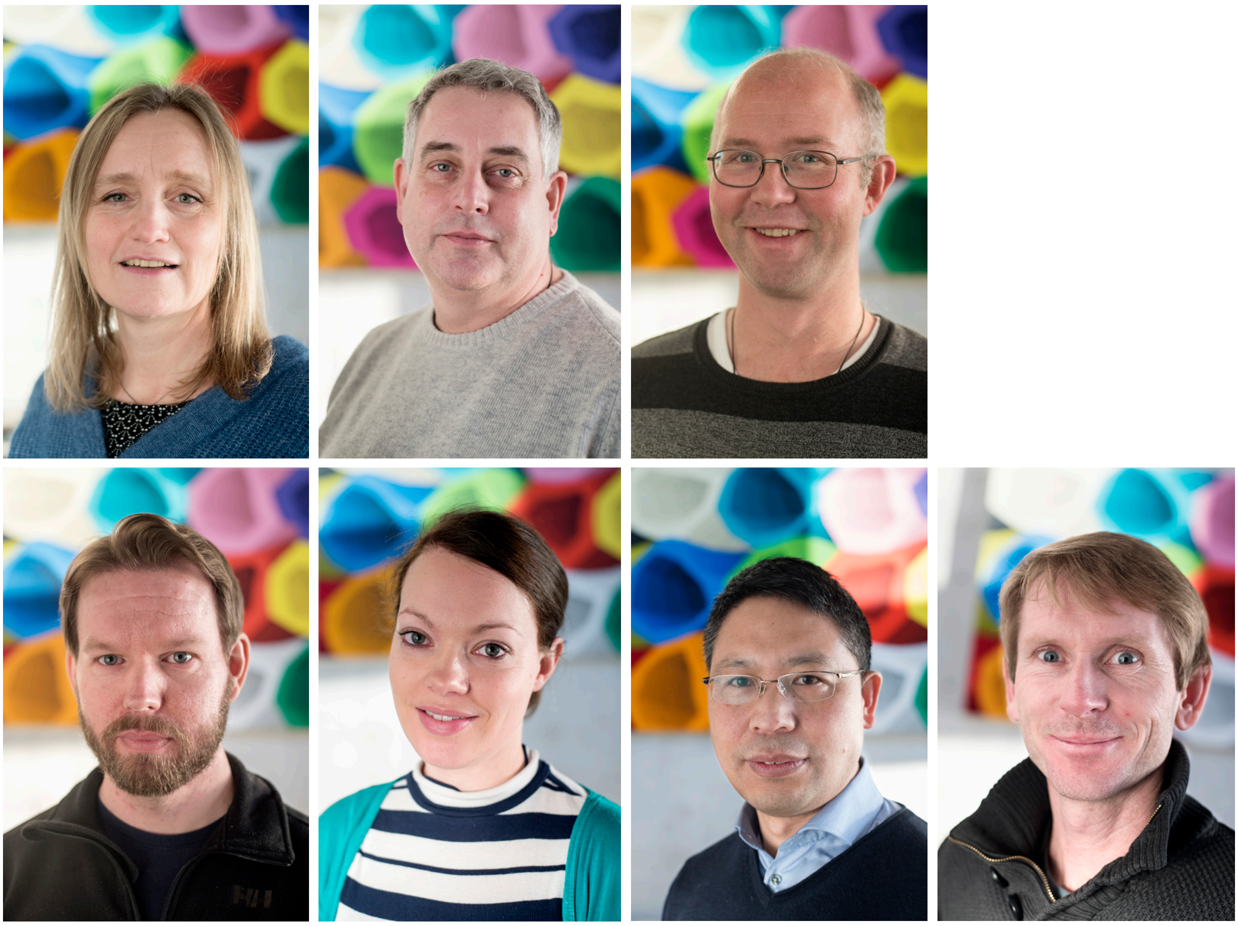
BOARD AND MANAGEMENT

TEM Gemini Centre board:

- **Erik Wahlström**, Department head, Department of Physics, NTNU
- **Ragnar Fagerberg**, Research manager, SINTEF Materials and Chemistry
- **Jostein Mårdalen**, Department head, Department of Materials Science and Engineering, NTNU

Centre management:

- **Randi Holmestad**, Physics, NTNU, Leader
- **Ragnar Fagerberg**, (**John Walmsley** up to March), SINTEF Materials and Chemistry
- **Ton van Helvoort**, Physics, NTNU
- **Bjørn Soleim**, Physics, NTNU
- **Ragnhild Sæterli**, Physics, NTNU
- **Yanjun Li**, Materials Science and Engineering, NTNU
- **Per Erik Vullum,** SINTEF Materials and Chemistry



Randi Holmestad, Professor, Physics, NTNU, leader; Ragnar Fagerberg, Research manager, SINTEF Materials and Chemistry; Ton van Helvoort, Professor, Physics, NTNU; Bjørn Gunnar Soleim, Senior engineer, Physics, NTNU; Ragnhild Sæterli, Senior engineer, Physics, NTNU; Yanjun Li, Professor, Materials Science and Engineering, NTNU; Per Erik Vullum, Research scientist/Associate Professor, SINTEF Materials and Chemistry.

THE NORTEM PROJECT

NORTEM (Norwegian Centre for Transmission Electron Microscopy) is a nationally coordinated large scale infrastructure project with three partners - SINTEF, NTNU and UiO, funded by the Research Council of Norway and the three partners. The budget for new equipment and the re-building in the project was about 75 MNOK for the two geographical nodes, Trondheim and Oslo. We have now been running the facility for close to four years. The support to NORTEM from the Research Council ended in 2016, but the project continues. We have now entered the next phase, and have started on a new proposal, NORTEM II, which will be due in October 2018. This will include upgrading and new investments in both nodes.

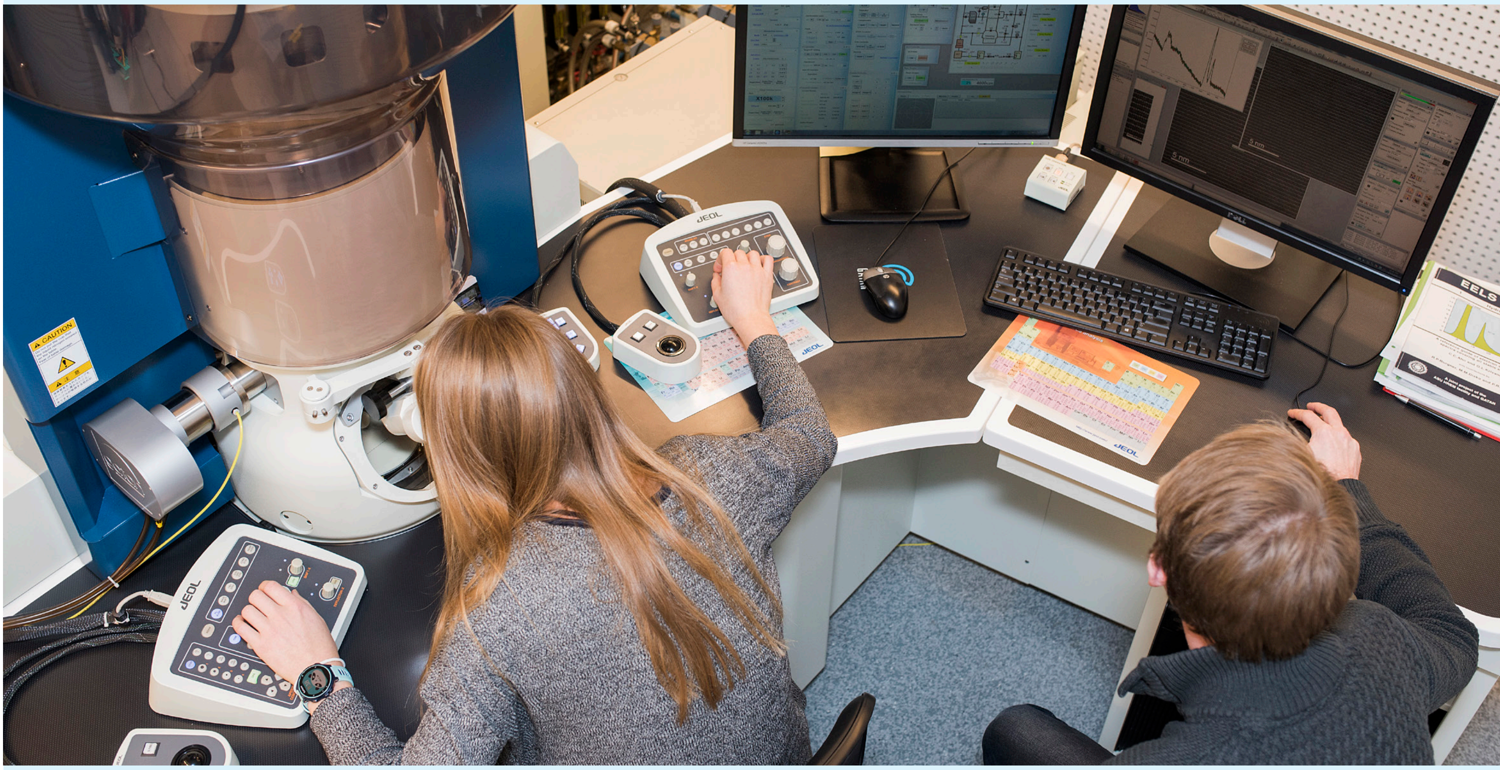
The vision of NORTEM is to be "A world-class TEM facility providing access to expertise and state-of-the-art infrastructure for fundamental and applied research within the physical sciences in Norway". Besides being a top research TEM lab, the infrastructure provides access to TEM for a broader user environment, addressing fundamental and applied research topics in physics, chemistry, materials science and geology.

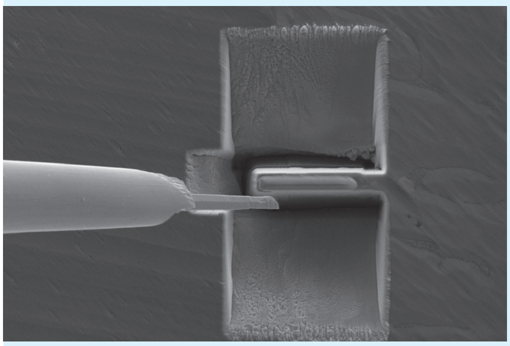
The combination of a research lab and a user facility requires a sustainable running model, and the TEM Gemini Centre has spent a considerable amount of effort during the last years to establish a sound running model for the infrastructure. This has now ended, NTNU owns and runs the infrastructure according to the NTNU model for 'leiested'. Further work is focused on securing the required resources for doing TEM are incorporated in new research applications written by user groups and to make sure that the correct level of use and access is tailored to the actual TEM needs; training of PhD students, close scientific collaboration with experienced users or use of operators in the Centre.

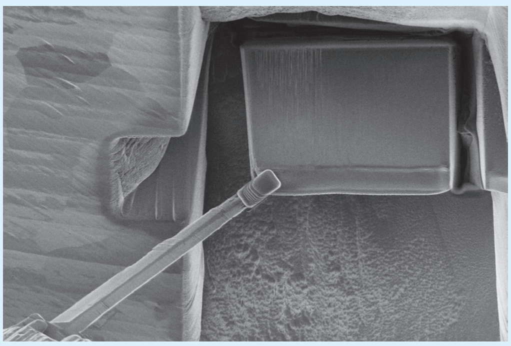
Attention has been paid to addressing the challenge of establishing and getting the best out of the huge investment. The Trondheim node NORTEM facility has two senior engineers, Bjørn Soleim and Ragnhild Sæterli supporting maintenance, training, competence and techniques. We have a high uptime and a number of operators from outside the host institution. John Walmsley (up to March 2017) and Per Erik Vullum have been working as adjunct (associate) professors, which particularly contributes to developing interaction between NTNU and SINTEF.

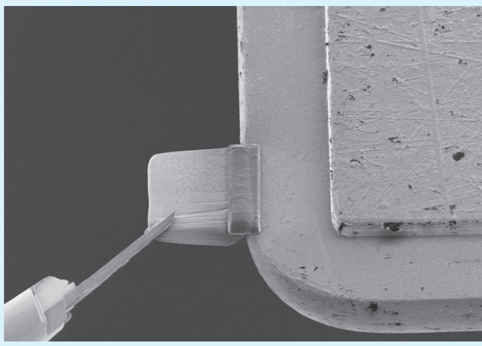
For more information on NORTEM see the webpages: www.nortem.no

INSTRUMENTATION









THE TEM INSTRUMENTS IN TRONDHEIM

The TEM Gemini Centre has five TEMs: three new ones, installed as part of the NORTEM project in 2013 (a JEM-2100 LaB_6, a JEM-2100F and a double corrected JEM-ARM200F). In addition, there are two older TEMs: a Philips CM30 and a JEOL JEM-2010.

