ANNUAL REPORT 2016

TEM GEMINI CENTRE

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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 Department of Physics (DP), NTNU and researchers from the Material Physics, Trondheim research group in SINTEF Materials and Chemistry.

In June 2009 the Centre was renominated as a Gemini Centre for a new three year 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 research groups work within materials physics and 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 TEM infrastructure in Norway.

THE GEMINI CENTRE CONCEPT

Gemini Centres form a model for strategic cooperation in which scientific groups with parallel interests coordinate their scientific efforts and jointly operate their resources. SINTEF -and NTNU - the Norwegian University of Science and Technology - have established a wide range of Gemini Centres. The concept has also been adopted as the model for SINTEF's cooperation with the University of Oslo (UiO).

The objective of the Gemini Centres is to develop large scientific groups of higher quality than either of the partners could manage to build up on their own. There is an international demand for first-class scientific groups from both project sponsors and students. For this reason, the Gemini Centres have adopted the following vision:

'**Together for International Excellence**'.

Graphic design: Ingebjørg Fyrileiv Guldvik, Sigurd Wenner
Photo: Ole Morten Melgård
Cover image: Titanium grade 5, alpha and beta grains, bright-field TEM.
Acquired by Eva A. Mørtsell. The field of view is 6 µm wide.
All images in this report are taken by employees at the TEM Gemini Centre.

CONTENT

INTRODUCTION

2016 has again been a good year for the TEM Gemini Centre. We have continued to systematically build up routines and established competence around the new Trondheim NORTEM instruments that were installed in 2013. It is a big responsibility and challenge to establish an effective role within Norway for such expensive and advanced equipment in the NTNU/SINTEF landscape. It is, therefore, very satisfying to see the high levels of use and quality of scientific and educational output achieved during the last year. We regard the NORTEM project as evidence of the successful partnership between NTNU and SINTEF. The total cost model for lab infrastructure was introduced at NTNU, in combination with SINTEF ownership. This resulted in a complex debate ongoing throughout 2016. The final outcome was the transfer to a full infrastructure ownership to NTNU. The next step now is to define NORTEMs national role and secure access for all research groups who need and are interested in using 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 and the relation to the national NORTEM project.

The Gemini Centre participates in a range of projects, including national, public, industrial and EU funding ones. The TEM Gemini 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). In addition, the TEM Gemini Centre is central in two ongoing KPN projects on aluminium with Norwegian aluminium industry – FICAL and AMPERE. Another very positive trend is that we have an increased cooperation with a broader range of industry (for example Saint Gobain Ceramic Materials and The Quartz Corp), other Norwegian universities (UiB), Norwegian institutions (IFE) and interest from research fields that we traditionally have had little collaboration with. Examples here are zero-emission building materials, biomaterials and geology.

The Centre organised two days of strategy discussions in June 2016. Here, discussions about new project markets, better collaboration between SINTEF and NTNU and the two departments in NTNU were discussed. Parts of this were used in the action plan for 2017. As documented in this report, the Centre had about 35 active users whose results have contributed to 36 journal publications (plus 9 in press) in 2016. Of these, 10 have co-authors from both NTNU and SINTEF. 11 of the 36 publications have international co-authors. They are found in a broad range of journals, showing how generic TEM is. 5 papers were in journals with an impact factor larger than 7. In

addition, 3 PhD and 6 Master candidates were educated with TEM as a substantial part of their theses in 2016. 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. We organized the Nordic Microscopy conference SCANDEM with 200 participants in June. The conference was very successful, with 100 presentations and 24 companies participating in the exhibition. The TEM introduction course was organized in September with 23 participants. In the fall we gave a PhD course of 7.5 credits for 11 PhD students. We had group meetings almost every week, and have given many guided tours for high school students and visitors to the microscopes. As seen from the publication list at the end, most members of the TEM group participated in international conferences and meetings in 2016. A very positive trend is that many people in the group have started to use the scanning precession electron diffraction (SPED) technique for a broad range of projects and several members contributed to open source code developments within HyperSpy.

Looking ahead, we are very happy that Jian Min Zuo has been awarded the Lars Onsager professorship and will stay in our group three months in 2017. Further, we will organize an international workshop in electron diffraction in June 2017. 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 2016. For more details, see our home page:
http://www.ntnu.edu/geminicentre/tem

TEM Gemini Centre management group,
February 2017.

BOARD AND MANAGEMENT

During 2016 the TEM Gemini Centre board has consisted of:

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

The management of the Centre has been:

- **Randi Holmestad**, Physics, NTNU, Leader
- **John Walmsley**, 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; John C Walmsley, Senior scientist/Professor II, 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/Professor II, SINTEF Materials and Chemistry.

THE NORTEM PROJECT

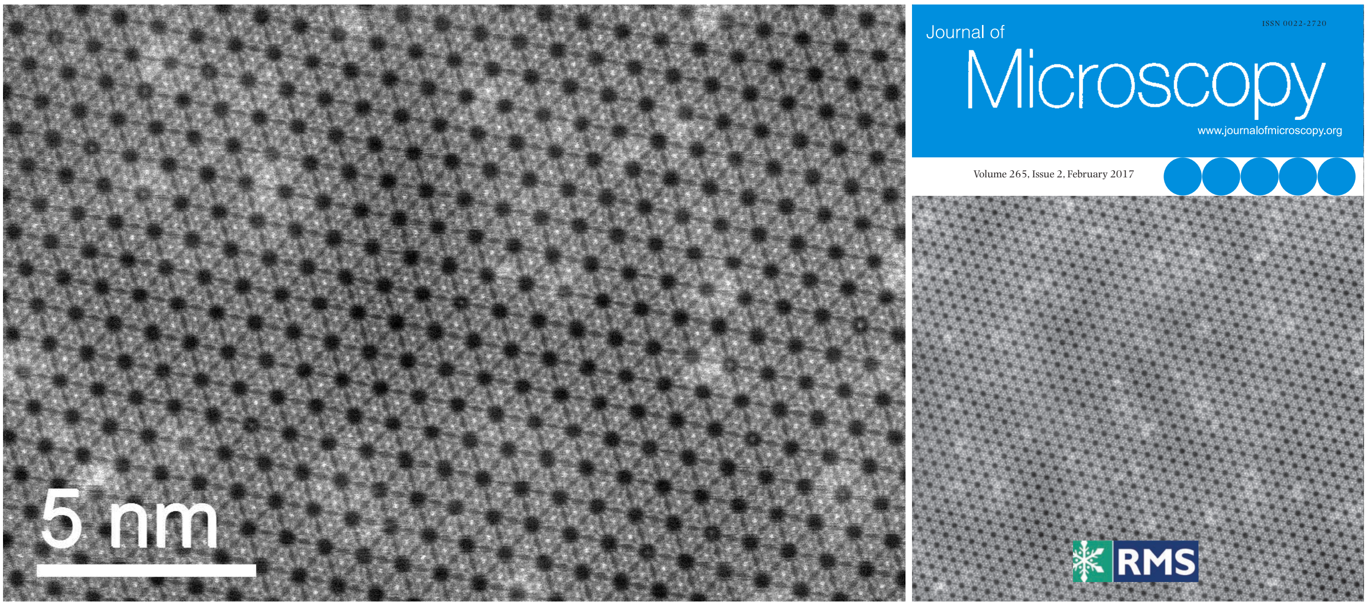
NORTEM (Norwegian Centre for Transmission Electron Microscopy) is a nationally coordinated large scale infrastructure project with three partners - SINTEF, NTNU and UiO, funded by the Research Council of Norway and the three partners. The budget for new equipment and the rebuilding in the project has been about 75 MNOK for the two geographical nodes, Trondheim and Oslo. We have now been running the facility for close to three years. The support to NORTEM from the Research Council ended in 2016, but the project will continue. It is important to start planning the medium and long-term running and development of the facilities.

The vision of NORTEM is to be "A world-class TEM centre providing access to expertise and state-of-the-art infrastructure for fundamental and applied research within the physical sciences in Norway". With infrastructure at two locations, close to potential users, the investment in TEM is consistent with high levels of recent investments made in areas such as nanotechnology and solar energy. It also matches the substantial investment in new TEM technology globally and in our neighbouring countries. Besides being a top research TEM lab, the infrastructure provide access to TEM for a broader user environment, addressing fundamental and applied research topics in physics, chemistry, materials science and geology.

For this combination of a research lab and a user facility, a sustainable running model is required, and the TEM Gemini Centre has spent a considerable amount of time during the last years to establish a sound running model for the infrastructure. As a part of this process, "seed project" funding has been included in the NORTEM budget, for proof-of-principle studies and training activities. The last seed projects finished in the first quarter of 2016. In Trondheim, these have contributed to several journal publications, which include TEM results using the NORTEM infrastructure, initiated new activities with other NTNU departments as well as external institutes and universities in Norway and abroad, and brought several new users of the facilities. Very importantly, the required resources to do TEM have been incorporated in new research applications. SINTEF Materials and Chemistry has also funded strategic projects where a large part of the budget was allocated to the development of methodology and use of NORTEM infrastructure. These have resulted in several journal publications, formed the basis for project funding applications, and contributed to increased use of TEM in existing projects.

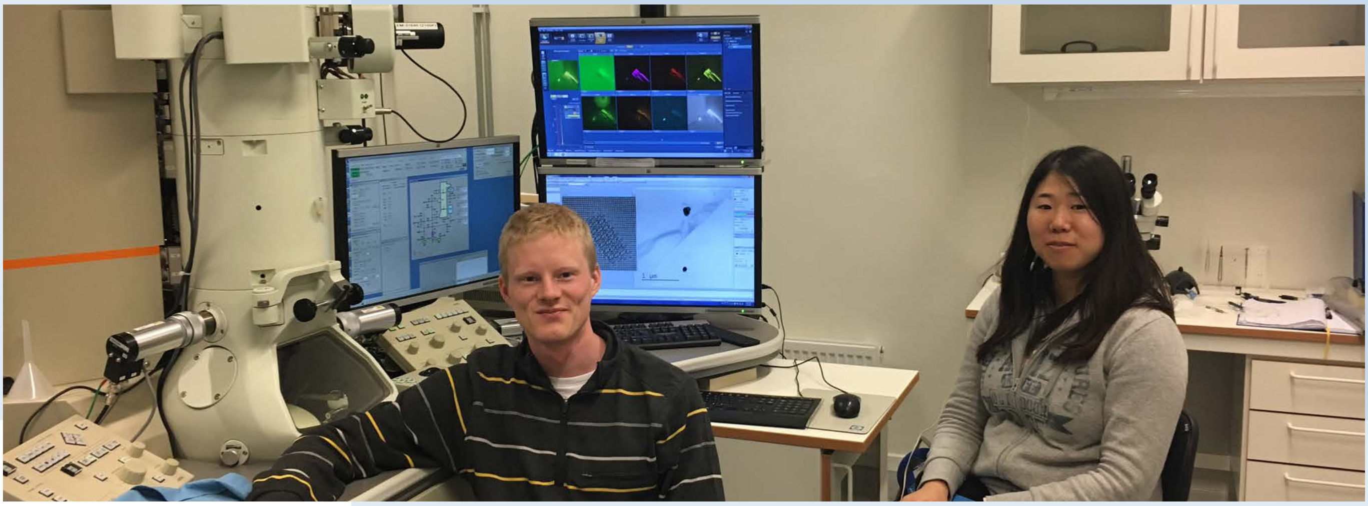
Attention has been paid to addressing the challenge of establishing and getting the best out of the facilities. The facility had in 2016 two senior engineers, Bjørn Soleim and Ragnhild Sæterli supporting maintenance, training, competence and techniques. We have a high uptime and an increasing number of operators from outside the host institution. John Walmsley and Per Erik Vullum have been working as adjunct (associate) professors, which particularly contributes to developing interaction between NTNU and SINTEF.

