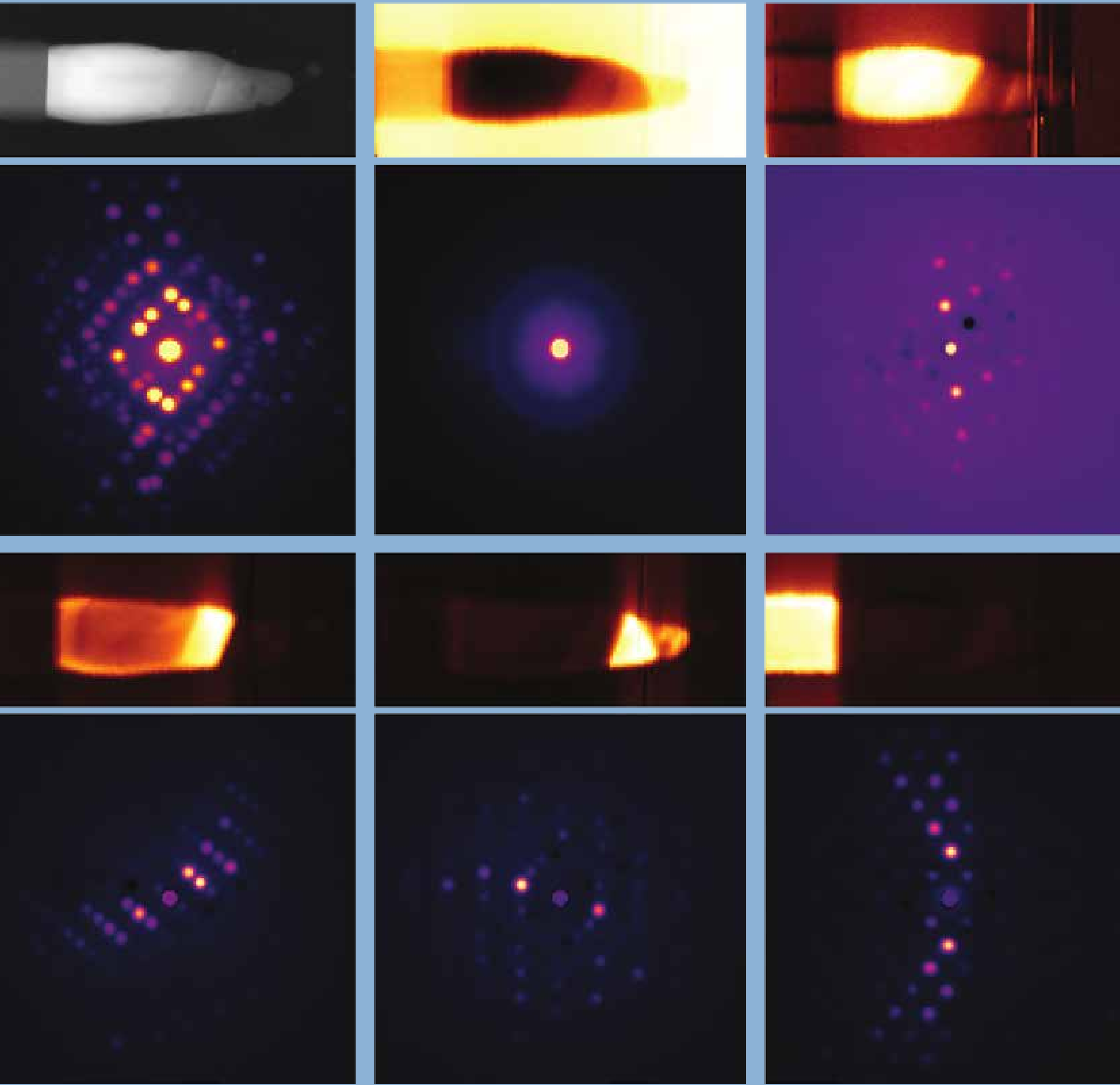


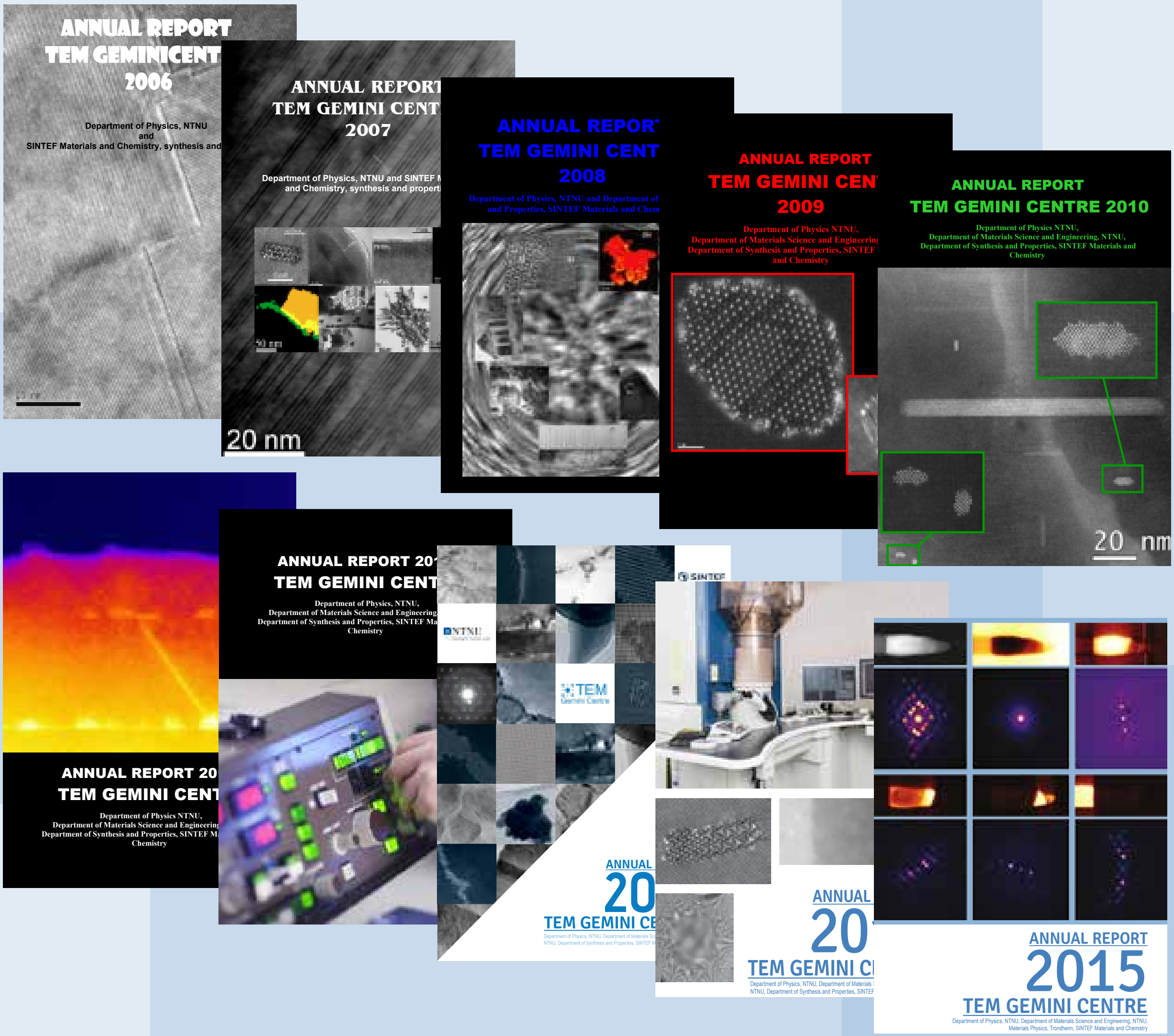
ANNUAL REPORT 2015

TEM GEMINI CENTRE

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

THE FIRST DECADE

The TEM Gemini Centre was inaugurated in March 2006. Since then, we have used a vast quantity of electrons (in the order of 10^{17}) for imaging, diffraction and spectroscopy. Many of the important results of this continuous endeavor were presented in the first 10 annual reports. Their front pages are shown below.



Graphic design: Ingebjørg Fyrileiv Guldvik, Sigurd Wenner
Photo: Ole Morten Melgård
TEM images: Scientists at the TEM Gemini Centre
Cover image: Blind source seperation of scanning precession electron diffraction data set. Material: Au-Ga alloy on a GaAs nanowire at 260 °C. Vidar Fauske (unpublished).