JEOL double corrected JEM-ARM200F (coldFEG)

This is one of the most advanced TEMs currently in Europe. The stable coldFEG with both probe and image spherical aberration correction and the most advanced EDX and EELS systems allow unique studies at the atomic scale. The microscope is placed in a custom designed room with water cooled walls and field cancellation.

The ARM features:

- Cold field emission gun with energy spread of 0.3 eV
- Cs-probe corrector
- Cs-image corrector
- Centurio SDD EDX (solid angle 0.98 sr)
- Quantum GIF with DualEELS
- 2k Orius CCD (side-mounted) and 2k UltraScan CCD (bottom mounted)
- Stable 5-axis gonio with piezo control in x, y and z-directions
- Detectors for BF, ABF, ADF and HAADF STEM
- Aligned at 80 and 200 kV

Foto: Ole Morten Melgård

JEOL JEM-2100F

This FEG TEM is optimized for all-round advanced materials studies with a special focus on precession diffraction, orientation mapping and tomography.

The 2100F features:

- 200 kV Schottky field emission gun (energy spread 0.7 eV)
- Gatan 2k UltraScan CCD (bottom mounted)
- Scanning option with BF and HAADF detector
- Oxford X-Max 80 SDD EDX (solid angle 0.23 sr)
- ASTAR Nanomegas orientation mapping and precession diffraction system (SPED)
- Gatan TEM/STEM tomography

JEOL JEM-2100

The 2100 LaB_6 is the workhorse for routine TEM studies, configured for easy access and a broad user group. This is the instrument new users are trained on. The set-up is optimized for conventional TEM techniques as BF/DF-TEM and SAED.

The 2100 features:

- Gatan 2k Orius CCD (side mounted)
- Scanning option with BF and HAADF detector
- Scanning option with BF and HAADF detector (DigiScan)
- GIF system with 2k CCD
- Oxford X-Max 80 SDD EDS (solid angle 0.23 sr)

Microscopes in the TEM Gemini Centre

Instrument	Location	Configuration	Installed
Philips CM30	DP, Natural Science building	LaB_6, 300 kV, PEELS, EDS, 1k camera	1989
JEOL 2010	DMSE, Alfred Getz vei 2	LaB_6, 200 kV, GIF, EDS, 2k camera	1993
JEOL 2100	DP, Chemistry building I	LaB_6, 200 kV, STEM, GIF, EDS, Orius camera	2013
JEOL ARM-200F	DP, Chemistry building I	Cold FEG,image and probe corrected, Quantum GIF, Centurio EDS, CCD cameras	2013
JEOL 2100F	DP, Chemistry building I	FEG, 200 kV, EDS, 2k CCD camera, ASTAR, tomography aquisition software	2013

SPECIMEN HOLDERS

Each TEM has its own set of single and double tilt (±30 degrees) holders. A broad range of additional holders is available for use on all three microscopes. This includes back-up double tilt holders, a cold stage holder, a conventional heating holder, an environmental cell holder, a transfer holder, several tomography holders and two tilt-rotation holders. In 2017 we invested in a biasing upgrade for our MEMS based heating holder to increase the scope of functionality the center can offer. In addition, we made good progress developing routines for preparing TEM lamella of metal and ceramic materials on the MEMS chip for in-situ TEM studies.

SPECIMEN PREPARATION

Given the fine TEM instruments, specimen quality is often the limiting factor. The Gemini Centre has well equipped specimen preparation facilities at both DP and DMSE, reflecting the broad range of materials studied. The Centre has different types of dimplers, saws, ultrasonic cutters and other tools for TEM specimen preparation of metal and ceramic cross sectional specimens.

The Centre has three Gatan PIPS instruments, including a PIPS II, to make more high-quality and reproducible specimens. A routine has been developed to polish focused ion beam (FIB)-made TEM lamellas to obtain the highest specimen quality and best possible TEM results. Increasingly more TEM projects utilize the FIB at NTNU NanoLab with lift-out option for site-specific TEM specimen preparation. Effective access to this technique is essential to the activities of the TEM Gemini Centre. Nanolab has in 2016 acquired a second FIB (operative February 2017). The increase in capacity will make this technique more accessible.

The electropolisher at DP has been essential in producing high quality Al TEM specimens. A semi-automatic tripod polishing set-up is available for large area preparation of hard materials. For soft materials, such as polymers, ultramicrotomy is an essential technique that is also used for preparation of catalysts, surface structures and nanoparticles. We invested in a new ultramicrotome at the end of 2016, which became operative February 2017.

SUPPORTING FACILITIES

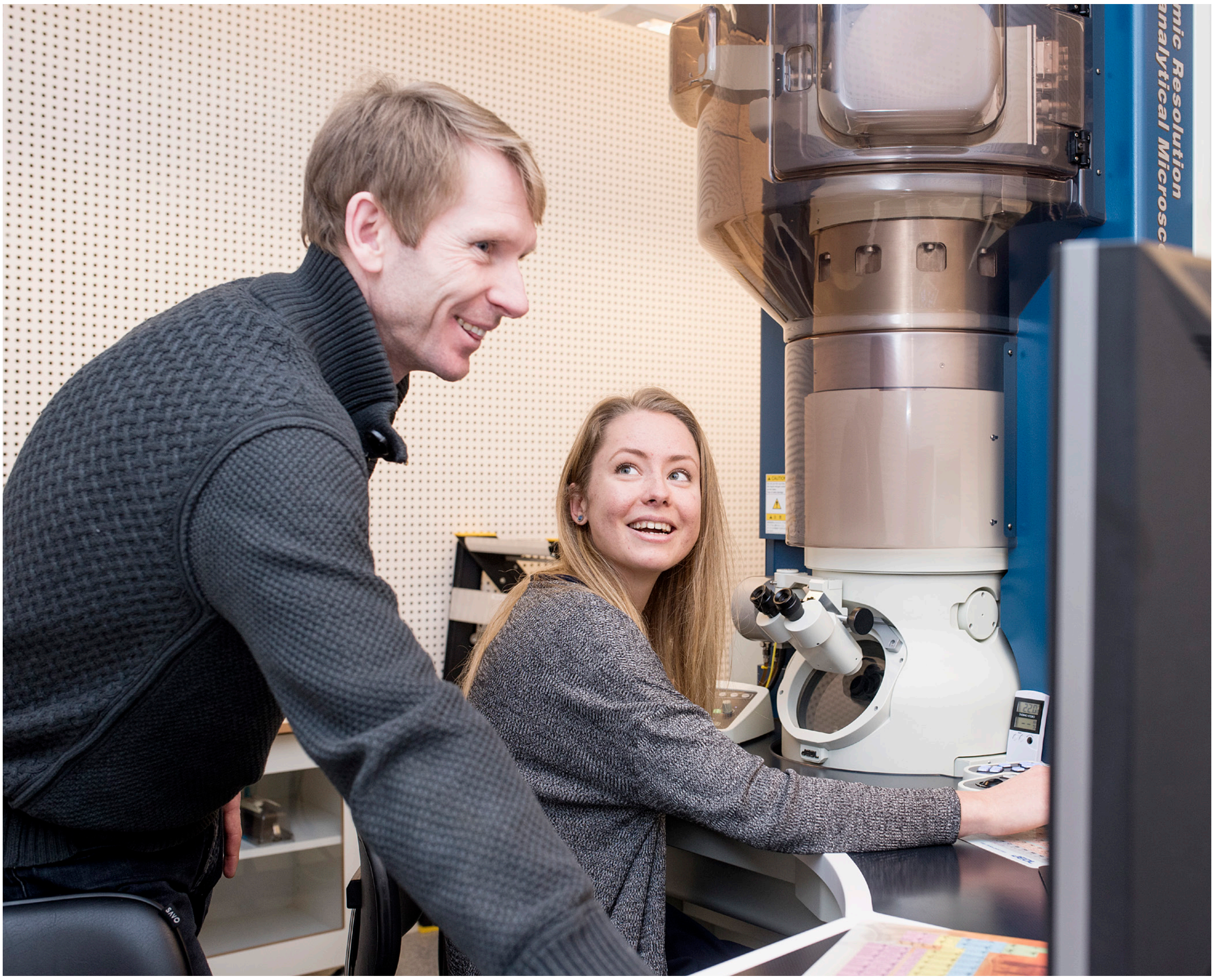
With the aberration corrected microscope, the cleanness requirements of the specimen and the holders increase. We have a dedicated room close to the microscope with general equipment such as a plasma cleaner, ozone cleaner, a stereomicroscope, user specimen storage and special holders that are used on all three TEMs. In addition, there is a data transfer room with additional facilities as a printer and a support PC with the most crucial software packages. The room has a sofa and tea/coffee machine for socializing and efficient breaks during long running sessions. The dedicated TEM computer room has five machines, some of which can be remotely accessed. All acquisition software is accessible via offline licenses in the computer room.

The specimen preparation room and the computer room are located in Realfagbygget in B4, where most Gemini Centre members have their offices. For completeness, the Centre has a darkroom for use of negatives and access to an image plate reader.

USER STATISTICS IN 2017

The total registered used time for the three instruments in 2017 was 4080 hours, including 223 non-paid hours used for testing, competence development, demonstrations and guided tours. Of the 3857 paid hours, the use by NTNU corresponds to 78 % and by SINTEF 22 %. NTNUs use is divided over six departments, where the main use is from Department of Physics (62 % of NTNUs paid hours). About 80 different projects used TEM.

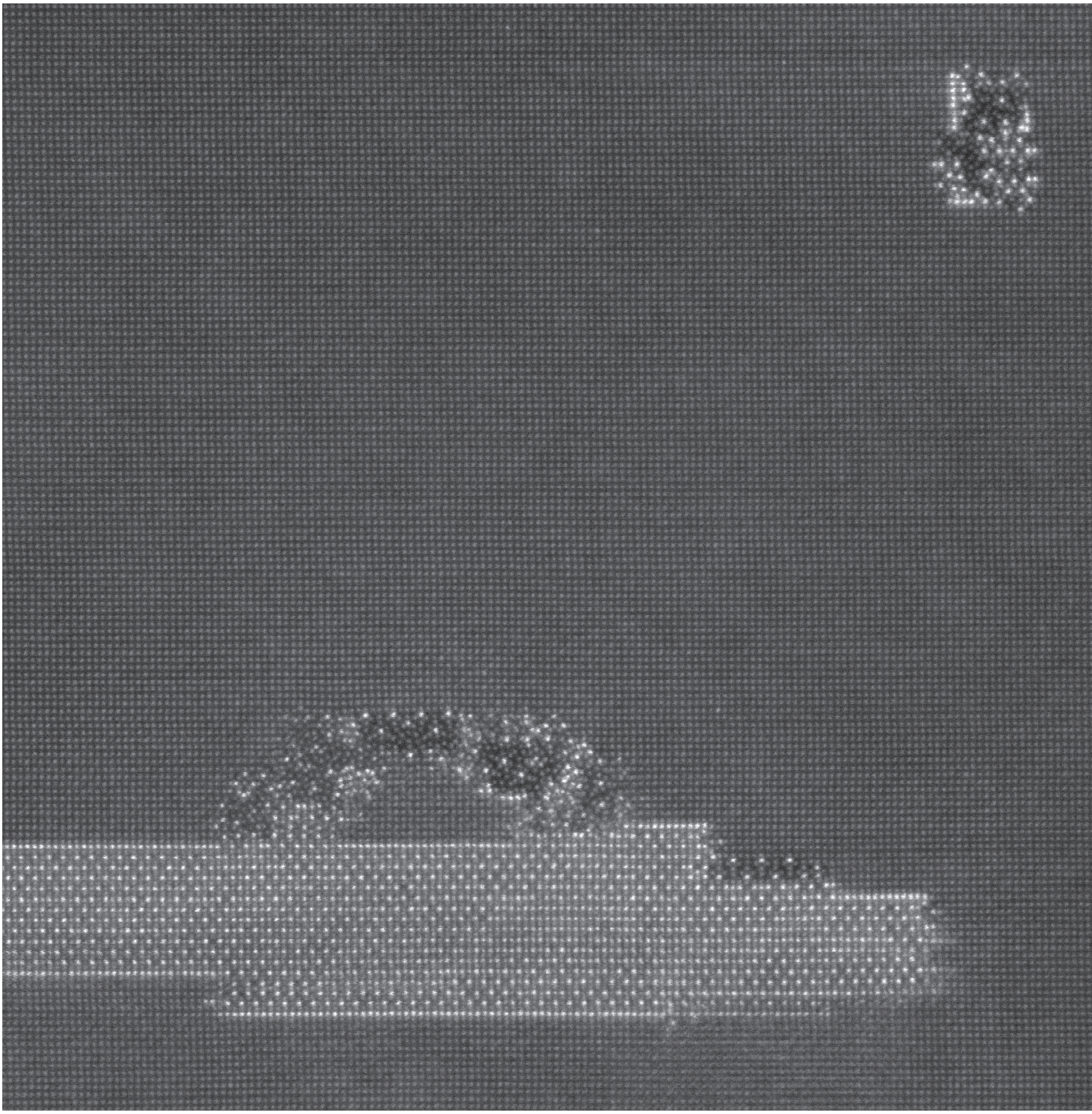
The infrastructure had 38 hands-on users in 2017, where 5 were based at SINTEF, 15 were PhD candidates and 10 Master students. The use from outside the Physics department has experienced an increase. 8 PhD candidates from other departments used the microscopes within the infrastructure in 2017.