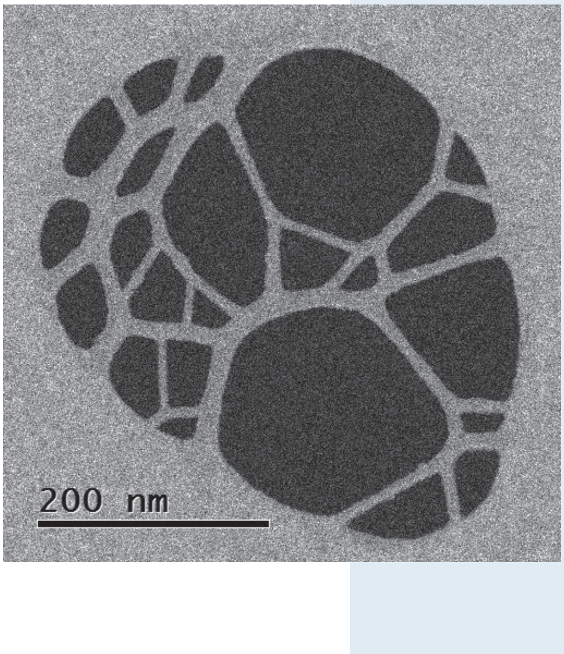
For more information on NORTEM see the webpages: www.nortem.no

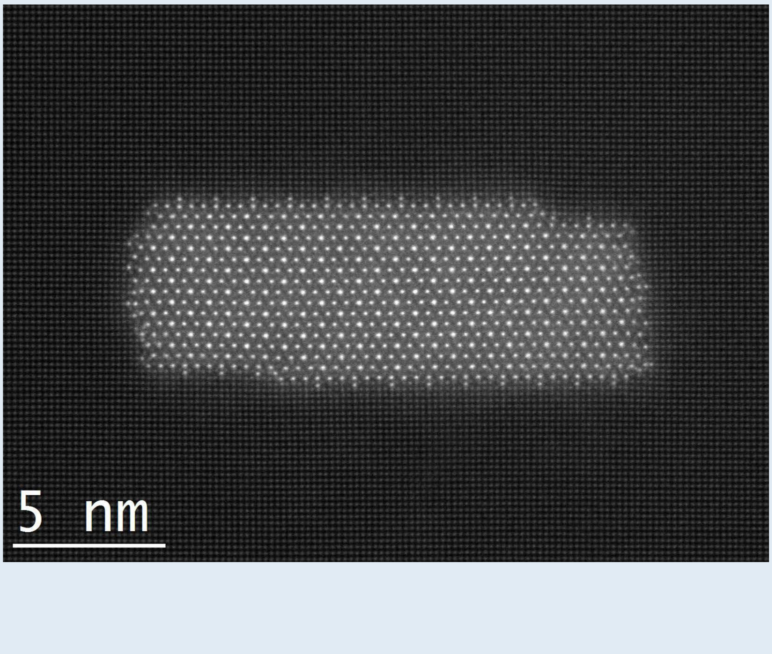


V. Arivazhagan, F. D. Schmitz, P. E. Vullum, A. T. J. van Helvoort and B. Holst, Atomic resolution imaging of beryl: an investigation of the nano-channel occupation. Journal of Microscopy, 265, 245-250, 2017.

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 LaB6, 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*

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 orientation mapping and precession diffraction system*
- *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*
- *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, US camera, ASTAR, gas reaction and tomography holders*	*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 2015 we invested in a new MEMS based heating holder, which can heat very rapidly, is highly controllable and extremely stable across the total temperature range (up to 1300 C) and when changing temperature. Early 2016 we had already the first publication with this holder. Investment in additional holders for dedicated advanced studies is continuously evaluated.

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 will be operative February 2017.

SUPPORTING FACILITIES

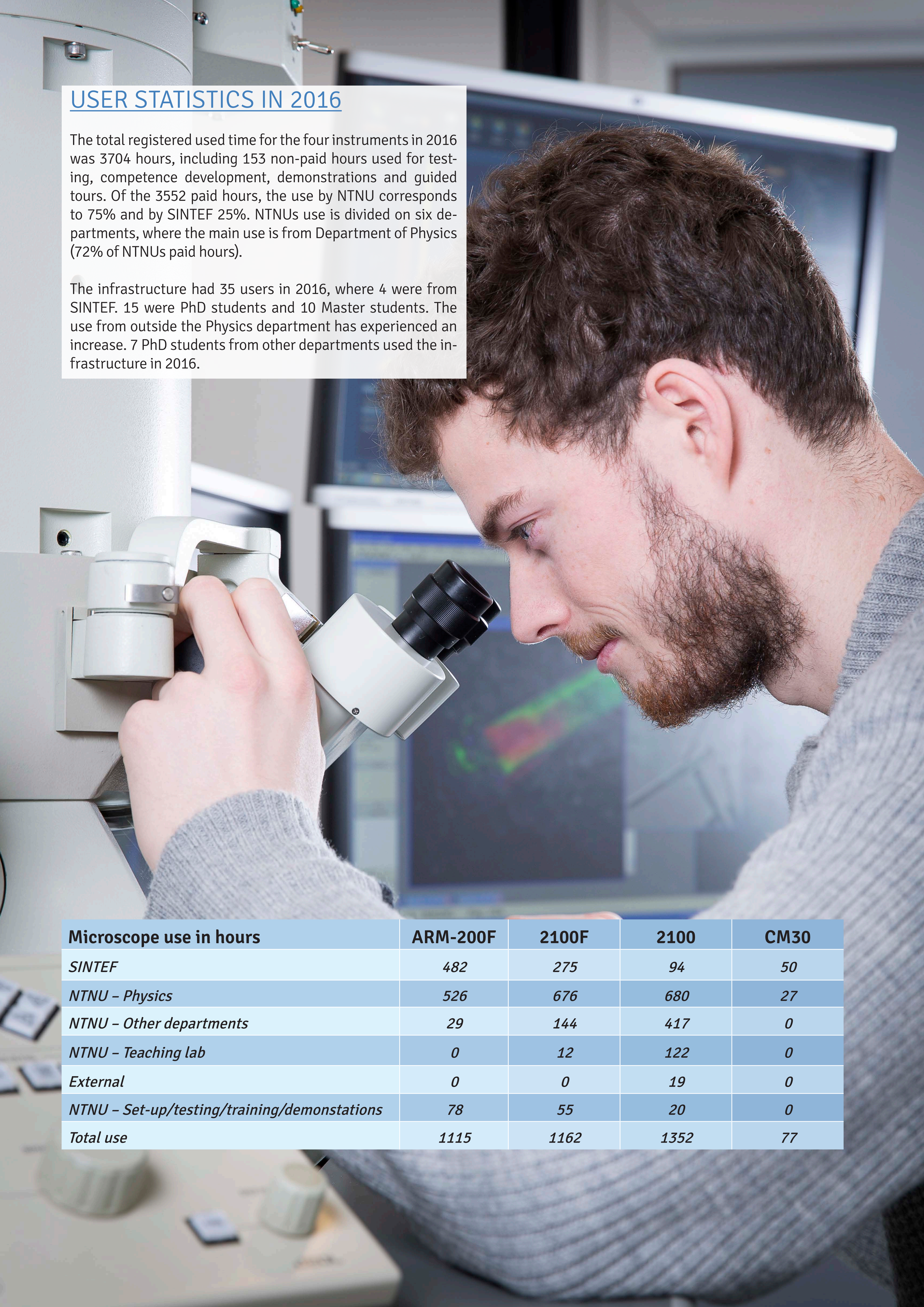
With the aberration corrected microscope, the cleanness requirements of the specimen and the holders increase. We have a dedicated room close to the microscope with general equipment such as a plasma cleaner, ozone cleaner, a stereomicroscope, user specimen storage and special holders that are used on 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. 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 (mainly used for the CM30) and access to an image plate reader.

USER STATISTICS IN 2016

The total registered used time for the four instruments in 2016 was 3704 hours, including 153 non-paid hours used for testing, competence development, demonstrations and guided tours. Of the 3552 paid hours, the use by NTNU corresponds to 75% and by SINTEF 25%. NTNUs use is divided on six departments, where the main use is from Department of Physics (72% of NTNUs paid hours).

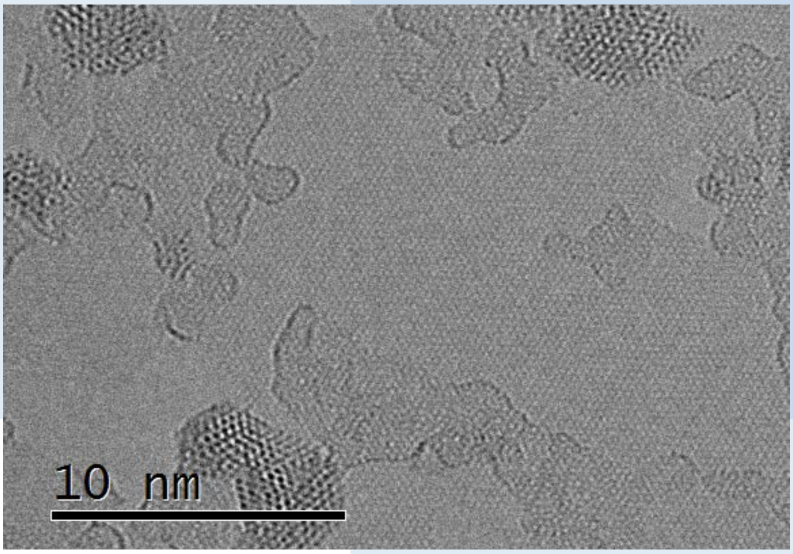
The infrastructure had 35 users in 2016, where 4 were from SINTEF. 15 were PhD students and 10 Master students. The use from outside the Physics department has experienced an increase. 7 PhD students from other departments used the infrastructure in 2016.

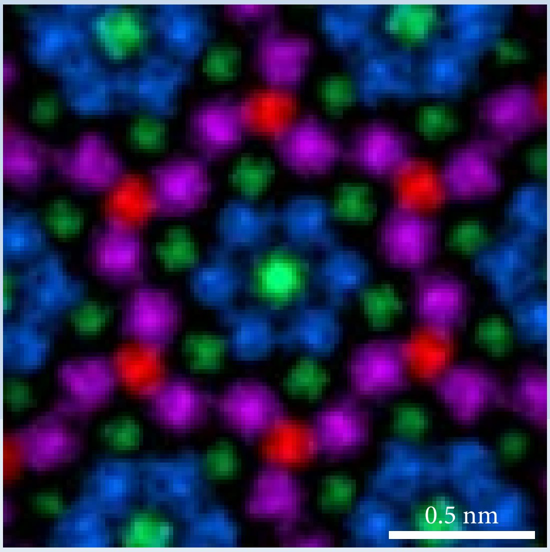


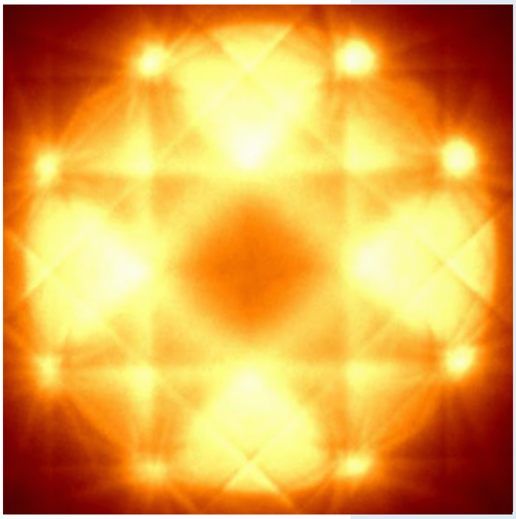
Microscope use in hours	ARM-200F	2100F	2100	CM30
SINTEF	*482*	*275*	*94*	*50*
NTNU – Physics	*526*	*676*	*680*	*27*
NTNU – Other departments	*29*	*144*	*417*	*0*
NTNU – Teaching lab	*0*	*12*	*122*	*0*
External	*0*	*0*	*19*	*0*
NTNU – Set-up/testing/training/demonstations	*78*	*55*	*20*	*0*
Total use	*1115*	*1162*	*1352*	*77*

ACTIVITIES IN 2016

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 already been important for the TEM Gemini Centre activities since the Centre was formed. Within these areas we see even potential for growth and an essential role for unsolved open 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 2016. The next sections describe these activities. Activities in aluminium alloy research are the largest.

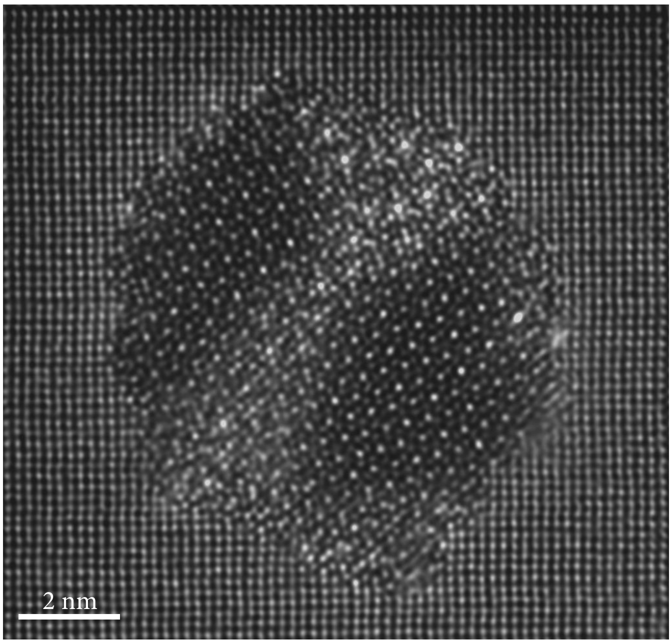
ALUMINIUM ACTIVITIES (LIGHT METALS)

The study of aluminium alloy systems 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 and finally, the bilateral competence building project 'The Japanese- Norwegian Al-Mg-Si precipitation 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.

Also the industrially initiated project RolEx (Smart 6xxx Alloy Development for Rolling and Extrusion) which is a collaboration between NTNU, SINTEF and Hydro Aluminium, is close to its end. Eva Anne Mørtsell finished her PhD in this project in September 2016 (see page 18). As the name indicates, this project focused on precipitation in extruded and rolled products with the goal of keeping/ improving properties while decreasing production costs. We have in this project ordered and screened a set of new alloys to test out the effect of different addition in very lean 6xxx alloys. Another project which will end in 2017, is the FRINATEK Research Council project called 'Fundamental investigations of precipitation in the solid state with focus on Al-based alloys'. Sigurd Wenner has a 3 year postdoc, and in 2017 he will work 50% at NTNU as postdoc and 50% as researcher in SINTEF until July 2017, before becoming a fulltime SINTEF researcher. In collaboration with SINTEF scientists, Sigurd is working on more fundamental alloy development and the more generic understanding of the connections between precipitates across several aluminium alloy systems. Alloys of the 6xxx, 7xxx and 2xxx series have been examined for different types of precipitates. STEM images from the new ARM microscope are a main contribution to this project. Two Master students finished their master thesis within aluminium alloys in 2016, these are Jonas K. Sunde, using scanning precession electron diffraction (SPED) on FRINATEK alloys, and Philip Østli, performing density functional theory studies of 2xxx alloy precipitates.

Two of the new 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, we are involved in the "lowest scale" of the multiscale activities, including TEM of precipitates, grain boundaries, precipitation free zones and interactions of them with dislocations in deformed, mostly industrial Al alloys. PhD student Emil Christiansen is working on TEM studies of deformed aluminium alloys in this project. In August 2016, Jonas Frafjord was hired to work on the modelling side on the lower scale. He will do density functional theory (DFT) in combination with other higher scale methods to explore dislocation movement in Al alloys. In the Manufacturing SFI project, headed by Sverre Guldbrandsen-Dahl from SINTEF RSM, joining of aluminium with other materials in multi-material products will be a central topic, and Tina Bergh started as a PhD student in August 2016 to study the microstructure of these joints.

Two KPN projects involving NTNU and SINTEF on aluminium research came up-to-speed during 2016. One is the project 'Fundamentals of intergranular corrosion in aluminium alloys' (FICAL) where John Walmsley is 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 substantial consortium of companies which represent the entire value chain, from alloy production to component manufacturer. The mechanisms of IGC will be studied at the nanometre-scale utilizing advanced laboratory infrastructure, especially TEM,



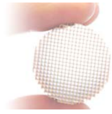
where NTNU and SINTEF will establish the detailed effects of composition and thermal history. Modelling tools will be developed to predict how the alloy microstructure and chemistry develop during processing and relate this to the IGC mechanisms that are observed in the experimental work. It is essential to place optimisation of corrosion resistance with mechanical and other alloy properties in establishing principles for optimising alloy design and performance. Adrian Lervik was hired as a PhD student in August 2016, and will focus particularly on TEM technique development and data-processing to obtain a quantitative understanding of nanoscale structure and chemistry around grain boundaries. The second KPN project 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. The project aims at providing new advances in experimental technologies, an experimental database, a set of modelling tools, a broad set of scientific publications and a multi-disciplinary research environment spanning from physics, through physical metallurgy, to computational mechanics. Jonas Sunde started as a PhD student on this project in August 2016.

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 working on measuring band gaps with electron energy loss spectroscopy. In 2016 we had one project student (Hogne Lysne) working on the deep level impurity approach doing Ag implantation into Si. The activities within intermediate band solar cell materials are in collaboration with Associate professor Turid W. Reenaas at DP. PhD Julie Stene Nilsen is part of the NANO2021 GRANASOL project (Low Cost, Ultra-High Efficiency Graphene/Nanowire Solar Cells). In her PhD the electronic properties of interfaces within a nanowire-based solar cell is central. PhD student Marina Jorge is working on CuO based solar cells. She will combine TEM and advanced photon emission in her PhD. Several of the external users and small projects were connected to solar cell materials or other forms of renewable energy. The TEM Gemini Centre is participating in the FME (Centre of Environmental Friendly Energy) on solar cells and project students, PhD students and SINTEF researcher within TEM are actively taking part in subprojects related to both conventional as well as third generation solar cells .

During the last five years, SINTEF has cooperated with a small Trondheim-based company, Integrated Solar, in two consecutive IPN projects. The first project ended in 2015, and the present project, "Improvement of efficiency in dual-junction solar cell", started last year. 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.

During the last 4-5 years, SINTEF has worked together with ELKEM and IFE in two consecutive IPN projects, "SiNODE" (2013–2015) and "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 behaviour of the



anode composites as a function of structure, morphology and cycling conditions.

SINTEF is also partner in two 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.

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 started 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.

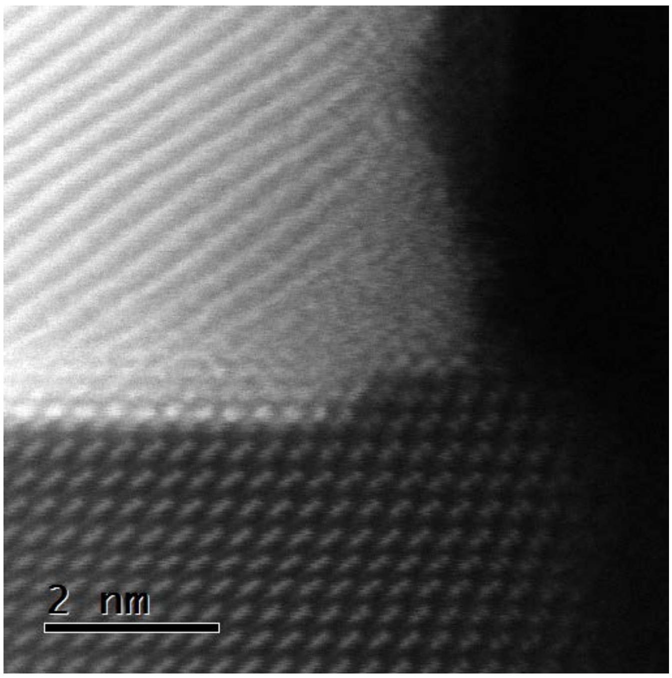
CHEMICAL ENGINEERING – CATALYSIS AND MEMBRANE MATERIALS

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 2016 include carbon nanofiber as oxygen reduction catalysts and in composite catalysts for Fisher Tropsch synthesis, carbon-ionic liquid supercapacitors and ceramic-ceramic hydrogen permeable membranes.

NANOTECHNOLOGY

TEM is an important tool within nanotechnology because of it resolving power for imaging and in analytical studies. The TEM Gemini Centre has several small and larger projects related to studying nanoparticles, 1D-nanostructures (i.e. nanowires, carbon nanotubes), thin films and there is an increasing interest in 2D-crystals such as graphene. NTNU NanoLab is an important partner, and NorFab-II is important for the TEM Gemini Centre activities within nanotechnology. PhD student Vidar Fauske, funded by NTNU NanoLab and based in the TEM Gemini Centre, defended his PhD June 2016 (see page 18). In his PhD Fauske combined advanced TEM techniques such as MEMS-based in-situ heating and precession diffraction with the complementary advanced techniques at NTNU NanoLab to understand and improve nanowire-based optolelectronic devices. PhD Aleksander Mosberg is funded by NTNU's "Enabling technologies: Nanotechnology" and



is based in the TEM Gemini Centre and mainly works in NanoLab's cleanroom. Mosberg's PhD is focused on using focus ion beam (FIB) for large scale nanostructuring. TEM is used to understand how the ion beam alters the materials. The Centre has close ties with the Norwegian PhD Network on Nanotechnology for Microsystems. We are grateful that the network sponsors travels and research exchanges for PhD students working within the TEM Gemini Centre during 2016.

The Norwegian PhD Network on Nanotechnology for Microsystems funded PhD student Magnus Nord, who defended his PhD thesis in the field of oxide electronics in December 2016 (see page 18). Magnus was investigating epitaxial complex oxide thin films of $LaSrMnO_3$ with advanced TEM, utilizing the unique strength of the ARM combined with a state-of-the-art energy loss spectrometer. This was a project in collaboration with Prof. Thomas Tybell at the Department of Electronic Systems. In 2016 we had Master students working with promising nanomaterials. Theodor Secanell Holstad did his Master on TEM of $BaTiO_3$/$LaSrMnO_3$/$SrTiO_3$ thin films, studying domains and dislocations in the thin films. Trond Henninen studied graphene he made in NTNU NanoLab. We work close together with Profs. Helge Weman and Bjørn-Ove Fimland at the Department of Electronic Systems. Their group is developing optoelectronic devices as solar cells and LEDs based on III-V nanowires. As can be seen from the publication list, many nano-related projects have taken advantage of the new TEM infrastructure and we expect that the amount of TEM work dedicated to Nanotechnology will increase further.

ADVANCED TEM DATA PROCESSING

Handling of ever larger data sets is in many research and technology areas the real new frontier. Beside size, the data sets become also more complex and multidimensional. Still transparency to verify scientific findings and a dynamic structure to incorporate new developments should be maintained. PhD students Magnus Nord and Vidar Fauske activity contributed over longer time to the open-source Python library HyperSpy (hyperspy.org) that has proven to be a powerful tool to tackle the above mentioned challenges. Their contributions included specific tools to handle spectral data sets, contributions to the underlying base structure and giving workshops (including in the TEM Gemini Centre and at SCANDEM). They developed items, such as an interactive user interface, that make the platform accessible to a larger group of TEM users. Other PhD and master students within the TEM Gemini Centre are now daily using the platform and contributing to the development of HyperSpy. It is great to see the rapid progress in advanced data processing within the Centre and how our students can contribute to the open-source movement and collaborate themselves internationally.

At the TEM Gemini Centre HyperSpy is becoming the main platform used to handle energy-dispersive and electron energy loss spectroscopy spectral imaging data sets. It is also a crucial tool in getting more out of scanning precession electron diffraction (SPED). The investment in the SPED hardware was made a few years ago, but through better data handling routines recently developed, as well as support from Paul Midgley's group in Cambridge, SPED became more powerful and more accessible to a large user group including master and PhD students, postdocs and SINTEF researchers working on a broad range of projects. A specific aspect of advanced data handling is machine learning applied to TEM data, which is now actively used and explored further.

ACTIVE PROJECTS IN 2016

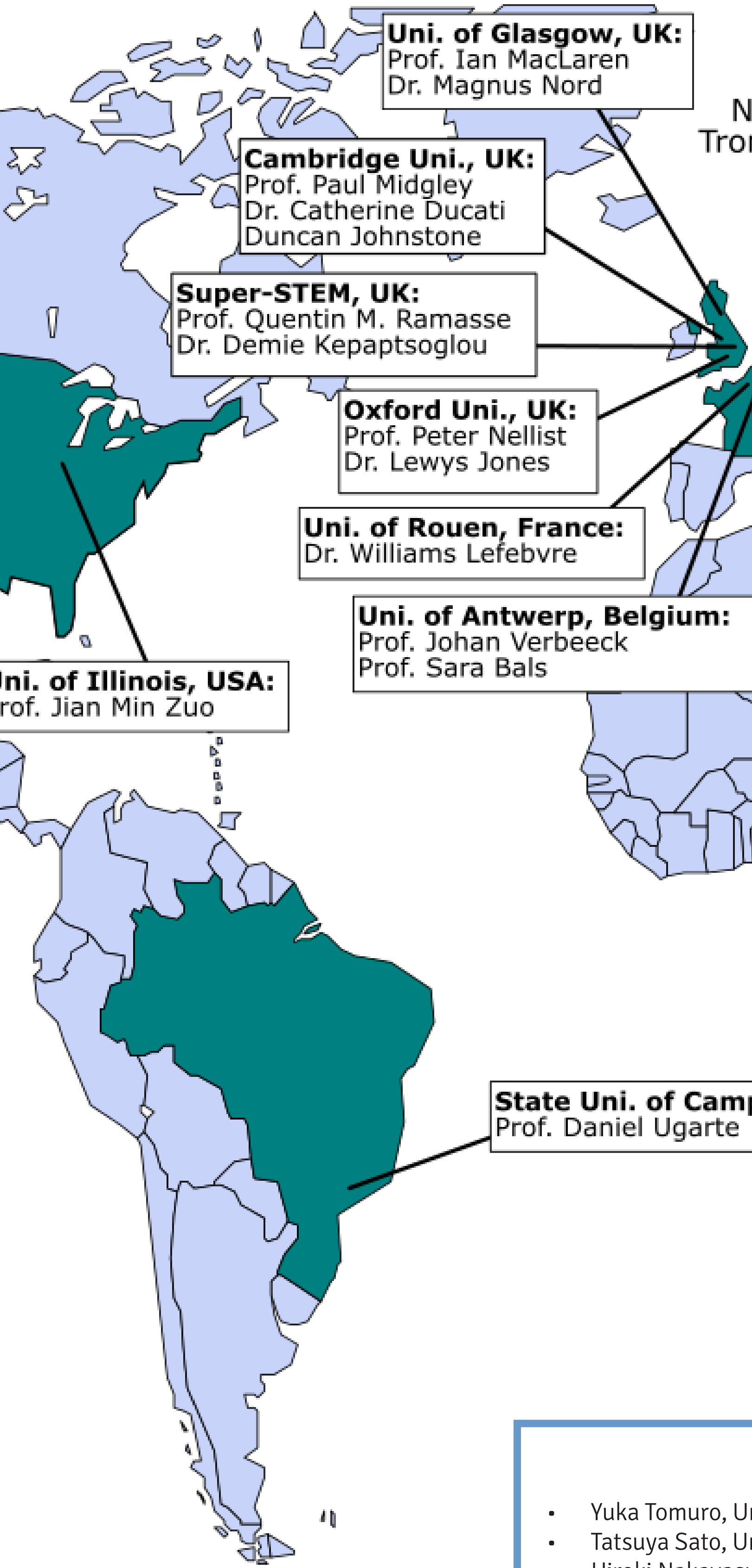
The table below shows the larger projects connected to TEM within the Gemini Centre. They are listed by funding type, title, duration and research partners. A number of smaller projects, both academic and with direct industrial support, are not listed here.