Microscope use in hours	ARM-200F	2100F	2100	SUM
SINTEF	495	295	63	853
NTNU – Physics	302	883	689	1874
NTNU – Other departments	148	139	511	798
NTNU – Visitors from abroad	50	70	54	174
NTNU – Teaching lab	21	106	20	147
External	0	0	11	11
NTNU – Set-up/testing/training/demonstrations	57	93	73	223
Total use	**1073**	**1586**	**1421**	**4080**

ACTIVITIES

RESEARCH AND EVENTS



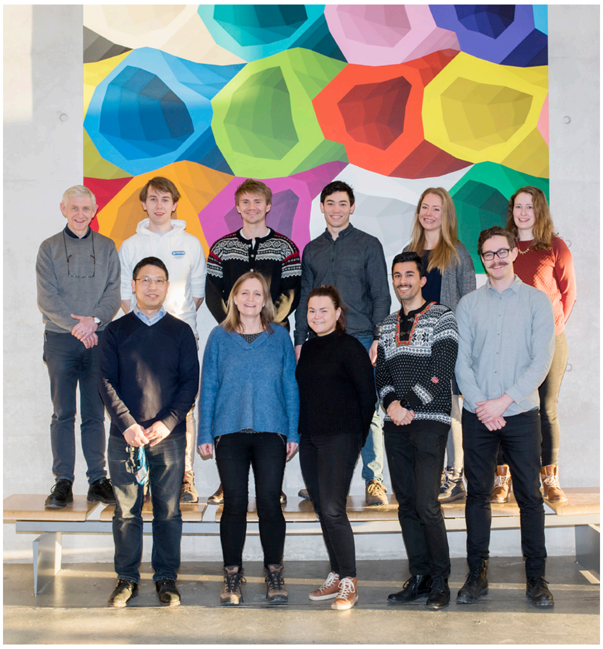
FOCUS AREAS

TEM is a powerful technique for fundamental and applied research in the physical sciences, in different fields from geology, metallurgy and semiconductor industry to fundamental chemistry and physics. NORTEM has identified four focus areas, which have been important for the TEM Gemini Centre activities since the Centre was formed. Within these areas we see potential for further growth and tackling unsolved issues. The focus areas are light metals, catalysis, energy materials and nanotechnology. TEM plays an important role in these research areas, which will be strategically important for Norway also in the future. The TEM Gemini Centre had activities in all these four areas in 2017. The next sections describe these activities. Activities in aluminium alloy research are the largest.

ALUMINIUM ACTIVITIES – LIGHT METALS

The study of aluminium alloys using TEM has been a pillar in the Trondheim TEM environment for many years. Several projects have ended during the last years – these are the Nucleation control (KK) project, the sustainable aluminium surface applications (SALSA) project, the eVITA Multiscale modelling in Aluminium (MultimodAl) project, the competence project MoReAl, the bilateral competence building project with Japan, the FRINATEK ('Fundamental investigations of precipitation in the solid state with focus on Al-based alloys') and finally the RolEx ('Smart 6xxx Alloy Development for Rolling and Extrusion') project. All these projects have been jointly between NTNU and SINTEF, and supported by the Research Council of Norway. In addition, many of them were supported by Hydro Aluminium. Fortunately, we have lately secured the funding for the years to come with new projects on aluminium alloys.

Two of the SFI Centres the Gemini Centre is involved in, are connected to aluminium. In the CASA project, headed by Magnus Langseth at the Mechanical Engineering department in the NTNU Engineering Faculty, we are involved in the "lowest scale" of the multiscale activities, including TEM and atomistic calculations of precipitates, grain boundaries, precipitation free zones and interactions between them and dislocations in deformed, mostly industrial Al alloys. PhD student Emil Christiansen is working on TEM studies of deformed aluminium alloys in this project. Jonas Frafjord is hired to work on the modelling side on the lower scale. He is doing density functional theory (DFT) in combination with other higher scale methods to explore dislocation movement in Al alloys. SFI CASA made in 2017 a promotion video – see https://www.youtube.com/watch?v=mQXCU9uNLUI where TEM on aluminium has a central part.



People working with alumimium and TEM at NTNU.

In the Manufacturing SFI project, headed by Sverre Guldbrandsen-Dahl from SINTEF Raufoss Manufacturing, joining of aluminium with other materials in multi-material products is a central topic, and Tina Bergh studies the microstructure of joints between aluminium and steels. Tina has studied different bonding methods with conventional and advanced TEM techniques.

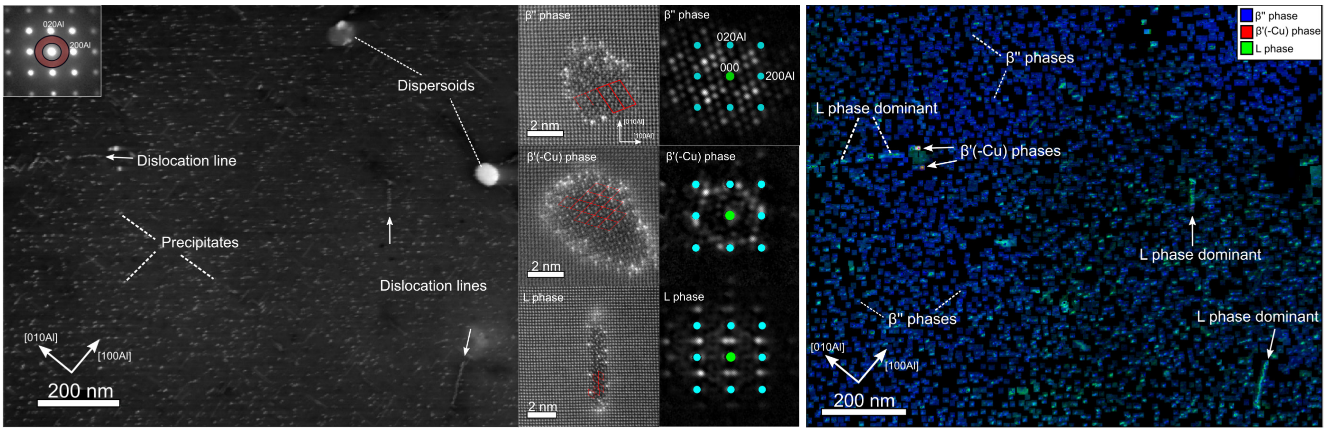
Two competence projects involving NTNU and SINTEF on aluminium research are ongoing. One is the project 'Fundamentals of intergranular corrosion in aluminium alloys' (FICAL) where Randi Holmestad took over after John Walmsley the project leader. FICAL is a 5 years project that has the objective of establishing new fundamental understanding of the mechanisms of intergranular corrosion (IGC) susceptibility. Industrial funding is provided by a consortium of four aluminium companies; Hydro Aluminium, Benteler, Gränges and Steertec. These companies represent the entire value chain, from alloy production to component manufacturing. The mechanisms of IGC are studied at the nanometre-scale utilizing advanced laboratory infrastructure, especially TEM, where NTNU and SINTEF establish the detailed connections between IGC, composition and thermal history. Modelling tools are developed to predict how the alloy microstructure and chemistry develop during processing and relate this to the IGC mechanisms that are observed in the experimental work. It is essential to optimize corrosion resistance with mechanical and other alloy properties for best alloy design and performance. Adrian Lervik is working as a PhD student in the FICAL project, and focuses now particularly on TEM technique development and data-processing to obtain a quantitative understanding of nanoscale structure and chemistry around grain boundaries. He has started to study stress corrosion cracking in 7xxx alloys from Benteler in detail.

The second competence project with aluminium industry is the 'Aluminium alloys with mechanical properties and electrical conductivity at elevated temperatures' (AMPERE), with Knut Marthinsen as a project leader. Here, Al alloys will be studied for several combined properties at elevated temperature (100°C and above), for example, the demand for a combination of high strength and high conductivity without degrading other properties such as fatigue resistance. Hydro Aluminium, Gränges, Nexans and Neuman Raufoss are partners. The project aims at providing new advances in experimental technologies, experimental databases and a set of modelling tools for combinations of aluminium properties. Jonas Kristoffer Sunde started as a PhD student on this project in August 2016, and has been studying the effect of very small Cu additions to the 6082 alloy by combining advanced TEM techniques, such as scanning precession electron diffraction (SPED) and HAADF-STEM.

NAPIC (National aluminium product innovation Centre) was established in 2017, and Håkon Wiik Ånes is hired as a PhD student in this centre to study nucleation of recrystallization. He will partly use TEM in these studies. One Master student, Øyvind Paulsen, finished his master thesis within aluminium alloys in 2017.

During the last years, we have had several aluminium alloy related collaborations abroad. In 2017, the most important have been Eleonora Balducci at University of Bologna, Italy and Dr. Benjamin Milkereit in Rostock, Germany, in addition to the Japanese collaborations further presented elsewhere in this report.

As an example of advanced nanoscale characterisation of aluminium alloys we apply SPED in order to obtain a detailed and reliable assessment of the alloy microstructure. We have demonstrated that this diffraction technique in combination with a machine learning approach allows the identification of Al-Mg-Si-Cu precipitate types from areas exceeding 1 μm^2, see Figure 1 (J.K. Sunde et al. J. Phys.: Conf Ser. **902** (2017) 012022).

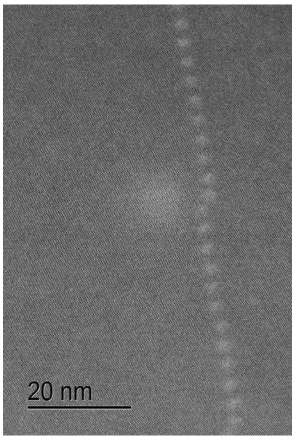


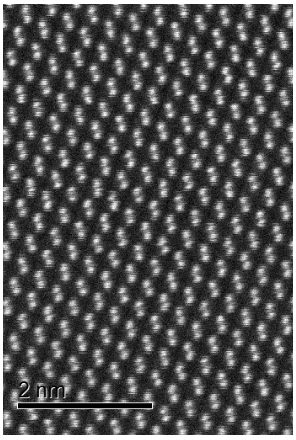
(a) (b)

Figure 1 (a) *Virtual dark-field image formed from a <100>$_{Al}$ SPED scan using the diffraction spots covered by the highlighted annulus (top left inset). The scan area covers 1.34 μm^2 in 550x450 pixels, and contains over 3700 precipitate cross-sections. Next to the scan area (middle column) are HAADF-STEM images of the three main precipitate types observed, where the unit cells of the phases are highlighted in red. The right column shows machine learning component patterns resembling (kinematical) diffraction patterns of the corresponding precipitate types.* ***(b)*** *Colour-map showing the distribution of all the 3700 labeled precipitate cross-sections.*

ENERGY MATERIALS – SOLAR CELLS

TEM has proven to be a crucial characterization tool to understand and improve the efficiency of both conventional and novel types of solar cells. The TEM Gemini Centre activities within solar cells include both types and a large range of materials. Maryam Vatanparast is a PhD student working on TEM characterization of intermediate band solar cells. The intermediate band is created by multiple layers of InAs quantum dots positioned inside a GaAs-based matrix semiconductor. Here we are studying the microstructure in detail, and also working on measuring band gaps with electron energy loss spectroscopy.





In 2017 we had one master student (Hogne Lysne) working on the deep level impurity approach doing Ag implantation into Si. Hogne studied this by electron tomography, and started as a PhD student on the same topic in August 2017. The activities within intermediate band solar cell materials are in collaboration with Associate professor Turid W. Reenaas at DP. PhD student Julie Stene Nilsen is part of the NANO2021 GRANASOL project (Low Cost, Ultra-High Efficiency Graphene/Nanowire Solar Cells).