<table>
<tr><th>Project type</th><th>Project title</th><th>Involved with TEM</th><th>Duration</th></tr>
<tr><td>SFI</td><td>CASA – Centre for Advanced Structural Analysis</td><td>1–2 PhDs, SINTEF</td><td>2015–2023</td></tr>
<tr><td colspan="4">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</td></tr>
<tr><td>SFI</td><td>SFI Manufacturing</td><td>1 PhD, SINTEF</td><td>2015–2023</td></tr>
<tr><td colspan="4">Partners: SINTEF SRM, SINTEF, NTNU, Benteler, Brødrene AA, Ekornes, GKN Aerospace, Hexagon composites, Kongsberg Automotive, Nammo, Raufoss Neuman, Plastal, Plasto, Rolls Royce, Teeness, Hybond</td></tr>
<tr><td>SFI</td><td>Industrial Catalysis Science and Innovation for a Competitive and Sustainable process Industry (iCSI)</td><td>SINTEF</td><td>2015–2023</td></tr>
<tr><td colspan="4">Partners: Yara Norge, K.A. Rasmussen, Dynea INOVYN Norge, Haldor Topsøe AS</td></tr>
<tr><td>FME</td><td>FME sol – Solar United</td><td>1 Postdoc, SINTEF</td><td>2009–2017</td></tr>
<tr><td colspan="4">Partners: IFE, NTNU, SINTEF, the University of Oslo, CleanSi, Dynatec, Elkem Solar, Mosaic, Norsun, Norwegian Crystals, Quartz Corp, REC Silicon, REC Solar, Semilab</td></tr>
<tr><td>IPN/BIA</td><td>Smart 6xxx Alloy Development for Rolling and Extrusion (RolEx)</td><td>2 PhDs, SINTEF</td><td>2012–2016</td></tr>
<tr><td colspan="4">Partners: Hydro, NTNU, SINTEF</td></tr>
<tr><td>KPN/BIA</td><td>Aluminium alloys with mechanical properties and electrical conductivity at elevated temperatures (AMPERE)</td><td>1–2 PhDs, SINTEF</td><td>2015–2020</td></tr>
<tr><td colspan="4">Partners: NTNU, SINTEF, Hydro, Nexans, Raufoss Neuman, SAPA, Gränges</td></tr>
<tr><td>KPN/BIA</td><td>Fundamentals of Intergranular Corrosion in Aluminum Alloys (FICAL)</td><td>1 PhD, SINTEF</td><td>2015–2020</td></tr>
<tr><td colspan="4">Partners: NTNU, SINTEF, Hydro, Benteler, Steertec, SAPA, Gränges</td></tr>
<tr><td>FP/FRINATEK</td><td>Fundamental investigations of precipitation in the solid state with focus on Al-based alloys</td><td>1 PhD, 1 Postdoc, SINTEF</td><td>2013–2017</td></tr>
<tr><td colspan="4">Partners: NTNU, SINTEF</td></tr>
<tr><td>FP/ENERGIX</td><td>High Efficiency Quantum Dot Intermediate Band Solar Cells (HighQ-IB)</td><td>1 PhD, SINTEF</td><td>2012–2017</td></tr>
<tr><td colspan="4">Partners: NTNU, SINTEF</td></tr>
<tr><td>FP/FRINATEK</td><td>Oxide Intermediate Band Photovoltaics (Ox-IB)</td><td>1 PhD, SINTEF</td><td>2015–2020</td></tr>
<tr><td colspan="4">Partners: NTNU</td></tr>
<tr><td>IFP/Nano2021</td><td>GRANASOL – Low Cost, Ultra-High Efficiency Graphene Nanowire Solar Cells</td><td>1 PhD</td><td>2014–2018</td></tr>
<tr><td colspan="4">Partners: NTNU, Sejong University, Aalto University, CRAYONANO AS</td></tr>
<tr><td>INTPART</td><td>Norwegian-Japanese Aluminium alloy Research and Education Collaboration (NJALC)</td><td>Travel, exchange students</td><td>2015–2018</td></tr>
<tr><td colspan="4">Partners: NTNU, SINTEF, Hydro, University of Toyama, Tokyo Institute of Technology</td></tr>
<tr><td>FP/PETROMAKS2</td><td>Fatigue and hydrogen degradation of steels (HyF-Lex)</td><td>SINTEF</td><td>2015–2018</td></tr>
<tr><td colspan="4">Partners: NTNU, SINTEF</td></tr>
<tr><td>FP/GASSMAKS</td><td>Development of materials and catalysts for compact reformers</td><td>SINTEF</td><td>2014–2017</td></tr>
<tr><td colspan="4">Partners: NTNU, SINTEF</td></tr>
<tr><td>IPN/BIA</td><td>Integrated Hardening and Sheet Press-forming of Aluminium (I-Pal)</td><td>SINTEF</td><td>2016–2019</td></tr>
<tr><td colspan="4">Partners: SINTEF, Hydro, SAPA, Raufoss Technology Neumann, AP&T, SINTEF Raufoss Manufacturing AS</td></tr>
</table>

PEOPLE WORKING IN THE TEM GEMINI CENTRE DURING 2016

Group picture TEM Gemini Centre, January 2016 Photo: Ole Morten Melgård

Randi Holmestad (Professor, DP, NTNU / Leader TEM Gemini Centre)
John Walmsley (Senior research scientist, SINTEF and Professor II, DP, NTNU)
Ton van Helvoort (Professor, DP, NTNU)
Knut Marthinsen (Professor, DMSE, NTNU)
Yanjun Li (Professor, DMSE, NTNU)
Per Erik Vullum (Research scientist, SINTEF and Assoc. Professor 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)
Sigurd Wenner (Postdoc, DP, NTNU)
Vidar Tonaas Fauske (PhD student, DP, NTNU)
Magnus Nord (PhD student, DP, NTNU)
Eva Anne Mørtsell (PhD student, DP, NTNU)
Maryam Vatanparast (PhD student, DP, NTNU)
Julie Stene Nilsen (PhD student, DP, NTNU)
Marina Jorge (PhD student, DP, NTNU)
Aleksander Mosberg (PhD student, DP, NTNU)
Emil Christiansen (PhD student, DP, NTNU)
Adrian Lervik (PhD student, DP, NTNU)
Tina Bergh (Master/ PhD student, DP, NTNU)
Jonas Sunde (Master /PhD student, DP, NTNU)
Andreas Garmannslund (Master student, DP, NTNU)
Trond R. Henninen (Master student, DP, NTNU)
Theodor Secanell Holstad (Master student, DP, NTNU)
Philip Østli (Master student, DP, NTNU)
Hogne Lysne (Project student, DP, NTNU)
Øyvind Paulsen (Project student, DP, NTNU)
Martin Rimbereid Vik (Project student, DP, NTNU)
Jochen Busam (Master student, DP, NTNU)
Ingrid Marie Andersen (Project student, DP, NTNU)
Håkon Wiik Ånes (Project student, DP, NTNU)
Steinar Myklebost (Project student, DP, NTNU)
Johannes Bogen (Project student, DP, NTNU)

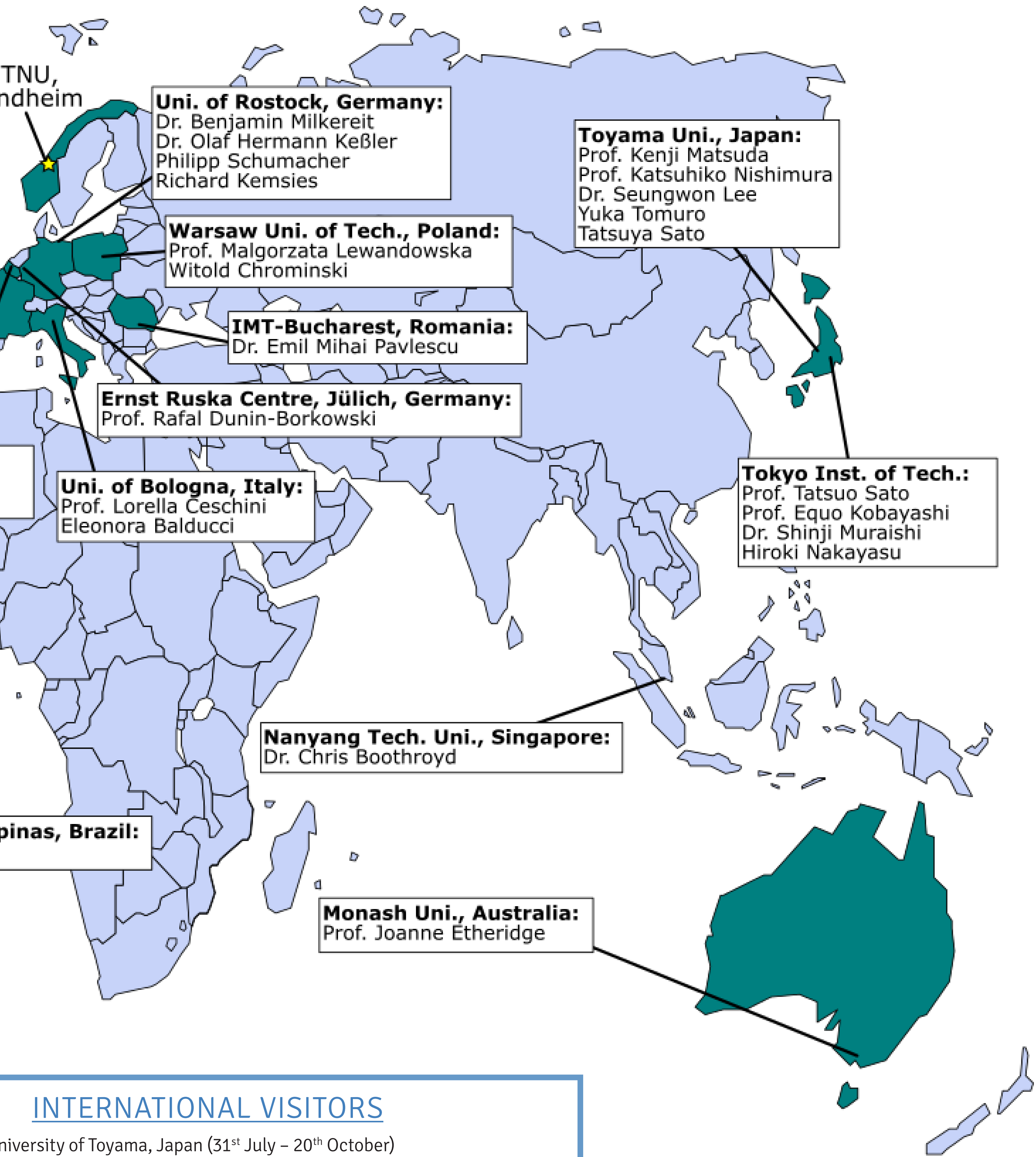


INTERNATIONAL COLLABORATION

As can be seen from the map and in the publication list, the TEM Gemini Centre has ties to many research institutions and researchers abroad. Some are long term collaborations, others are new initiatives. The map illustrates the direct scientific collaboration and initiated contact with groups/ people across the world. We had several visitors from abroad applying and using the TEM facility in Trondheim. An example of a long-standing international cooperation is the contact with two universities in Japan, now supported by the INTPART project (see page 20). In 2017 Jian Min Zuo will be visiting the group for three months as an Onsager professor at NTNU. People from the group were chairs and contributors to international conferences and acted as PhD examiners abroad. The aluminium alloy related collaborations with Eleonora Balducci at University of Bologna, Italy, and the group of Benjamin Milkereit in Rostock, Germany, are established and directly operative in 2016.

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

- Yuka Tomuro, U
- Tatsuya Sato, U
- Hiroki Nakayasu
- Seungwon Lee,
- Ian MacLaren, U
- Maria Varela, U
- Magnus Garbre
- Patricia Donnad
- Zhihong Jia, Ch
- Lewys Jones, U
- Duncan Johnsto



INTERNATIONAL VISITORS

niversity of Toyama, Japan (31st July – 20th October)
niversity of Toyama, Japan (31st July – 20th October)
ı, Tokyo University of Technology, Japan (1st August – 31st January 2017)
University of Toyama, Japan (3rd August – 20th October)
Jniversity of Glasgow, UK (14th – 16th December)
niversidad Complutense Madrid, Spain (14th – 16th December)
cht, Lindköping University, Sweden (13th – 16th December)
lieu, CNRS-Grenoble INP-UJF, France (28th September – 1st October)
ongqing University, China (27th September – 3rd October)
niversity of Oxford, UK (7th – 10th June)
one, University of Cambridge, UK (7th – 10th June)

PHD DISSERTATIONS

VIDAR FAUSKE

Vidar Tonaas Fauske defended his PhD thesis on the 29th of June 2016. The title of the thesis is 'Electron Microscopy Based Characterization of Semiconductor Nanowires'. Vidar studied the interface between III-V nanowires and graphene using high resolution scanning transmission electron microscopy (STEM) to better understand and improve nanowire-based III-V optoelectronics. He did an elaborated in-situ STEM study on the solid state exchange of GaAs by Au revealing both the kinetics and the dynamics of this exchange reaction. Furthermore, he performed in-situ heating, electrical and mechanical studies in the focus ion beam (FIB). He was (co)-author on nine papers by the end of his PhD, including five Nano Letters. Together with Magnus Nord he established working routines and explored the boundaries of the new NORTEM microscopes that became available during their PhDs. Many of the routines and software tools developed during Fauske's PhD are still daily used within the TEM Gemini Center. Fauske's PhD was financed by NTNU NanoLab and his main duty work there was related to training new FIB users and exploring the capacities of FIB. His co-supervisor was Prof. Helge Weman at the Department of Electronic Systems. After his PhD became a postdoc at Simula Research Laboratory developing Jupyter for interactive, shared scientific computing.

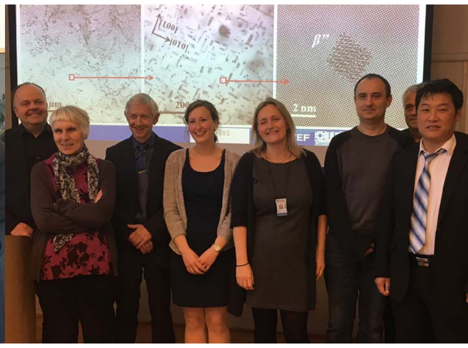
EVA ANNE MØRTSELL

Eva Anne Mørtsell defended her PhD thesis on the 30th of September 2016. The title of the thesis is 'Precipitation in multicomponent, lean Al-Mg-Si alloys – A transmission electron microscopy study'. Eva was financed through the RolEx project, in the BIA programme from the Research Council, with Hydro as a project owner, in collaboration with NTNU and SINTEF. The thesis contains seven papers, covering use of advanced TEM techniques as HAADF-STEM and EELS to explore small additions of Cu, Ag and GE to lean alloys and has demonstrated that extrudability of 6xxx alloys can be improved by smart alloy design. Randi Holmestad was her main supervisor, with co-supervisors Jostein Røyset from Hydro and Calin D. Marioara from SINTEF. The presented trial lecture was entitled 'In-situ transmission electron microscopy (TEM) studies of microstructure phenomena in metals and alloys'. Eva is now working as a postdoc, doing mainly TEM, in the Metallurgy group in the Department of Materials Science and Engineering, NTNU.

MAGNUS NORD

Magnus K. Nord defended his thesis on the 15th of December 2016. The title of the thesis is 'EELS and STEM studies of perovskite oxide heterostructures'. Magnus was financed through the Norwegian PhD Network on Nanotechnology for Microsystems. Randi Holmestad was his main supervisor, with co-supervisors professor Thomas Tybell from the Electronics department and Per Erik Vullum (NTNU/SINTEF). Magnus' thesis includes 5 papers, mainly on advanced EELS and STEM analysis studying epitaxial complex oxide thin films (LSMO/LFO/STO) and interfaces. He contributed to method development, exploring advanced possibilities of the new ARM microscope in Trondheim. The presented trial lecture was entitled 'Compressed sensing used in (transmission) electron microscopy'. Magnus is now working with STEM and pixelated detectors at University of Glasgow as a postdoc in the group of Professor Ian MacLaren.

From the left, Opponent Martien den Hertog, CNRS-NEEL, Grenoble, France; Administrator Dag Werner Breiby; PhD candidate Vidar T. Fauske, Opponent Jakob Wagner, DTU-CEN, Copenhagen, Denmark, Supervisor Ton van Helvoort.



From the left, Co-supervisor Jostein Røyset; Opponent Patricia Donnadieu, CNRS-Grenoble INP-UJF, France; Prof. Knut Martinsen; PhD candidate Eva Anne Mørtsell; Supervisor Randi Holmestad; Co-supervisor Calin D. Maroiara; Administrator Ragnvald Mathiesen; Opponent Zhihong Jia, Chongqing University, China.

From the left, Co-supervisor Thomas Tybell; Opponent Maria Varela, Universidad Complutense de Madrid; Supervisor Randi Holmestad; PhD candidate Magnus K. Nord; Opponent Magnus Garbrecht, Linköping University, Sweden; Co-supervisor Per Erik Vullum; Administrator Ragnvald Mathiesen.

Nordic Microscopy Society

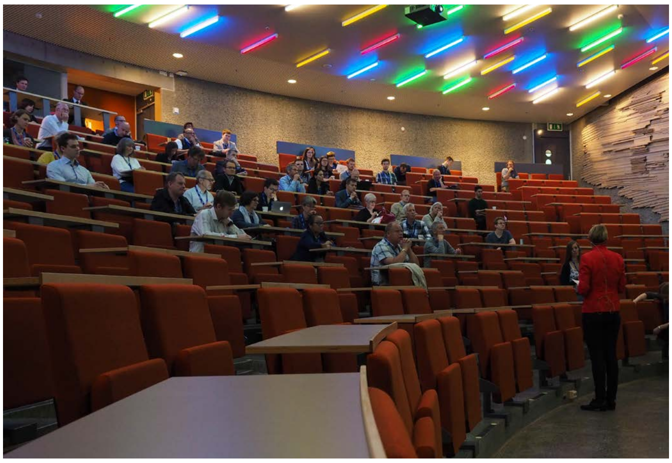
SCANDEM 2016

JUNE 7–10

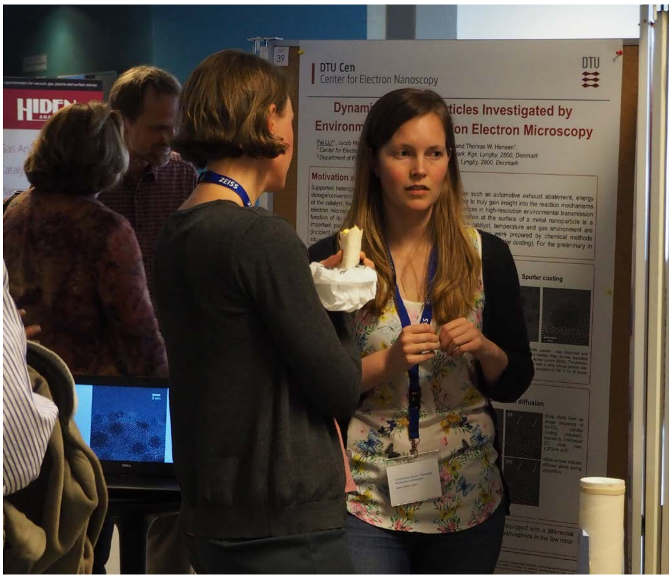
NTNU Trondheim

The 67th SCANDEM conference, the annual conference in the Nordic Microscopy Society, was organized at NTNU in Realfagbygget, Trondheim, Norway, 7.–10. June 2016. The meeting had not been in Trondheim since 1995! Professor Randi Holmestad from Department of Physics, NTNU, was the chair of the organizing committee, and Professor Johannes van der Want from the Medical faculty was the co-chair. Randi and Johannes were responsible for the materials and the life science sessions, respectively. The conference home page with all details is found here:

https://www.ntnu.edu/physics/scandem2016.



There were three splendid invited plenary lectures (Peter Peters from Maastricht, Paul Midgley from Cambridge and Sara Bals from Antwerp). We had ten sessions in two categories (materials and life sciences, run in parallel) with 1–2 invited speakers in each session. The ten sessions were functional materials, structural materials, nano-materials, instrumentation, geology, data analysis, bio-nano, cellular imaging, neuroscience and correlative microscopy. The cross-disciplinary character of the meeting was inspiring. The session chairs came from different Trondheim departments and stimulated a closer local cooperation between different fields and departments.



Traditionally the SCANDEM conferences have attracted many companies to show equipment and their latest developments. This year there were 24 companies that participated in the exhibition. It is impressive that the companies participated with so many people and lots of equipment – all the way to Trondheim for the entire meeting! The exhibition was open for two days.

The conference had a total of 200 participants from 16 countries, 50 participants were exhibitors, and about 50 were from NTNU. There were 62 oral presentations and 37 posters were presented. The lectures held a very high level. Further there were guided tours to different local labs (e.g. SEM, TEM, NanoLab, biophysics, geology and several labs at the medical faculty). Furthermore, there were four workshops organized the day prior to the meeting. These were a hands-on HyperSpy workshop, an in-situ heating workshop organized by DENS Solutions, an EBSD workshop and a workshop on 3D SEM.

The social program consisted of a welcome reception Tuesday evening, organ concert in Nidarosdomen (Trondheim municipality) with a reception at the visitor center on Wednesday evening and a conference dinner on Thursday night in Banksalen. The meeting got financial support from two programmes in the Norwegian Research Council, and partly due the higher than expected number of participants and exhibitor numbers, a positive financial result was obtained. Overall SCANDEM 2016 was a big success for all involved.

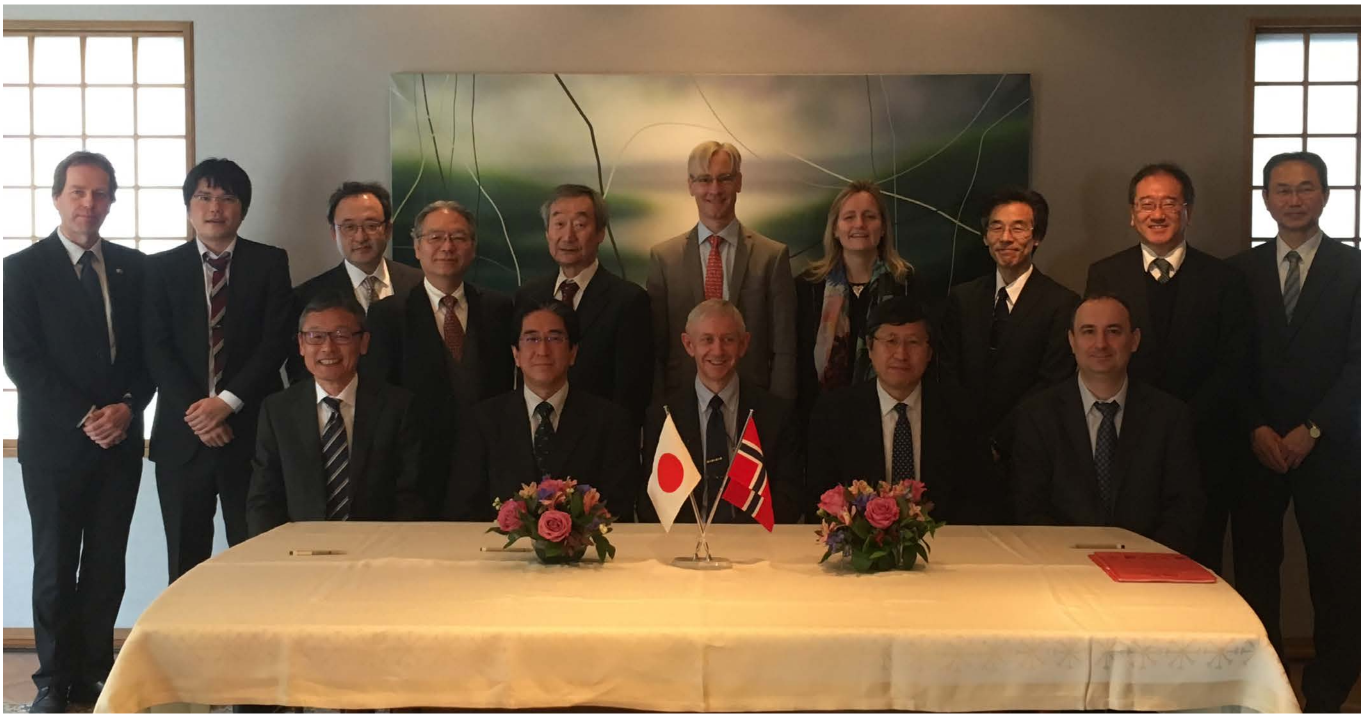
INTPART PROJECT WITH JAPAN

In 2016 we signed a new 3-year international Partnership (INTPART) project from the Norwegian Research Council and the Norwegian Centre for International Cooperation in Education called "Norwegian-Japanese Aluminium alloy Research and Education Collaboration". 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 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. The consortium agreement was signed at the Norwegian Embassy in Tokyo in March 2016 with participation from the Norwegian Ambassador in Japan. During this visit, we had the first workshop of this program, where 65 FYSMAT students from NTNU on excursion participated. We here discussed issues as energy, gender equality and study environment in general.