The researcher project IN-Situ characterization and Simulation of Defect Evolution in Silicon (INSIDES), funded within the RCN ENERGIX program started in 2016. Maria Tsoutsouva is working as a post-doc in the project and she will combine in-situ synchrotron x-ray solidification studies with TEM studies to explore fundamental aspects of the evolution of crystallographic defects that limit the performance of polycrystalline silicon in solar cell applications.

Several of the external users and small projects in the TEM Gemini Centre were connected to solar cell materials or other forms of renewable energy. The Gemini Centre is participating in the FME (Centre of Environmental Friendly Energy) on solar cells and project students, PhD students and SINTEF researchers within TEM are actively taking part in subprojects related to both conventional as well as third generation solar cells.

During the last six years, SINTEF has cooperated with a small Trondheim-based company, Integrated Solar, in two consecutive IPN projects. The present project, “Improvement of efficiency in dual-junction solar cell”, started in 2016. The aim of the project is to develop a prototype, high efficiency solar cell based on epitaxial growth of a III-V cell on top of a Si cell by using a process that can be up-scaled to industrial production. Advanced TEM, with a resolution that can only be achieved by NORTEM’s top-level instruments, has been one of the most important techniques to reach the goals of the project.

SINTEF has worked together with ELKEM and IFE in two consecutive IPN projects within production of tailored Si powders for use in Li-ion batteries. The last one is called “SiCANODE” (2016–2018). The aim is to develop Si/graphite based composites as anodes in commercial Li-ion batteries. TEM has been one of the primary tools to characterize and understand the behavior of the anode composites as a function of structure, morphology and cycling conditions.

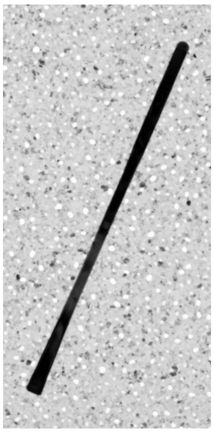
SINTEF is also partner in two other projects related to Li-ion battery R&D: “LiMBAT” is a researcher project where SINTEF, IFE, UiO, CNRS (France) and Hiroshima University (Japan) are cooperating to develop high capacity anodes based on metal hydrides for use in conversion electrodes in Li-ion batteries. TEM is in this project one of the most important tools to understand the correlations between morphologies and the electrochemical performances of the synthesized anode materials. In a second researcher project, in cooperation with Prof. Ann-Mari Svensson and Assoc. Prof. Fride Vullum-Bruer at DMSE, the aim is to develop cathode materials for use in Li-air batteries. The importance of TEM is here reflected by that the newly employed post-doc in the project is now trained to perform basic TEM, in addition to the more advanced TEM performed by SINTEF.

CHEMICAL ENGINEERING – CATALYSIS AND MEMBRANE MATERIALS

The Centre has continued a strong interaction with the national catalysis environment, including the NTNU Chemical Engineering department, the SINTEF Materials and Chemistry, Chemistry Oil and Gas Process Technology Departments. The Gassmaks programme project to develop catalysts and materials for a compact steam reformer is addressing both catalyst and materials issues, such as degradation of steels by metal dusting corrosion during exposure to synthesis gas. EU funded activities include the EU FP7 project FASTCARD, which aims at rational development of catalyst systems for biomass conversion, and SmartCAT that aims to develop new and innovative electrodes for commercial automotive PEM fuel cells. We hope that the SFI Innovation for a Competitive and Sustainable process Industry (iCSI), headed by professor Hilde Johnsen Venvik, will provide a platform for further applications of TEM in both academic and industrial catalysis research. Membrane research has included contributing to a study in the BIGCCS carbon capture FME project.

Journal publications utilising TEM during 2017 include carbon nanofiber as oxygen reduction catalysts and in composite catalysts for Fisher-Tropsch synthesis, carbon- ionic liquid supercapacitors and ceramic-ceramic hydro- gen permeable membranes.

NANOTECHNOLOGY

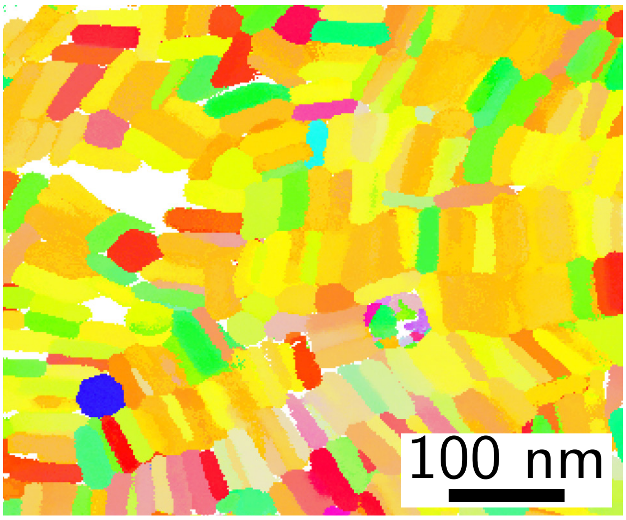


In nanotechnology and nanosciences, understanding the relation between functional properties and morphology, structure and composition at the (sub)nm scale is crucial. For achieving this understanding, TEM is important because of its resolving power for imaging and in analytical studies. The TEM Gemini Centre activities cover nanoparticles, 1D-nanostructures (i.e. nanowires, carbon nanotubes), thin films and 2D-materials such as graphene. NTNU NanoLab and NorFab-II is an important partner for the TEM Gemini Centre. PhD Aleksander Mosberg is funded by NTNU's "Enabling technologies: Nanotechnology", is based in the TEM Gemini Centre and works mainly in NanoLab's cleanroom. Mosberg's PhD is focused on using focused ion beam (FIB) for large scale nanostructuring. TEM is used to understand how the ion beam alters the materials.

Further are several core TEM Gemini people actively involved in different nanotechnology projects, for example as co-applicant or co-supervisor. The Centre has close ties with the Norwegian PhD Network on Nanotechnology for Microsystems. As can be seen from the publication list, many TEM studies on nanomaterials resulted in journal publications in 2017.

ADVANCED DATA PROCESSING

Big/smart data, machine learning, open source, digital transformation, etc. are hot terms in science and technology that are also central in several running TEM projects. We increased the data storage and handling capacity in 2017 and are discussing a substantial capacity upgrade to anticipate the increased demand by future detector developments. In TEM, especially multidimensional data set acquisition and handling, data processing transparency and dynamic in-situ studies are focus points. Students within the group have contributed over many years to the open-source software, especially the Python library HyperSpy (hyperspy.org). Data from all our detectors can now be handled in the same user interface. For large datasets or demanding calculations there is access to a powerful cluster. SINTEF has funded an internal project to support these developments and incorporates them in their TEM activities. Hands-on data processing labs were part of the organized electron diffraction workshop. Furthermore, we organized an internal workshop on scanning precession electron diffraction (SPED) data handling using Python and clusters. The main concrete achievements in advanced data processing in 2017 are related to atomic scale spectroscopy and precipitation statistics from large area SPED data.



ACTIVE PROJECTS

The table below shows the larger projects connected to TEM within the Gemini Centre. They are listed by funding type, title, duration and research partners. A number of smaller projects not listed, both academic and with direct industrial support, run in parallel. In total the Centre had over 80 different projects using the facilities.

Project type	Project title	Involved with TEM	Duration
SFI	**CASA - Centre for Advanced Structural Analysis**	1-2 PhDs, SINTEF	2015-2023
Partners: NTNU, SINTEF, Statens vegvesen, Forsvarsbygg, Norwegian ministry of local government and modernisation, NSM, Audi, Benteler, BMW, DNV GL, Gassco, Honda, Hydro, Sapa, Statoil, Toyota, Renault			
SFI	**SFI Manufacturing**	1 PhD, SINTEF	2015-2023
Partners: SINTEF SRM, SINTEF, NTNU, Benteler, Brødrene AA, Ekornes, GKN Aerospace, Hexagon composites, Kongsberg Automotive, Nammo, Raufoss Neuman, Plastal, Plasto, Rolls Royce, Teeness, Hybond			
SFI	**Industrial Catalysis Science and Innovation for a Competitive and Sustainable process Industry (iCSI)**	SINTEF	2015-2023
Partners: Yara Norge, K.A. Rasmussen, Dynea INOVYN Norge, Haldor Topsøe AS			
FME	**FME sol - Solar United**	1 Postdoc, SINTEF	2009-2017
Partners: IFE, NTNU, SINTEF, the University of Oslo, CleanSi, Dynatec, Elkem Solar, Mosaic, Norsun, Norwegian Crystals, Quartz Corp, REC Silicon, REC Solar, Semilab			
FP/FRINATEK	**Fundamental investigations of precipitation in the solid state with focus on Al-based alloys**	1 PhD, 1 Postdoc, SINTEF	2013-2017
Partners: NTNU, SINTEF			
FP/FRINATEK	**Fundamental investigations of precipitation in the solid state with focus on Al-based alloys**	1 PhD, 1 Postdoc, SINTEF	2013-2017
Partners: NTNU, SINTEF			
IPN/BIA	**Smart 6xxx Alloy Development for Rolling and Extrusion (RolEx)**	2 PhDs, SINTEF	2012-2017
Partners: Hydro, NTNU, SINTEF			
IPN/BIA	**Integrated Hardening and Sheet Press-forming of Aluminium (I-Pal)**	SINTEF	2016-2019
Partners: SINTEF, Hydro, SAPA, Raufoss Technology Neumann, AP&T, SINTEF Raufoss Manufacturing AS			
KPN/BIA	**Aluminium alloys with mechanical properties and electrical conductivity at elevated temperatures (AMPERE)**	1-2 PhDs, SINTEF	2015-2020
Partners: NTNU, SINTEF, Hydro, Nexans, Raufoss Neuman, SAPA, Grnges			
KPN/BIA	**Fundamentals of Intergranular Corrosion in Aluminum Alloys (FICAL)**	1 PhD, SINTEF	2015-2020
Partners: NTNU, SINTEF, Hydro, Benteler, Steertec, SAPA, Grnges			
IFP/Nano2021	**GRANASOL - Low Cost, Ultra-High Efficiency Graphene Nanowire Solar Cells**	1 PhD	2014-2019
Partners: NTNU, Sejong University, Aalto University, CRAYONANO AS			
INTPART	**Norwegian-Japanese Aluminium alloy Research and Education Collaboration (NJALC)**	Travel, exchange students	2015-2018
Partners: NTNU, SINTEF, Hydro, University of Toyama, Tokyo Institute of Technology			
FP/PETROMAKS2	**Fatigue and hydrogen degradation of steels (HyF-Lex)**	SINTEF	2015-2018
Partners: NTNU, SINTEF			
FP/GASSMAKS	**Development of materials and catalysts for compact reformers**	SINTEF	2014-2017
Partners: NTNU, SINTEF			
IPN/BIA	**Integrated Hardening and Sheet Press-forming of Aluminium (I-Pal)**	SINTEF	2016-2019
Partners: SINTEF, Hydro, SAPA, Raufoss Technology Neumann, AP&T, SINTEF Raufoss Manufacturing AS			