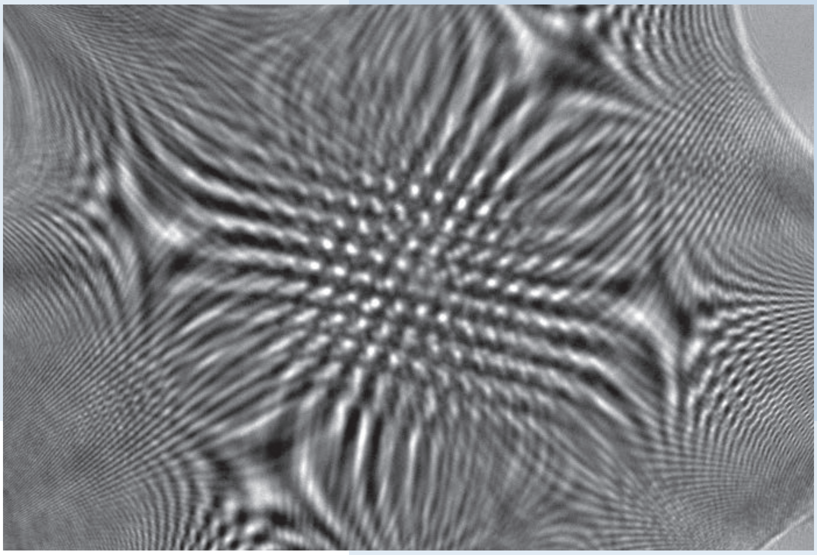
Tina Bergh and Jonas Sunde posing in front of the JEOL JEM-F200 at the JEOL factory in Akishima.

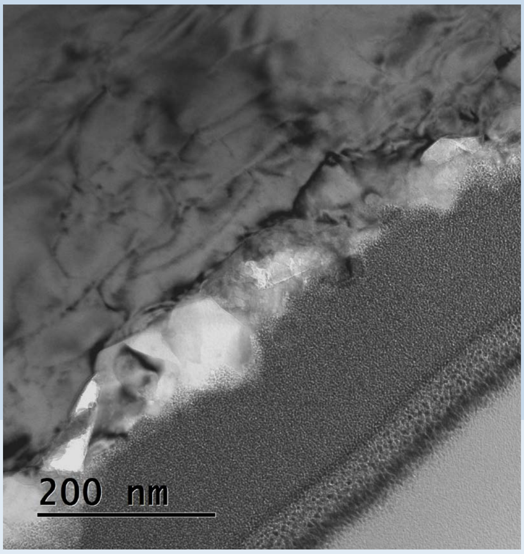
In August, three students from Japan (Master students Yuka Tomuro and Tatsuya Sato from University of Toyama and PhD student Hiroki Nakayasu from Tokyo Institute of Technology) had all 1 month internships in Hydro at Sunndalsøra. After this they stayed 2 months (Yuka and Tatsuya) and 5 months, respectively, at the departments of Physics and of Materials Science and Engineering at NTNU to do TEM. In addition, assistant professor Seungwon Lee from University of Toyama stayed 3 months at Department of Physics.

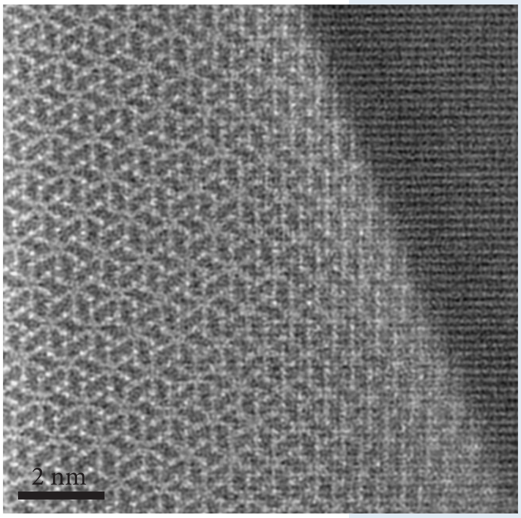
In October a delegation of ten Norwegians (Calin (SINTEF), Takeshi (Hydro), Knut, Randi, Jonas S, Jonas F, Adrian, Tina, Emil, Øyvind (NTNU)) went to Japan for a week to visit Tokyo Institute of Technology and Toyama University. At Toyama we attended a two days forum and organized a one day INTPART workshop. In addition we visited the JEOL factory in Akishima, close to Tokyo.

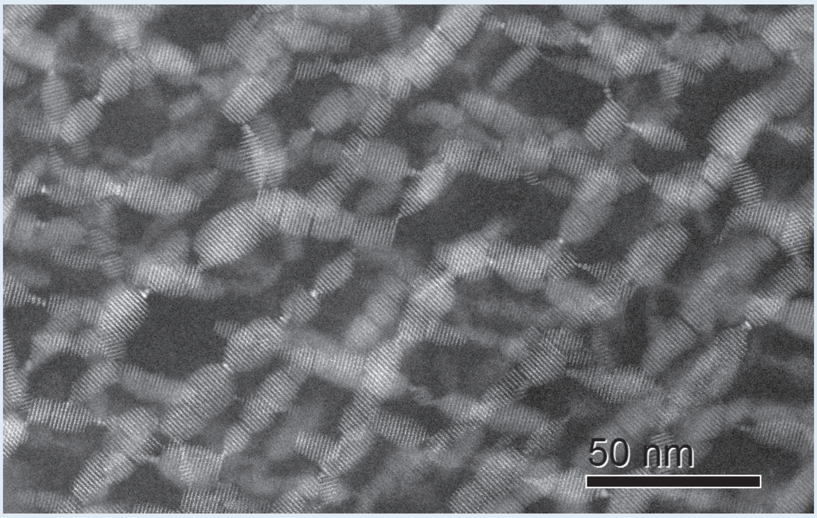

Signing of the INTPART consortium agreement at the Norwegian Embassy in Tokyo, March 2016.

SELECTED
SCIENTIFIC PAPERS









Ultramicroscopy 169 (2016) 98–106

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journal homepage: www.elsevier.com/locate/ultramic

Assessing electron beam sensitivity for $SrTiO_3$ and $La_{0.7}Sr_{0.3}MnO_3$ using electron energy loss spectroscopy

Magnus Nord [a,*], Per Erik Vullum [a,c], Ingrid Hallsteinsen [b], Thomas Tybell [b], Randi Holmestad [a]

[a] Department of Physics, NTNU, Trondheim, Norway
[b] Department of Electronics and Telecommunications, NTNU, Trondheim, Norway
[c] Materials and Chemistry, SINTEF, Trondheim, Norway

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ABSTRACT

Thresholds for beam damage have been assessed for $La_{0.7}Sr_{0.3}MnO_3$ and $SrTiO_3$ as a function of electron probe current and exposure time at 80 and 200 kV acceleration voltage. The materials were exposed to an intense electron probe by aberration corrected scanning transmission electron microscopy (STEM) with simultaneous acquisition of electron energy loss spectroscopy (EELS) data. Electron beam damage was identified by changes of the core loss fine structure after quantification by a refined and improved model based approach. At 200 kV acceleration voltage, damage in $SrTiO_3$ was identified by changes both in the EEL fine structure and by contrast changes in the STEM images. However, the changes in the STEM image contrast as introduced by minor damage can be difficult to detect under several common experimental conditions. No damage was observed in $SrTiO_3$ at 80 kV acceleration voltage, independent of probe current and exposure time. In $La_{0.7}Sr_{0.3}MnO_3$, beam damage was observed at both 80 and 200 kV acceleration voltages. This damage was observed by large changes in the EEL fine structure, but not by any detectable changes in the STEM images. The typical method to validate if damage has been introduced during acquisitions is to compare STEM images prior to and after spectroscopy. Quantifications in this work show that this method possibly can result in misinterpretation of beam damage as changes of material properties.

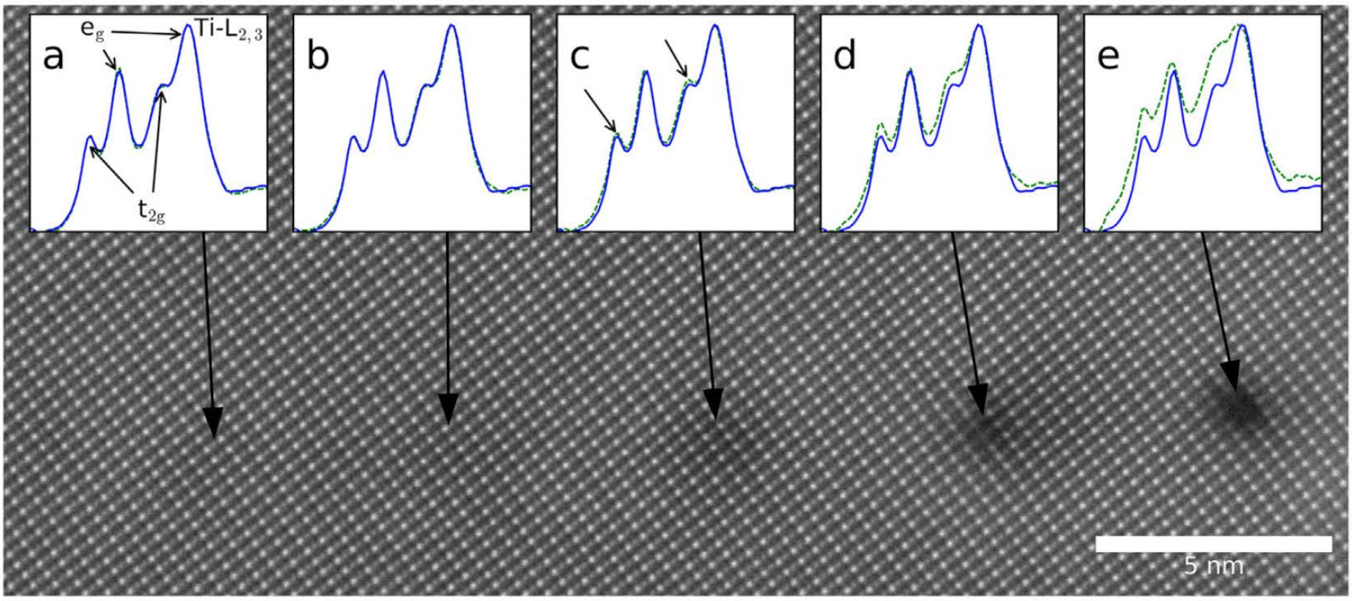

Fig. 3. STEM-HAADF image of STO after beam exposure at 200 kV in the points highlighted by the arrows. The leftmost area (a) having received the least amount of electron dose, and the rightmost (e) the most. After the beam exposures, a line scan was done across all the exposed points. Insets show the Ti-$L_{2,3}$ edge from an unexposed area (solid blue) and the exposed points (dashed green). The insets show an increasing amount of sample damage with increasing electron dose, as expected. No changes in HAADF intensity and the Ti-$L_{2,3}$ edge are observed in (a) and (b). In (c) there are some subtle changes, indicated by the arrows, in the form of increasing intensity in the t_{2g} peaks, and a small change in the HAADF intensity. (d) and (e) show larger changes in both the Ti-$L_{2,3}$ and the HAADF intensity. (For interpretation of the references to color in this figure caption, the reader is referred to the web version of this paper.)

Scripta Materialia 118 (2016) 5–8

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Regular article

Accurately measured precipitate–matrix misfit in an Al–Mg–Si alloy by electron microscopy

Sigurd Wenner *, Randi Holmestad

Department of Physics, NTNU, Høgskoleringen 5, NO-7491 Trondheim, Norway

ARTICLE INFO

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Misfit strain

ABSTRACT

The age-hardenable Al–Mg–Si alloy system is strengthened by needle-shaped coherent β″ phase precipitates. Using distortion-corrected high-resolution scanning transmission electron microscopy images, we measure the considerable misfit between β″ particles and the Al matrix. The β″ phase is found to adapt its lattice parameters to the particle shape, distributing the strain in the Al matrix evenly in its cross-sectional plane. The measured misfits give a good match to reported atomistic simulations for a β″ phase with composition $Mg_5Al_2Si_4$.

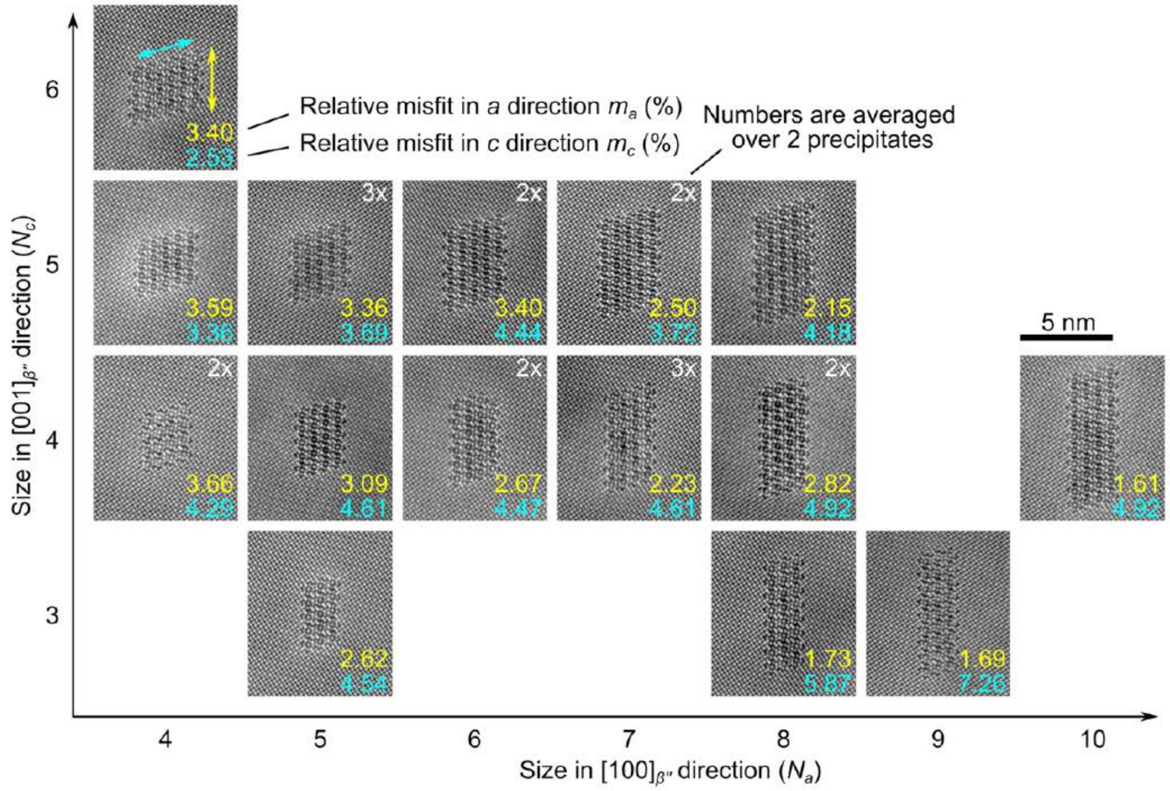

Fig. 2. The collection of precipitate shapes found in this study, listing the measured misfit with Al. If more than one precipitate is found, average numbers are given. The size units are defined in Fig. 1(a).

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Elemental electron energy loss mapping of a precipitate in a multi-component aluminium alloy

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ABSTRACT

The elemental distribution of a precipitate cross section, situated in a lean Al-Mg-Si-Cu-Ag-Ge alloy, has been investigated in detail by electron energy loss spectroscopy (EELS) and aberration corrected high angle annular dark field scanning transmission electron microscopy (HAADF-STEM). A correlative analysis of the EELS data is connected to the results and discussed in detail. The energy loss maps for all relevant elements were recorded simultaneously. The good spatial resolution allows elemental distribution to be evaluated, such as by correlation functions, in addition to being compared with the HAADF image.

The fcc-Al lattice and the hexagonal Si-network within the precipitates were resolved by EELS. The combination of EELS and HAADF-STEM demonstrated that some atomic columns consist of mixed elements, a result that would be very uncertain based on one of the techniques alone. EELS elemental mapping combined with a correlative analysis have great potential for identification and quantification of small amounts of elements at the atomic scale.

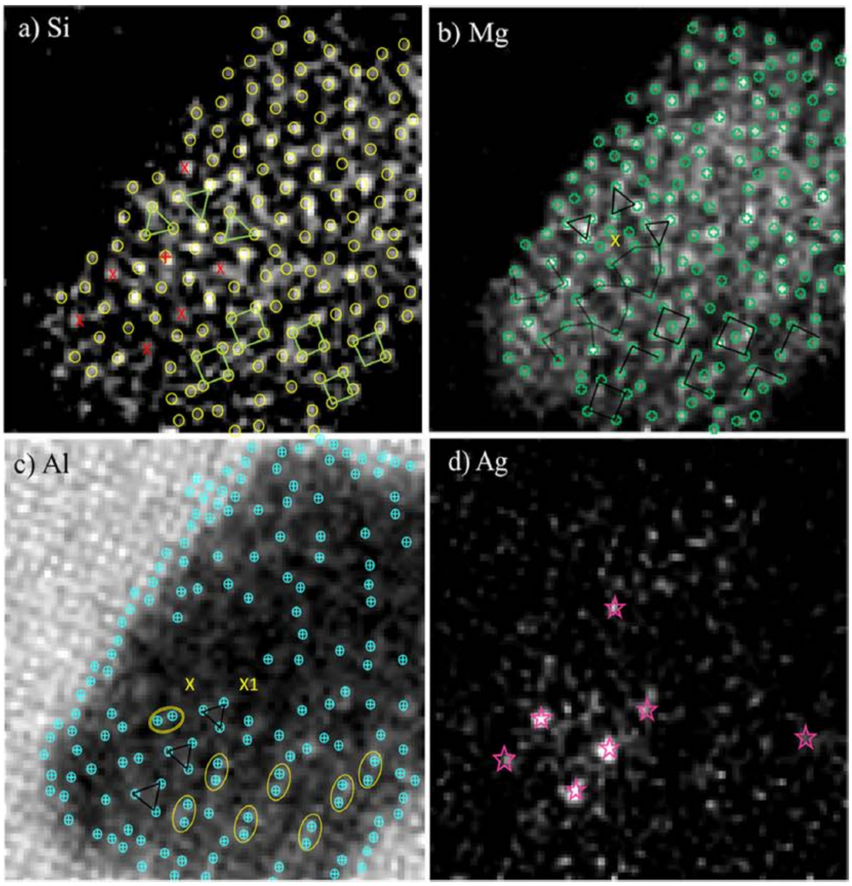

Fig. 5. EELS elemental maps of (a) The Si-K edge, (b) the Mg-K edge, (c) the Al-K edge and (d) the Ag-M edge with overlays of specific details in the atomic columns. See text for detailed explanations.(For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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In Situ Heat-Induced Replacement of GaAs Nanowires by Au

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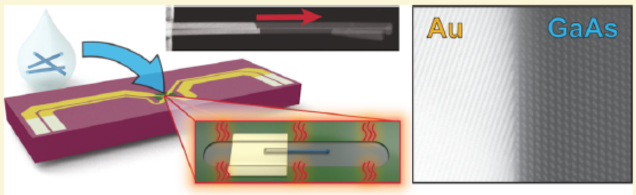
Supporting Information

ABSTRACT: Here we report on the heat-induced solid-state replacement of GaAs by Au in nanowires. Such replacement of semiconductor nanowires by metals is envisioned as a method to achieve well-defined junctions within nanowires. To better understand the mechanisms and dynamics that govern the replacement reaction, we performed in situ heating studies using high-resolution scanning transmission electron microscopy. The dynamic evolution of the phase boundary was investigated, as well as the crystal structure and orientation of the different phases at reaction temperatures. In general, the replacement proceeds one GaAs(111) bilayer at a time, and no fixed epitaxial relation could be found between the two phases. The relative orientation of the phases affects the replacement dynamics and can induce growth twins in the Au nanowire phase. In the case of a limited Au supply, the metal phase can also become liquid.



KEYWORDS: *Nanowire, solid-state replacement, in situ, annealing, GaAs, Au*

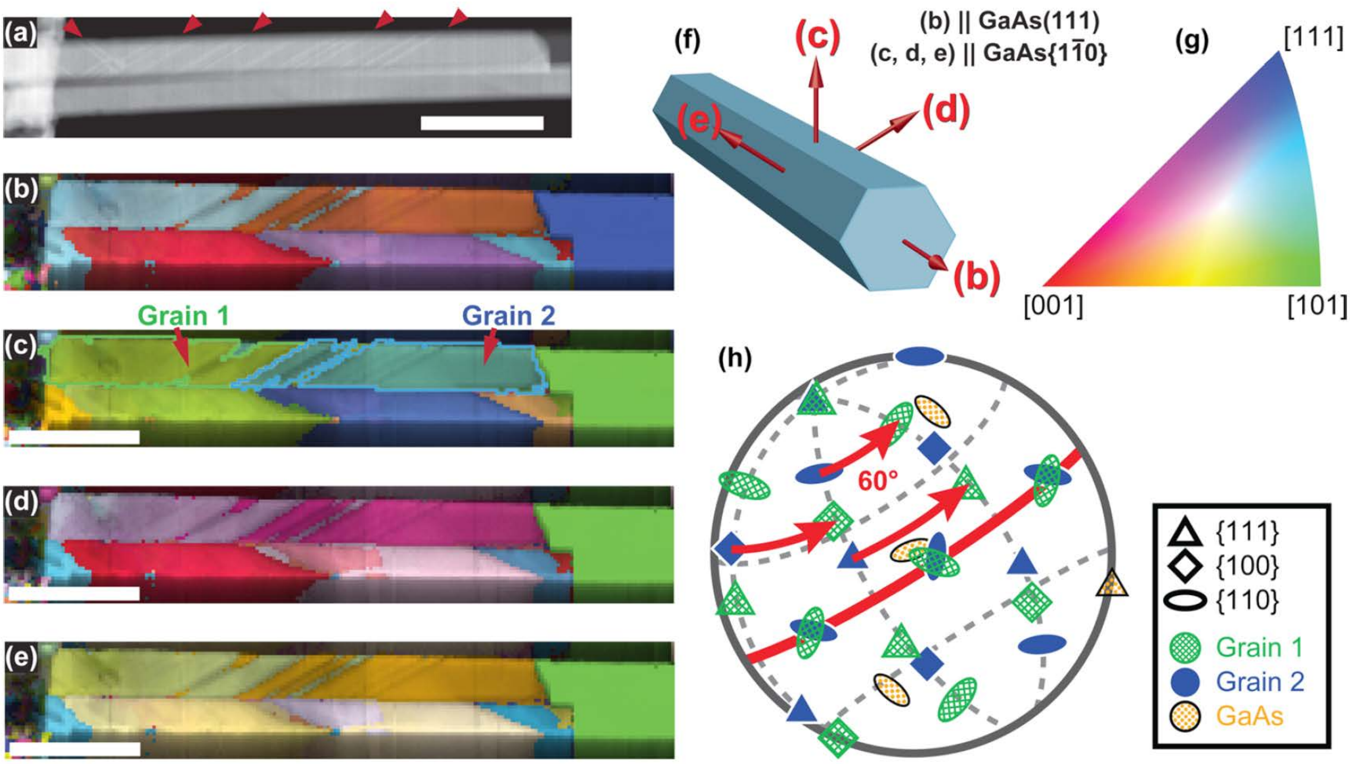


Figure 2. (a) HAADF STEM close-up of the Au phase of NW-A and NW-B where twins in NW-B are directly visible. Red triangles mark the location and direction of some of the twins. (b−e) Crystal orientation maps taken from an angle similar to that in (a). Each map shows the indexed crystal orientations parallel to the directions indicated in (f). The crystal orientation is color-coded according to the color key shown in (g). (h) Pole figure of GaAs and grains 1 and 2 as indicated in (c). The scale bars in (a−e) are 200 nm.

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Vanadium Substitution in Li_2MnSiO_4/C as Positive Electrode for Li Ion Batteries

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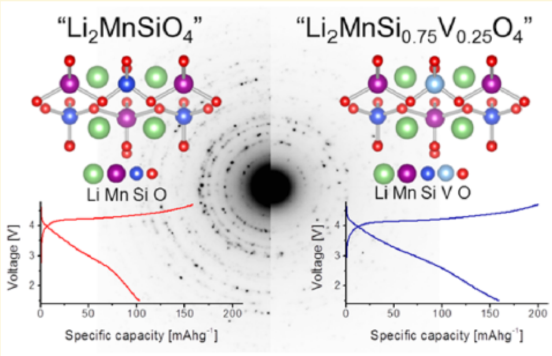
ABSTRACT: Vanadium substitution is an interesting approach to manipulate the properties of the poor electronic and ionic conducting lithium transition metal orthosilicates. Especially, if incorporated on the Si site, it could alter the highly insulating character of the SiO_4 framework. This study addresses the feasibility and limitations of V substitution in Li_2MnSiO_4. Nominal compositions of $Li_2Mn_{1-x}V_xSiO_4$ ($0 \leq x \leq 0.2$) and $Li_2MnSi_{1-x}V_xO_4$ ($0 \leq x \leq 0.3$) were synthesized by a sol–gel method, and the structural evolution was analyzed by X-ray diffraction and transmission electron microscopy (TEM) coupled with electron energy loss spectroscopy (EELS). While the solid solubility of V on tetrahedral Mn sites was shown to be limited, substantial amounts of V entered the structure when intended to substitute Si. Elemental mapping by TEM showed that V was highly inhomogeneously distributed, and high energy resolution EELS demonstrated that the majority of V was present in a tetravalent state. The nominal compositions $Li_2MnSi_{1-x}V_xO_4$ ($0 \leq x \leq 0.3$) showed superior electrochemical performance, with reduced charge transfer resistance and an increased Li ion diffusion coefficient. Furthermore, cyclic voltammetry revealed increased redox activity which can be attributed to V within the concentration series. The best performance was achieved with 25 mol % V substitution. V substitution beyond 25 mol % caused deterioration of the properties.