PEOPLE IN THE TEM GEMINI CENTRE

Randi Holmestad (Prof., DP, NTNU / Leader TEM Gemini Centre)
John Walmsley (Senior research scientist, SINTEF and Prof. II, DP, NTNU)
Ton van Helvoort (Prof., DP, NTNU)
Knut Marthinsen (Prof., DMSE, NTNU)
Yanjun Li (Prof., DMSE, NTNU)
Per Erik Vullum (Research scientist, SINTEF and Assoc. Prof. II, DP, NTNU)
Bjørn Gunnar Soleim (Senior engineer, DP, NTNU)
Ragnhild Sæterli (Senior engineer, DP, NTNU)
Yingda Yu (Senior engineer, DMSE, NTNU)
Sigmund J. Andersen (Senior research scientist, SINTEF)
Jesper Friis (Senior research scientist, SINTEF)
Calin Marioara (Senior research scientist, SINTEF)
Ruben Bjørge (Research scientist, SINTEF)
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Marat Gazizov (Postdoc, DP, NTNU)
Maria Tsoutsouva (Postdoc, DP, NTNU)
Maryam Vatanparast (PhD student, DP, NTNU)
Julie Stene Nilsen (PhD student, DP, NTNU)
Aleksander Mosberg (PhD student, DP, NTNU)
Emil Christiansen (PhD student, DP, NTNU)
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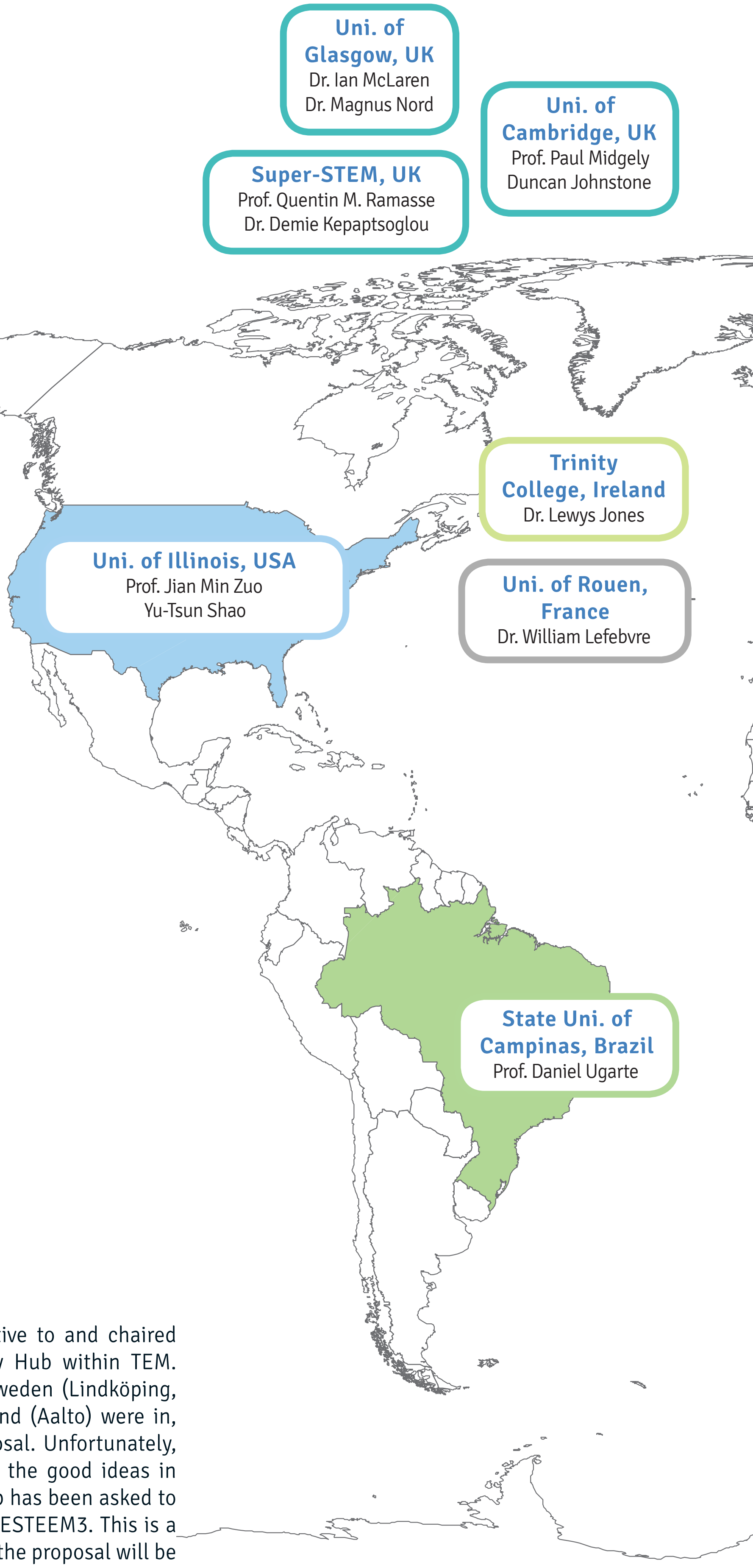
Jochen Busam (Master/PhD student, DP/DMSE, NTNU)
Steinar Myklebost (Master student, DP, NTNU)
Martin Rimbereid Vik (Project student, DP, NTNU)
Inger-Emma Nylund (Project / Master student, DP, NTNU)
Elise Otterlei Brenne (External Master student at Tokyo University)
Johanna Neumann (Project student, DP, NTNU)
Susanne Araya (Project student, DP, NTNU)
Sigurd Ofstad (Project student, DP, NTNU)
Elisabeth Thronsen (Project student, DP, NTNU)
Andreas Toresen (Project student, DP, NTNU)



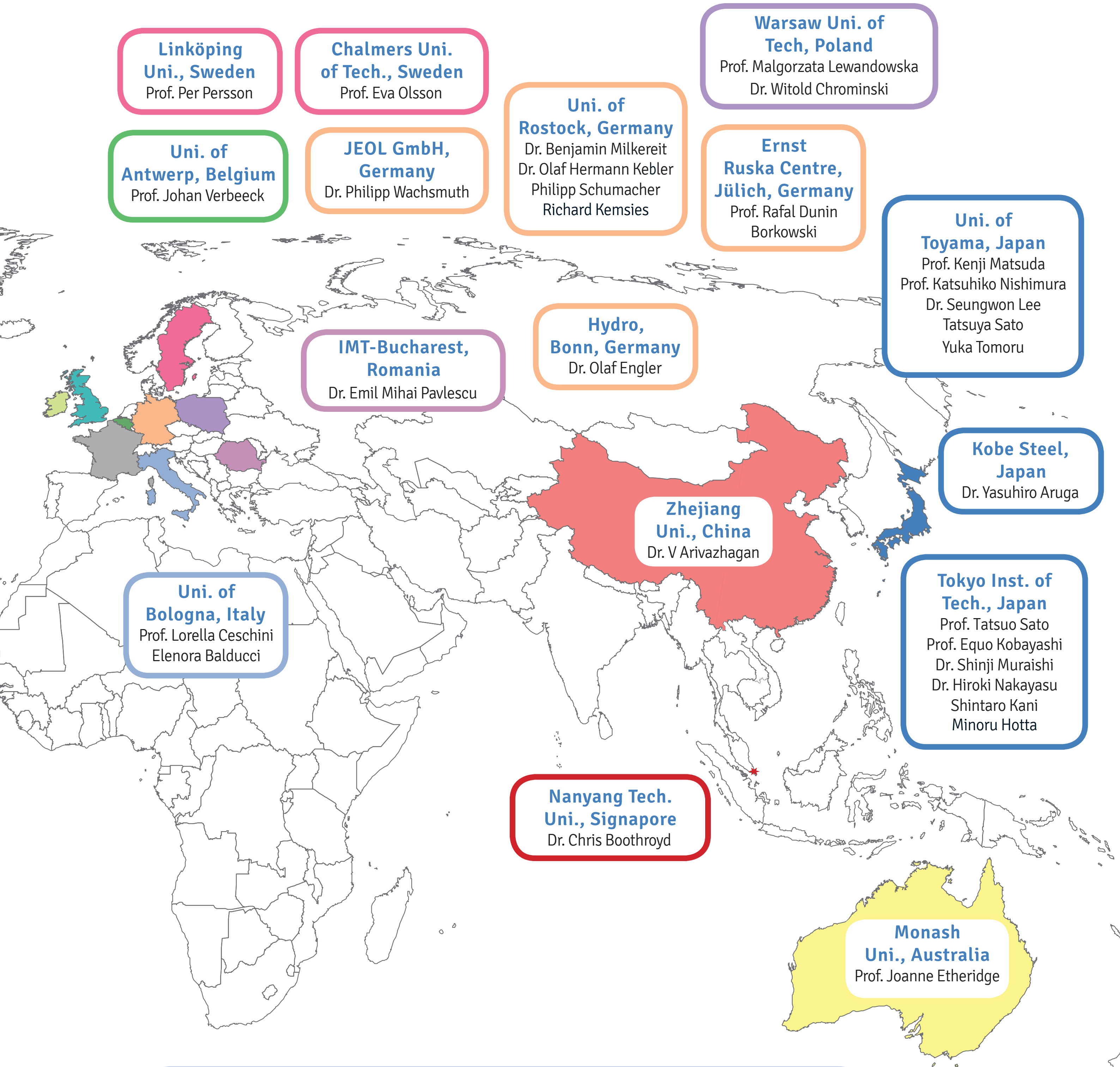
INTERNATIONAL COLLABORATION

As can be seen from the map and in the publication list, the TEM Gemini Centre has ties to many research institutions and researchers across the world. Some are long term collaborations, others are new initiatives (as the Nordic university Hub and ESTEEM3). The map illustrates the direct scientific collaborations. We have yearly several visitors from abroad applying and using the TEM facility in Trondheim. An example of a long-standing international cooperation is the contact with Toyama University and Tokyo Institute of Technology in Japan, now supported by the INTPART project. Moreover, in 2017 Jian Min Zuo was visiting the group for three months as an Onsager professor at NTNU. People from the group were chairs and invited contributors to international conferences and acted as PhD examiners abroad.

We thank all our international collaborators for the productive and stimulating interaction and hope we can continue the cooperation in the coming years!

NORDIC HUB AND ESTEEM3

The Gemini Centre took in the spring the initiative to and chaired a proposal to NordForsk for a Nordic University Hub within TEM. Seven partners from Norway (NTNU and UiO), Sweden (Lindköping, Stockholm, Chalmers), Denmark (DTU) and Finland (Aalto) were in, and a lot of effort was put into making the proposal. Unfortunately, the Hub was not granted, but we hope to reuse the good ideas in future calls. We are also very happy that our group has been asked to participate in the new EU Horizon 2020 initiative ESTEEM3. This is a network of the leading European TEM groups, and the proposal will be submitted in March 2018.



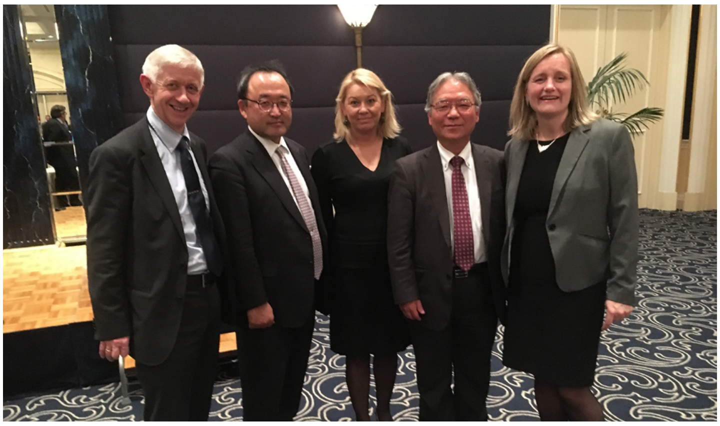
INTERNATIONAL VISITORS IN 2017

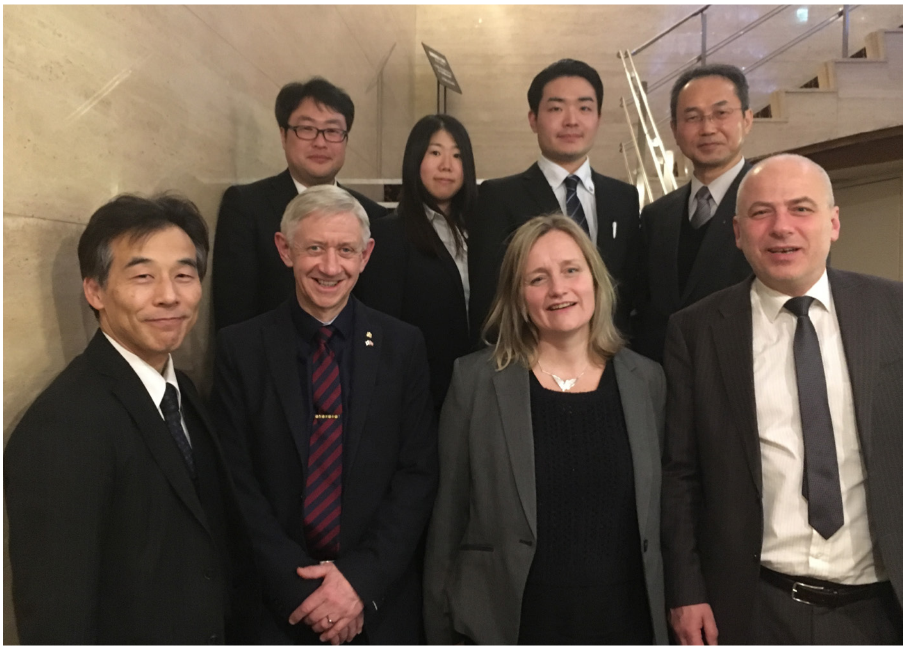
- Hiroki Nakayasua, Tokyo Institute of Technology, Japan, 3 months, January - March
- Jian Min Zuo, University of Illinois, Urbana-Champaign, USA, honorary Onsager Professor, NTNU 2017, 3 months, March, May-July, and December
- Yu-Tsun Shao, University of Illinois, Urbana-Champaign, USA, 1 month, June-July
- Aram Yoon, University of Illinois, Urbana-Champaign, USA, 1 week, June
- Haw-Wen Hsiao, University of Illinois, Urbana-Champaign, USA, 1 week, June
- Duncan Johnstone, University of Cambridge, UK, 1 week, June
- Minoru Hotta, Tokyo Institute of Technology, Japan, 3 months, September – December
- Shintaro Kani, Tokyo Institute of Technology, Japan, 3 months, September – December

INTPART PROJECT WITH JAPAN

A 3-year International Partnership (INTPART) project from the Norwegian Research Council and the Norwegian Centre for International Cooperation in Education (SIU) called "The Norwegian-Japanese Aluminium alloy Research and Education Collaboration" was started in 2016. In addition to NTNU and SINTEF, Hydro, University of Toyama and Tokyo Institute of Technology are partners. The objective of this project is to continue the fruitful partnership we obtained through the earlier BILAT project, and also include and formalize educational issues, such as guest lecturers, workshops and joint courses. Furthermore, exchange of students on Master/PhD levels between the university partners with research close to the aluminium industry, will ensure strong and long-lasting international collaboration.