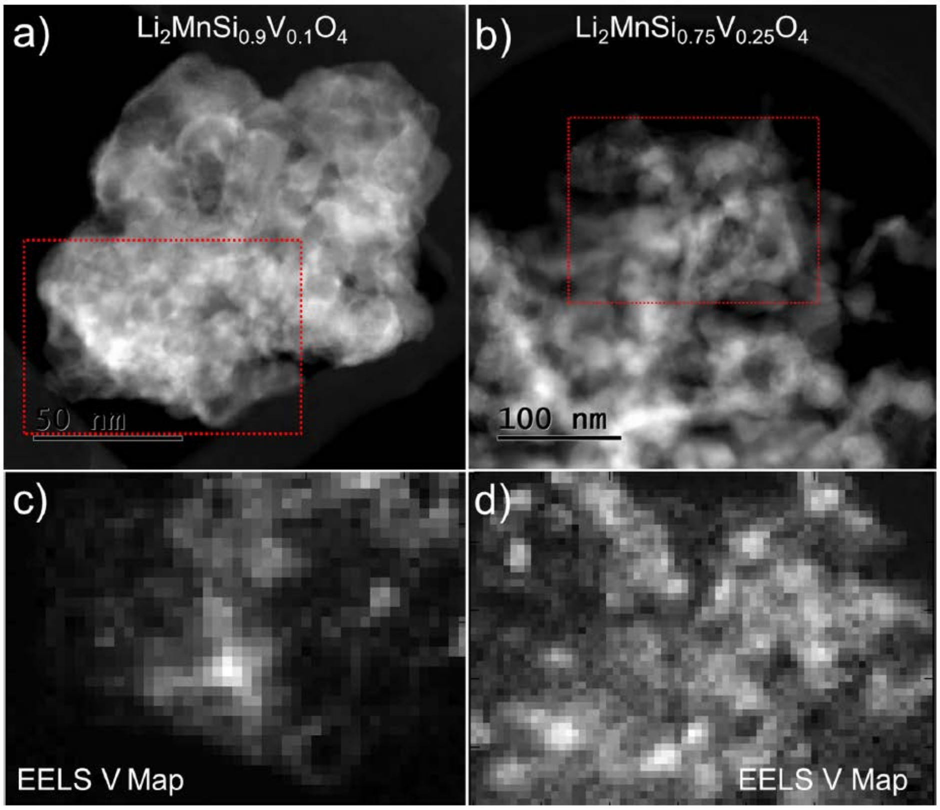


Figure 4. HAADF STEM micrographs of nominal compositions (a) $Li_2MnSi_{0.9}V_{0.1}O_4$ and (b) $Li_2MnSi_{0.75}V_{0.25}O_4$. The regions for EELS spectrum imaging are highlighted. V maps of (c) $Li_2MnSi_{0.9}V_{0.1}O_4$ and (d) $Li_2MnSi_{0.75}V_{0.25}O_4$.

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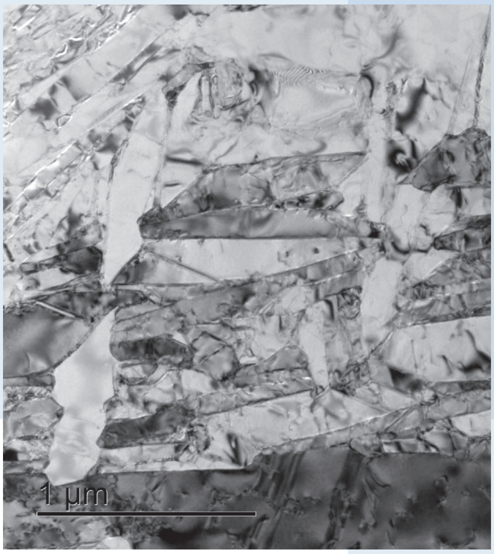

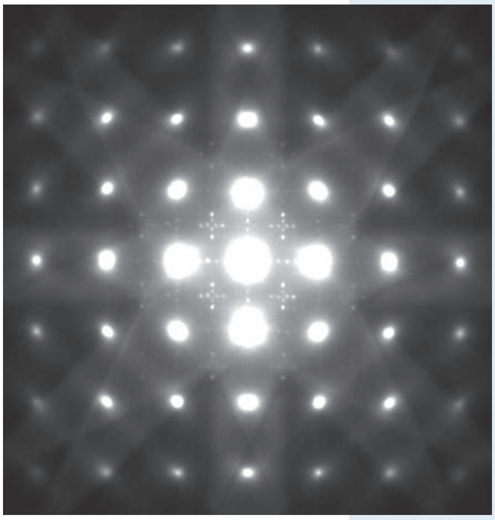

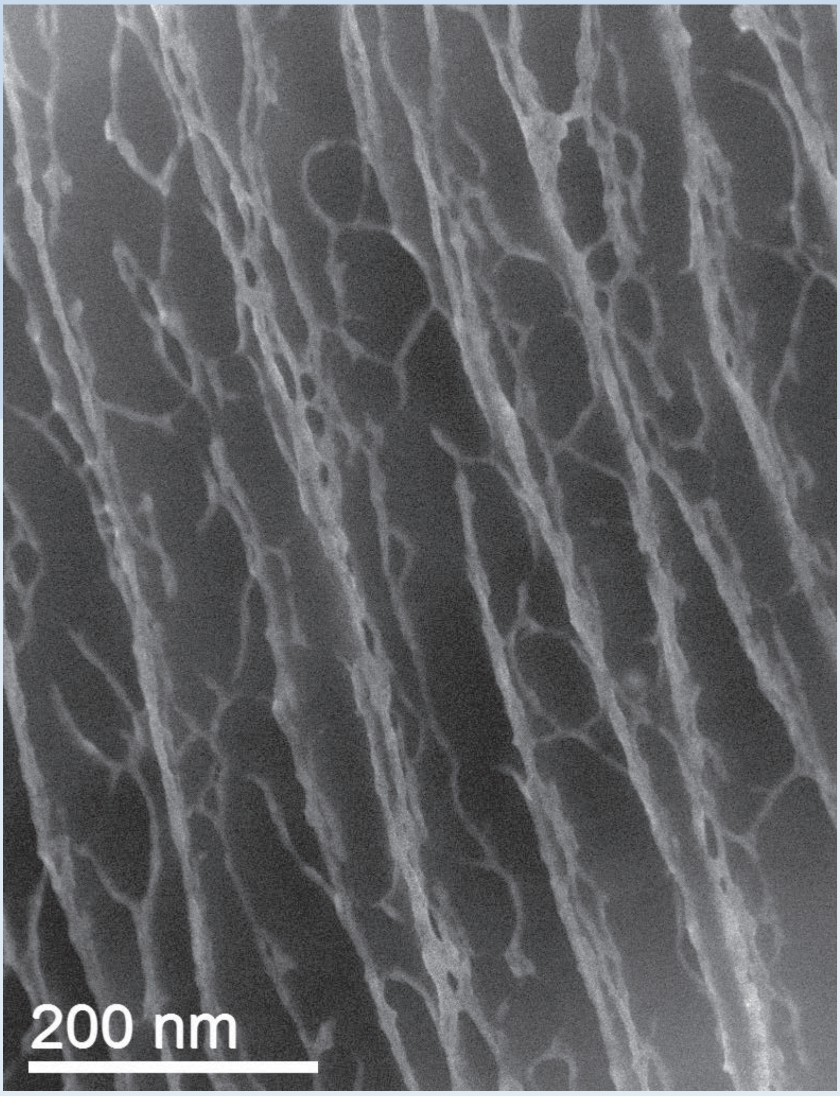

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Vidar Tonaas Fauske, "Electron Microscopy Based Characterization of Semiconductor Nanowires", 29 June 2016, Doctoral theses at NTNU, 2016:181.

Eva Anne Mørtsell, "Precipitation in multicomponent, lean Al-Mg-Si alloys – A transmission electron microscopy study", 30 September 2016, Doctoral theses at NTNU, 2016:266.

Magnus Kristofer Nord, "EELS and STEM studies of perovskite oxide heterostructures", 15 December 2016, Doctoral theses at NTNU, 2016:361.

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Tina Bergh, "Transmission Electron Microscopy Characterization of Sintered and Hot-Pressed Silicon Carbide" (Diploma, June 2016).

Andreas Garmannslund, "Refinement of the ζ-factor Method for Quantitative Energy-Dispersive X-ray Spectroscopy in Scanning Transmission Electron Microscopy" (Diploma, June 2016).

Trond R. Henninen, "Chemical Vapour Deposition and Electron Microscopy Analysis of Graphene", (Diploma, June 2016).

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Jonas K Sunde, "Scanning precession electron diffraction study of 2xxx series aluminium alloys exhibiting several coexisting strengthening phases" (Diploma, June 2016).

Philip Østli, "Density Functional Theory Studies of Precipitate Interfaces in Aluminium Alloys, with Focus on Theta'-Al_2Cu" (Diploma, June 2016).

Ingrid Marie Andersen, "TEM characterization of III-V nanowires for laser applications" (Diploma, started August 2016).

Johannes Bogen, "Data processing of Multidimensional TEM data sets" (Project, December 2016).

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Steinar Myklebost, "Quantitative image processing of electron microscopy data sets" (Project, started August 2016).

Øyvind Paulsen, "Effects of Germanium and Lithium in Al-Mg-Si alloys" (Project, December 2016)

Håkon Wiik Ånes, "Transmission Electron Microscopy Characterization of Hydride-based Smart Windows", (Project, December 2016).

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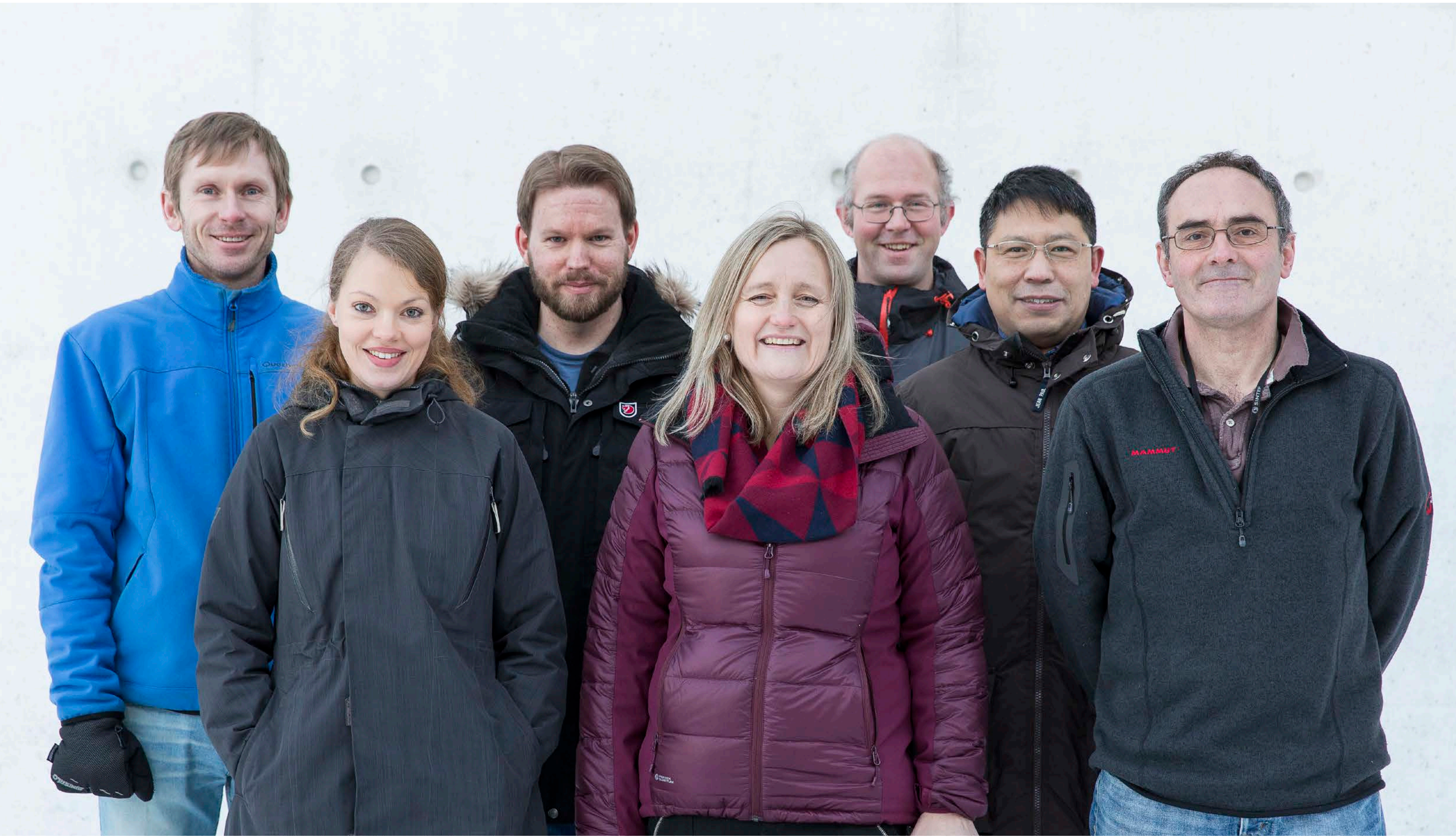

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