In January 2017 Knut Marthinsen and Randi Holmestad went to Japan in a delegation with the industry minister Monica Mæland. She visited the aluminium company YKK, which is also one of the worlds larges zipper producers, close to Toyama, and we got the opportunity to present our INTPART project for her. Master students Yuka Tomuro and Tatsuya Sato from University of Toyama who stayed





in Trondheim in 2016, told about their 'Norway experience' as part of the offical program.

In 2017 we had two visitors from Tokyo Institute of Technology, master students Minoru Hotta and Shintaru Kani stayed for 3 months, from September to December; one month as internship in Hydro Sunndal, and two months, at DP, NTNU, collecting TEM results on their own aluminium alloys. Project students Elisabeth Thronsen and Iven Erga stayed one month in YKK in Kurobe (Toyama) in September, and did their project work on an YKK aluminium alloy. In 2018 the plan is that three more students from NTNU will do internships in Japanese aluminium companies (Nippon light metals, UAJC and KobelCo).

ONSAGER PROFESSOR ZUO

Jian Min Zuo was awarded the Lars Onsager professorship from NTNU for 2017 (https://www.ntnu.edu/onsager/professorship) and stayed in the TEM Gemini Centre for three months. During this period he gave lectures, for example in the diffraction workshop and the DP Friday colloquium. Furthermore, he contributed to and was a discussion partner in the Gemini Centre research, in particular on diffraction and strain measurements. We hope to get some common publications in the near future. Zuo's PhD student Yu-Tsun Shao stayed for one month, using the ARM to do some scanning electron nanodiffraction (SEND) and collaborated with different people in the Centre.

Dean of the Natural Sciences Faculty, NTNU, Prof. Anne Borg; Onsager Prof. Jian Min Zuo; Chairman of the Onsager Committee, Prof. Helge Holden.

RESEARCHERS NIGHT, SCHOOL VISITS AND OUTREACH

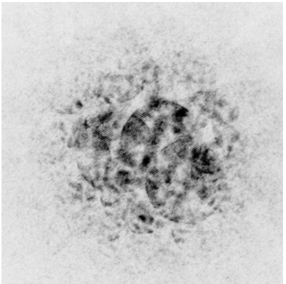
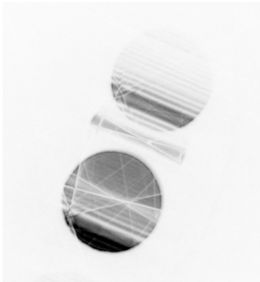
As in earlier years, the TEM Gemini Centre has contributed to a large number of high school visits through 2017. In September we had a stand about TEM and gave lab tour to students attending the Researchers Night in the Natural Science building. Further, a substantial number of international visitors had a tour of our facilities.

JOHN WALMSLEY MOVED TO CAMBRIDGE

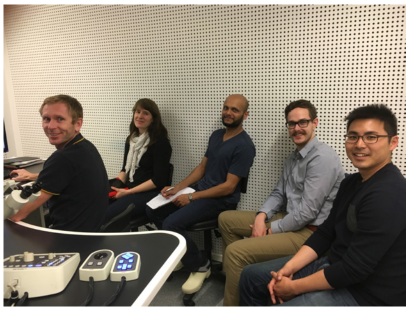
In April 2017 John Walmsley left Trondheim after nearly 20 years and moved back to England. He become technical head officer in Paul Midley's TEM lab at the University of Cambridge, one of the world's most influential TEM labs. John Walmsley came to Trondheim when he was hired in SINTEF Materials and Chemistry in 1998. He has been a very central person in the Trondheim TEM environment for nearly two decennia. In addition to being a SINTEF (senior) researcher, he was an adjunct professor at NTNU for more than 10 years. He was central in initiating the TEM Gemini Centre in 2006 and in the realization of NORTEM, from proposals (2010) to implementation (->2017). John has built up a broad network in Trondheim within materials, and it will be difficult to replace his broad competence. His competence covers a range of materials and application areas, including metals, surfaces, corrosion, and catalysts. In addition to TEM, he initiated activities focused on SEM, XPS and Auger. We want to thank John for his dedication and immense contributions to TEM and the materials environment in Trondheim and being a pleasant colleague and guide to many of us. We wish him good luck in Cambridge! We will miss you, but hope to keep in contact and collaborate further.

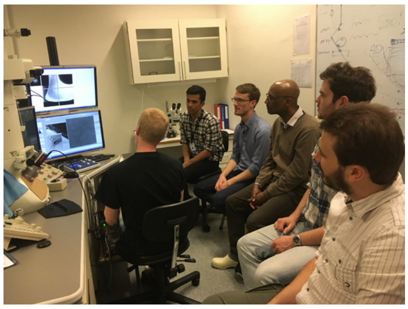
ELECTRON DIFFRACTION WORKSHOP

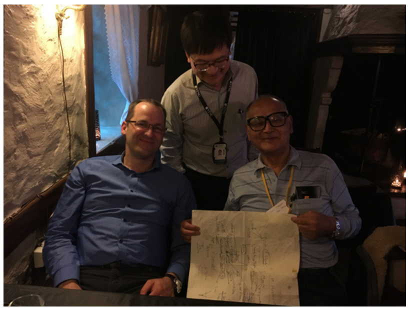
The TEM Gemini Centre at NTNU/SINTEF organized a diffraction workshop 'The reciprocal reality: electron diffraction in the new age' in Trondheim 12-14 June 2017. 27 participants from 16 labs and 11 countries participated, and learned about new developments in diffraction. Lecturers were Jian Min Zuo, University of Illinois, USA, Christoph Koch, Humboldt University, Germany, Duncan Johnstone, University of Cambridge, UK and Randi Holmestad, NTNU.

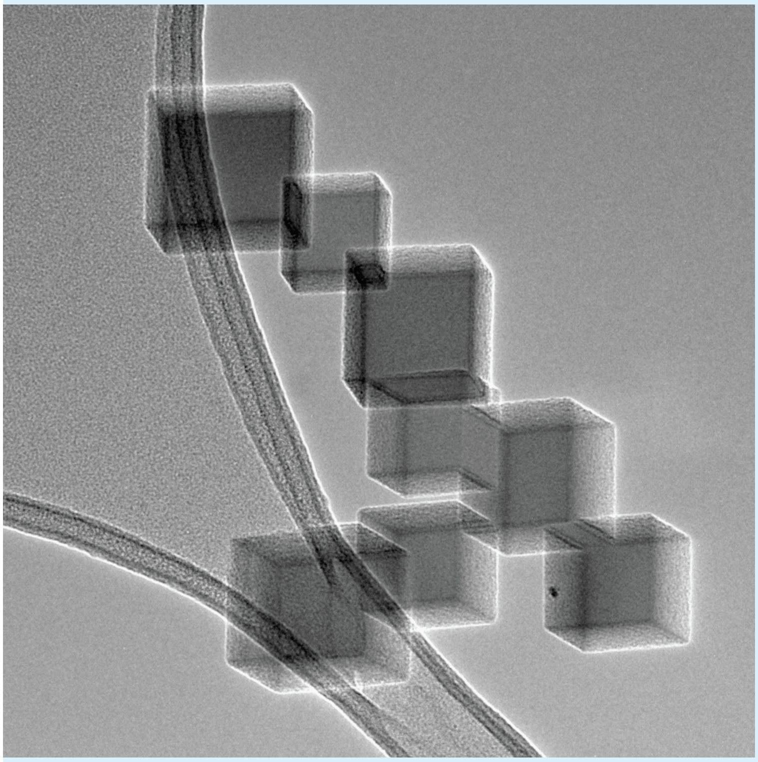
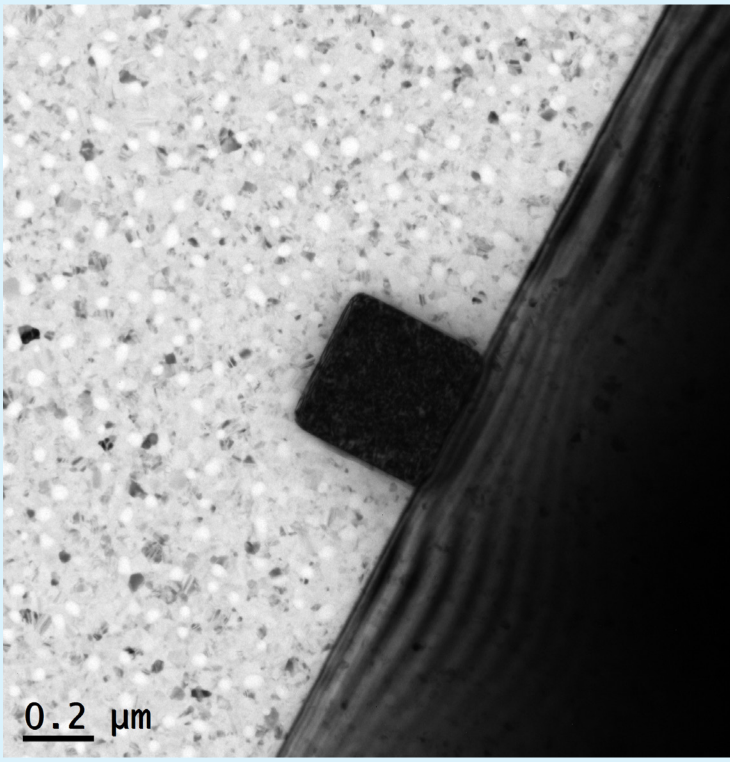
Different aspects of scanning diffraction were covered, including precession and convergent beam methods. Advanced methods with rocking and scanning beams were discussed in the lectures. There were experimental labs where the different diffraction techniques were demonstrated. In the computer labs a variety open source programs for simulating and analyzing diffraction patterns were demonstrated and used, including QED, QSTEM and Hyperspy. The workshop was followed up by an internal workshop on SPED in November which Prof. Zuo also attend.

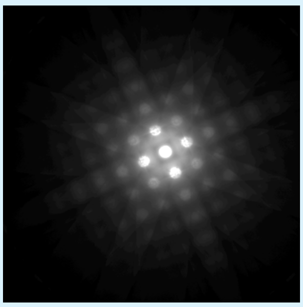
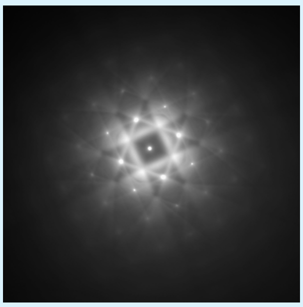
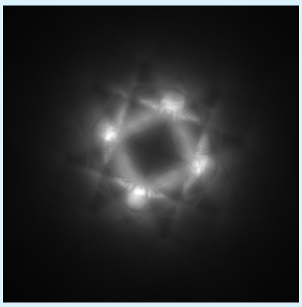
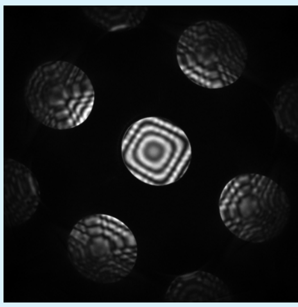






SELECTED
SCIENTIFIC PAPERS

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Atomic resolution imaging of beryl: an investigation of the nano-channel occupation

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Key words. Beryl, colour centre, HAADF STEM, nano-channel, transmission electron microscopy.

Summary

Beryl in different varieties (emerald, aquamarine, heliodor etc.) displays a wide range of colours that have fascinated humans throughout history. Beryl is a hexagonal cyclo-silicate (ring-silicate) with channels going through the crystal along the *c*-axis. The channels are about 0.5 nm in diameter and can be occupied by water and alkali ions. Pure beryl ($Be_3Al_2Si_6O_{18}$) is colourless (variety goshenite). The characteristic colours are believed to be mainly generated through substitutions with metal atoms in the lattice. Which atoms that are substituted is still debated it has been proposed that metal ions may also be enclosed in the channels and that this can also contribute to the crystal colouring. So far spectroscopy studies have not been able to fully answer this. Here we present the first experiments using atomic resolution scanning transmission electron microscope imaging (STEM) to investigate the channel occupation in beryl. We present images of a natural beryl crystal (variety heliodor) from the Bin Thuan Province in Vietnam. The channel occupation can be visualized. Based on the image contrast in combination with ex situ element analysis we suggest that some or all of the atoms that are visible in the channels are Fe ions.

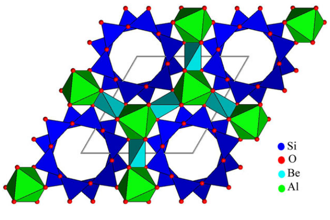

Fig. 1. Atomic model of beryl.

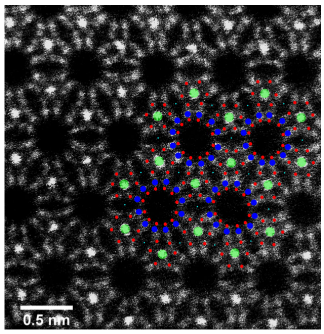

Fig. 3. Atomic resolution HAADF STEM image of a natural Heliodor crystal along the *c*-axis. Be atoms are not visible. The colour code for the atomic model superimposed on the image is similar to that used in the atomic model presented in Figure 1.

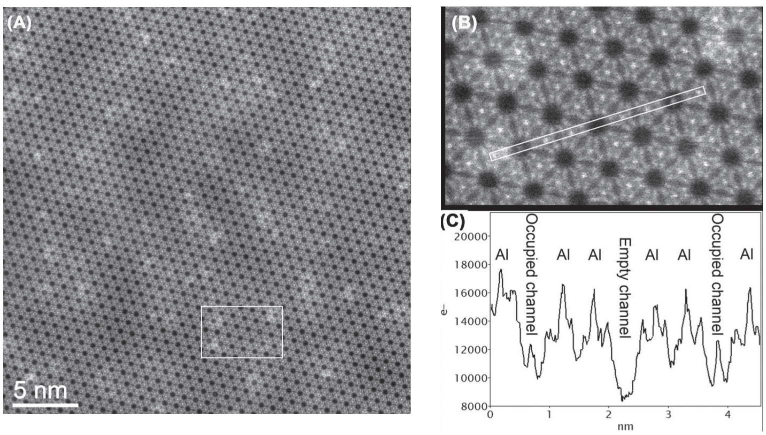

Fig. 4. (A) Atomic resolution dark field TEM image of a natural Heliodor crystal along the *c*-axis. Individual atoms can be seen in the centre of some of the nano-channels. (B) Enlarged image of box marked in (A). (C) Intensity line profile of box marked in (B).

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Atomistic details of precipitates in lean Al–Mg–Si alloys with trace additions of Ag and Ge studied by HAADF-STEM and DFT

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ABSTRACT
Bonding energies and volume misfits for alloying elements and vacancies in multicomponent Al–Mg–Si alloys have been calculated using density functional theory (DFT). A detailed atomic scale analysis has been done for characteristic precipitate structures, using high-angle annular dark-field scanning transmission electron microscopy. Two new stacking configurations of the important strengthening phase β'' were discovered in the Ge-added alloy. All three stacking variations were found to be energetically favourable to form from DFT calculations. The second stacking configuration, β_2'', contains vacated columns in its unit cell, consequently requiring less solute to create the same volume fraction of precipitate needles. DFT suggests a lower formation enthalpy per atom for β_2'' when Si is exchanged with Ge. In the alloy containing Ag additions, a new Q′/C-like local configuration containing Ag instead of Cu was discovered, also this phase was deemed energetically favourable from DFT.

ARTICLE HISTORY
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KEYWORDS
Aluminium alloys; HAADF-STEM; density-functional theory; Si/Ge; nanoscale precipitates; crystal structure

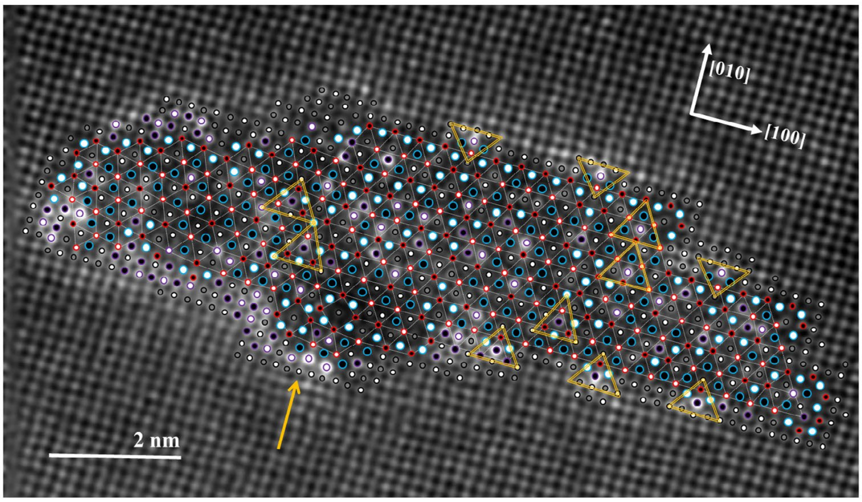

Figure 2. (colour online) Precipitate cross section in the Ag-added alloy with atomic overlay, see Figure 3 (a) for the original image. The segregation of Ag at the precipitate-matrix interface is evident. The hexagonal Si-network is indicated with dashed, white lines. The Q′/C-like local configurations, where Cu sites are replaced by Ag, are indicated with yellow triangles. For explanation of the symbolic representation of elements, see legend in Table 2.

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Bandgap measurement of high refractive index materials by off-axis EELS

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ABSTRACT

In the present work Cs aberration corrected and monochromated scanning transmission electron microscopy electron energy loss spectroscopy (STEM-EELS) has been used to explore experimental set-ups that allow bandgaps of high refractive index materials to be determined. Semi-convergence and –collection angles in the μrad range were combined with off-axis or dark field EELS to avoid relativistic losses and guided light modes in the low loss range to contribute to the acquired EEL spectra. Off-axis EELS further supressed the zero loss peak and the tail of the zero loss peak. The bandgap of several GaAs-based materials were successfully determined by simple regression analyses of the background subtracted EEL spectra. The presented set-up does not require that the acceleration voltage is set to below the Čerenkov limit and can be applied over the entire acceleration voltage range of modern TEMs and for a wide range of specimen thicknesses.

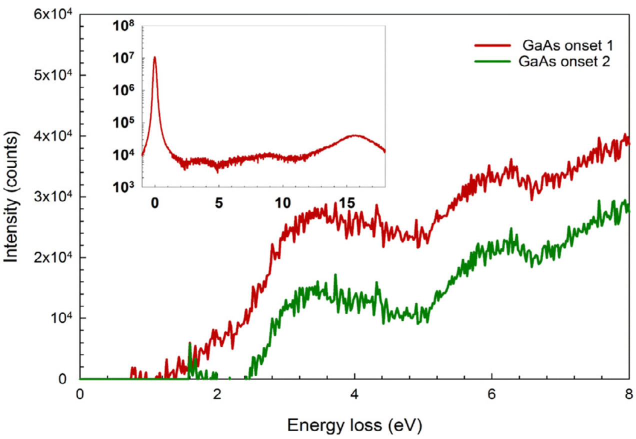

Fig. 6. Different bandgap onsets for GaAs in the Low-Mag STEM set-up after background subtraction. Low-Mag on-axis EEL spectrum from GaAs, acquired at 80 kV and with semi-convergence and –collection angles both equal to 0.11 mrad. No sign of any optical onset from a direct bandgap at 1.42 eV can be observed directly in the unprocessed spectrum. Attempts to remove the background end up similar to the case where larger semi-angles are used and give a background subtracted signal with an onset energy anywhere between 1 and 2.5 eV, depending on the background subtracted. The entire EEL spectrum in logarithm scale is shown in the inset.

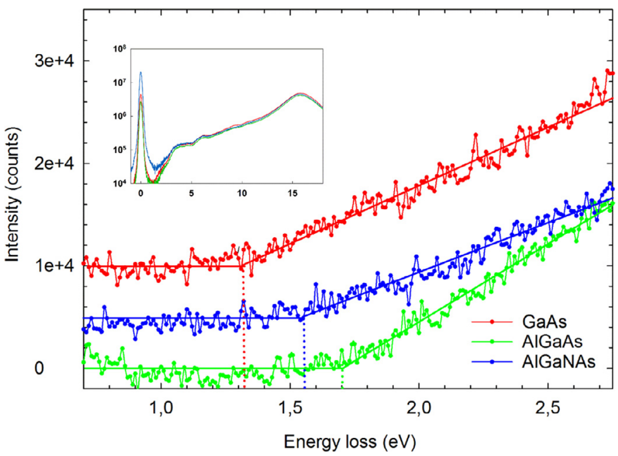

Fig. 7. Background subtracted spectra from GaAs, Al0.25Ga0.75As and Al0.25Ga0.75As1-xNx (x < 1) acquired in the Low-Mag off-axis set-up as shown in Fig. 3b). These spectra are taken from a single line scan that goes from vacuum and through the various layers and ends in the GaAs substrate. Each of the spectra are from the central part of the GaAs, AlGaAs and AlGaNAs layers to avoid any contribution from the nearby layers or from interfaces. The spectra from AlGaNAs and GaAs are shifted vertically by 5000 and 10,000 counts for clarification. The bandgap is defined as where the regression lines from the least square fitting cross zero intensity (represented by the horizontal lines). The significant decrease of the ZLP under off-axis conditions is shown in the unprocessed EEL spectra in the inset.

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Advanced Structural and Chemical Imaging

RESEARCH

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Atomap: a new software tool for the automated analysis of atomic resolution images using two-dimensional Gaussian fitting

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Abstract

Scanning transmission electron microscopy (STEM) data with atomic resolution can contain a large amount of information about the structure of a crystalline material. Often, this information is hard to extract, due to the large number of atomic columns and large differences in intensity from sublattices consisting of different elements. In this work, we present a free and open source software tool for analysing both the position and shapes of atomic columns in STEM-images, using 2-D elliptical Gaussian distributions. The software is tested on variants of the perovskite oxide structure. By first fitting the most intense atomic columns and then subtracting them, information on all the projected sublattices can be obtained. From this, we can extract changes in the lattice parameters and shape of A-cation columns from annular dark field images of perovskite oxide heterostructures. Using annular bright field images, shifts in oxygen column positions are also quantified in the same heterostructure. The precision of determining the position of atomic columns is compared between STEM data acquired using standard acquisition, and STEM-images obtained as an image stack averaged after using non-rigid registration.

Keywords: Quantitative STEM, Strain mapping, Image processing, Oxygen octahedral distortion, Non-rigid registration

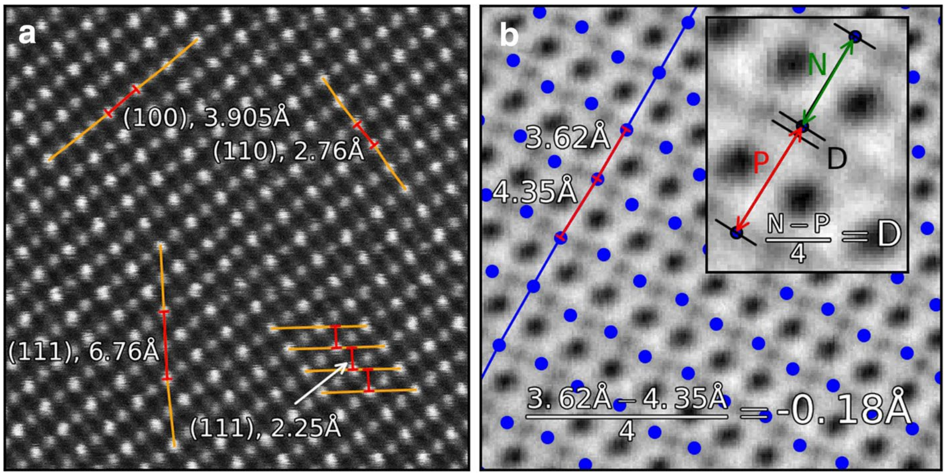

Fig. 2 Quantifying distances between atomic planes. **a** Distances between Sr atomic planes in different directions. Showing how finding the distance between atomic planes is straightforward for the [001] and [110] directions, but not for the [111] direction. **b** Calculating displacement (*D*) from a centrosymmetric position between atomic columns, here shown on oxygen columns. The *inset* shows how the distance difference is calculated for oxygen atoms in the [001]-direction for an LFO film. The distance to the next (*N*) and previous (*P*) atom is calculated, as shown with the *green* and *red double headed arrows*

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Atomic-resolution chemical mapping of ordered precipitates in Al alloys using energy-dispersive X-ray spectroscopy

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ABSTRACT

Scanning transmission electron microscopy (STEM) coupled with energy-dispersive X-ray spectroscopy (EDS) is a common technique for chemical mapping in thin samples. Obtaining high-resolution elemental maps in the STEM is jointly dependent on stepping the sharply focused electron probe in a precise raster, on collecting a significant number of characteristic X-rays over time, and on avoiding damage to the sample. In this work, 80 kV aberration-corrected STEM-EDS mapping was performed on ordered precipitates in aluminium alloys. Probe and sample instability problems are handled by acquiring series of annular dark-field (ADF) images and simultaneous EDS volumes, which are aligned and non-rigidly registered after acquisition. The summed EDS volumes yield elemental maps of Al, Mg, Si, and Cu, with sufficient resolution and signal-to-noise ratio to determine the elemental species of each atomic column in a periodic structure, and in some cases the species of single atomic columns. Within the uncertainty of the technique, S and β” phases were found to have pure elemental atomic columns with compositions Al_2CuMg and $Al_2Mg_5Si_4$, respectively. The Q’ phase showed some variation in chemistry across a single precipitate, although the majority of unit cells had a composition $Al_6Mg_6Si_{7.2}Cu_2$.

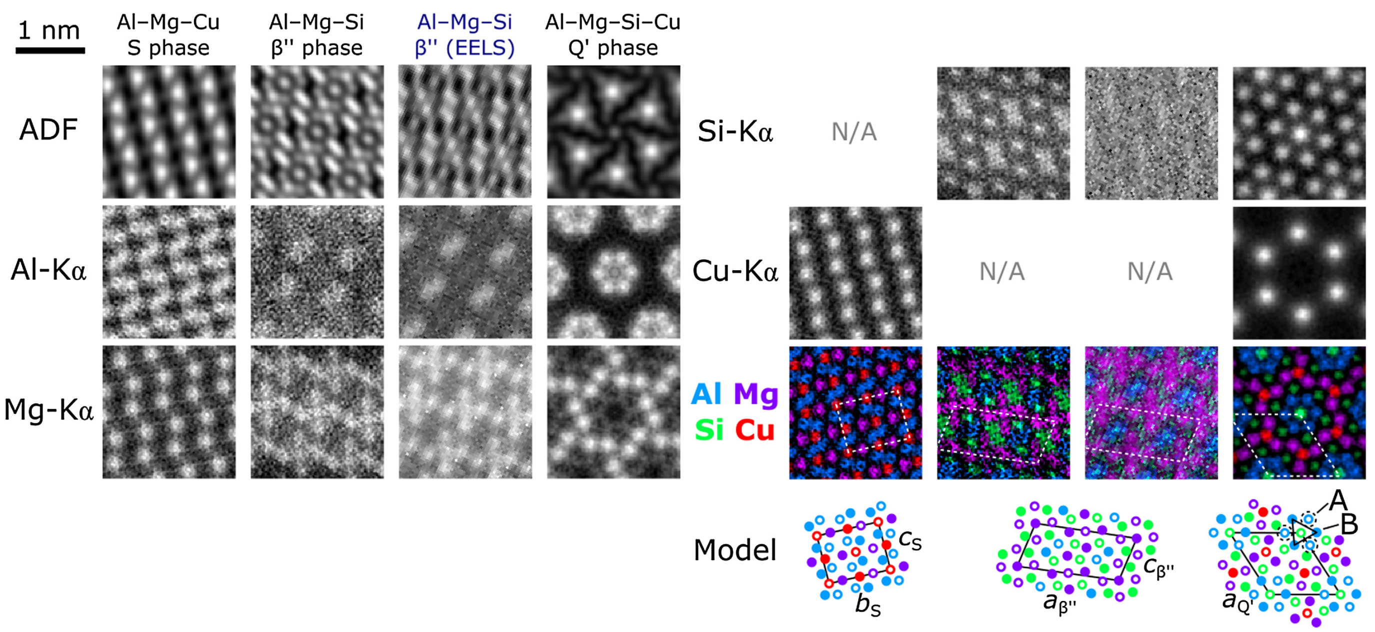

Fig. 3. Symmetry-averaged ADF and EDS images for three precipitates (S, β” and Q’ phases) in Al-Mg-(Si)-(Cu) alloys. EELS images from β” are shown for comparison. All images are spatially averaged, and S/β” are 2-fold rotation averaged, while Q’ is 6-fold averaged. A common scale bar is given in the upper left corner. Color codes in the two last rows; blue = Al, purple = Mg, green = Si, red = Cu. In the atomic models, filled (z = 0) and empty circles (z = 1/2) are separated by $a_{Al}/2 = 202$ pm in the paper normal direction.

PUBLICATIONS 2017

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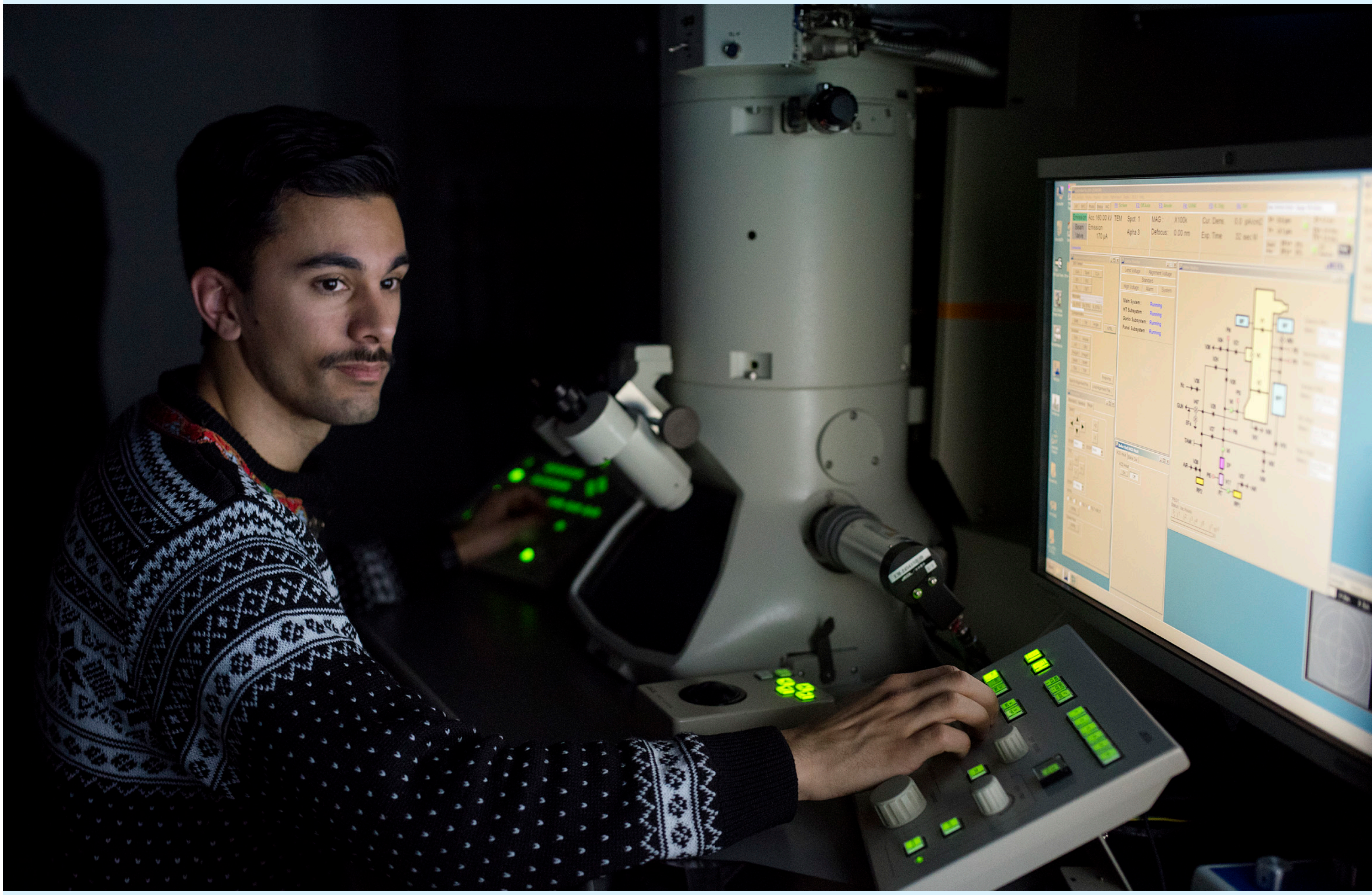

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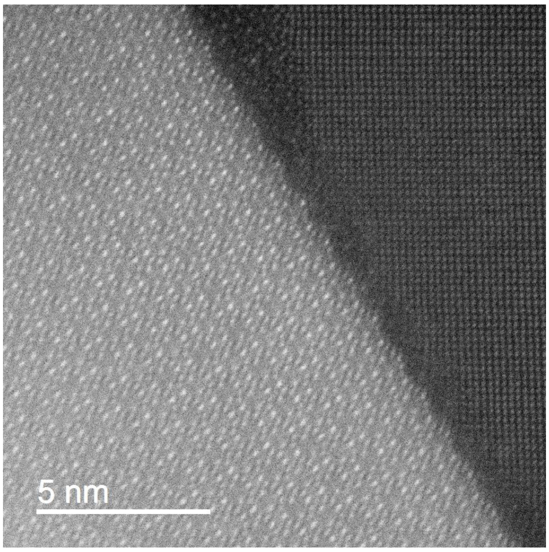

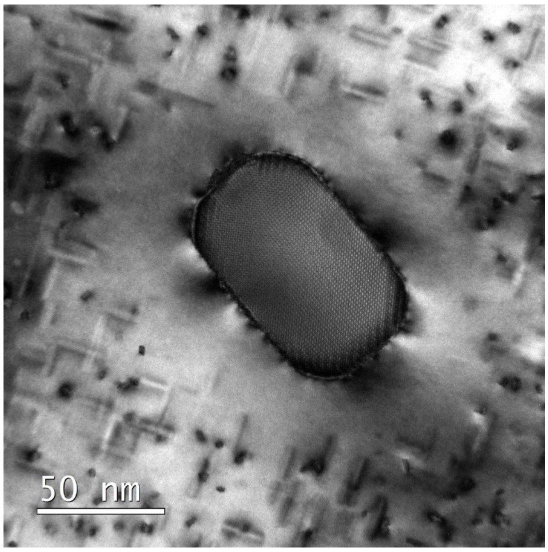

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Project students in the TEM Gemini Centre fall 2017.

MASTER/ PROJECT THESES

Ingrid Marie Andersen, "TEM characterization of III-V nanowires for laser applications" (Diploma, June 2017).

Susanne Araya, "TEM investigations of microstructure in additive manufactured Aerospace-grade Titanium Titanium" (Project, December 2017).

Jochen Busam, "TEM characterization of high-purity quartz" (Diploma, June 2017).

Hogne Lysne, "3D TEM characterization of silver implanted silicon for intermediate band solar cells" (Diploma, June 2017).

Steinar Myklebost, "Developing Quantitative Image Processing of Scanning Electron Microscopy Data Sets to Evaluate Nanowire Growth" (Diploma, June 2017).

Johanna Neumann, "Electron Microscopy Characterisation of GaAsSb Nanowires on Graphene Glass" (Project, December 2017).

Inger-Emma Nylund, "Evaluation of energy-dispersive spectroscopy characteristics for improved compositional analysis" (Project, June 2017).

Inger-Emma Nylund, "Electron Microscopy Characterisation of GaN Nanowires grown on Graphene Glass" (Diploma, start August 2017).

Sigurd Ofstad, "First principles study of the displacement field surrounding β'' in Aluminium - Density functional calculations with boundary conditions defined by linear elasticity theory" (Project, December 2017).

Øyvind Paulsen, "Transmission Electron Microscopy Study of Two Peak Hardened Al-Mg-Si-Cu Alloys" (Diploma, June 2017).

Elisabeth Thronsen, "Natural ageing effect in a pre-deformed hybrid 6xxx/2xxx series Aluminium Alloy" (Project, December 2017).

Andreas Toresen, "The Search for Lead-Free Piezoelectric Materials - A TEM Characterisation of KNN Thin Films" (Project, December 2017).

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The microscopes are in the basement of Chemistry building 1 - K1.

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TEM Gemini Centre management 2017.