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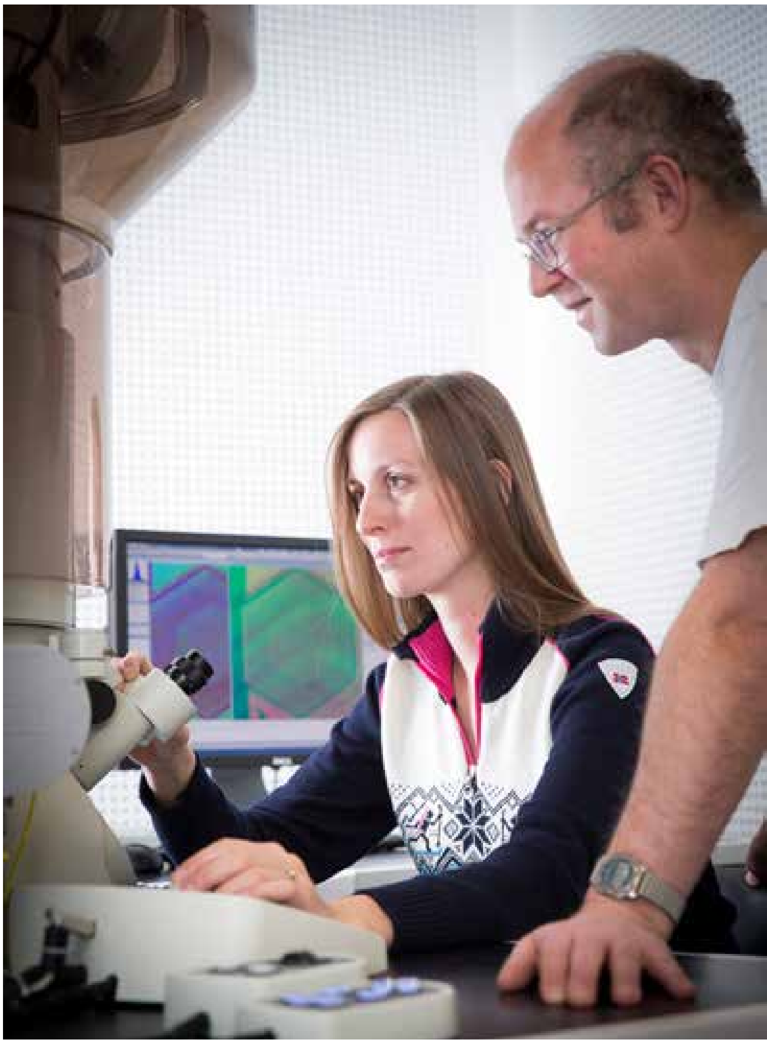
THE TEM GEMINI CENTRE

The TEM (Transmission Electron Microscopy) Gemini Centre was established in 2006, and consisted of professors, post-docs, 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 has its 10th anniversary in 2016.

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 is also being 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**’.

INTRODUCTION

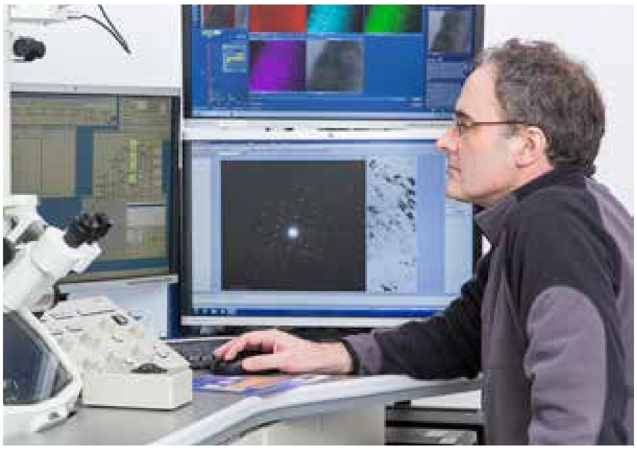
In 2015 the TEM Gemini Centre has systematically built up routines and established competence on the new NORTEM instruments, that were installed in 2013, and establishing its most effective role in the NTNU/SINTEF system. We regard the NORTEM project as evidence of the successful partnership between NTNU and SINTEF. During the recent renomination process we received a positive evaluation and feedback on how the Centre has functioned in the last years and have plans to improve further, particularly in its role as part of a national infrastructure. The NORTEM project is a partnership between NTNU, UiO, SINTEF and the Research Council of Norway. The new facilities are the culmination of many years of strategic discussion and work in order to secure full support and funding from the partner institutions and, more recently, in partnership with UiO. This process has involved active participation and support within all three institutions which are gratefully acknowledged. Looking forward, we are discussing the role of the TEM Gemini Centre and the relation to the national NORTEM project. The identity of the Trondheim node, the development of the national role and strategic objectives for the years to come, have to be clarified and addressed.

The Gemini Centre participates in a range of projects, including national public, industrial and EU funding ones. Three new long-term SFI projects where TEM is included started in 2015 – 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 new KPN projects – 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 Company) and interest from alternative research fields than we traditionally have worked with. Examples here are zero-emission building materials and biomaterials.

As documented in this report, the Centre had about 30 active users whose results have contributed to 38 journal publications in 2015. Of these, 10 have co-authors from both NTNU and SINTEF. 6 of the 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, 1 PhD and 5 Master candidates were educated with TEM as a substantial part of their theses in 2015. A positive trend is that the infrastructure gets more NTNU users from outside the Physics department. Three courses, with a total of approximately 120 students, used the facilities as part of their education at NTNU. We organized a TEM introduction course with 18 participants and a workshop on the HyperSpy multi-dimensional dataset software toolbox in September. 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 2015.

To look ahead, the Centre will host the annual conference for the Nordic Microscopy Society, SCANDEM in June 2016, along with St Olav's hospital and NTNU Nanolab. In addition, we are very happy that Jian Min Zuo has been awarded the Lars Onsager professorship and will stay in our group a few months in 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 2015. For more details we refer to our home pages of the TEM Gemini Centre (http://www.ntnu.edu/geminicentre/tem).

John Walmsley and Randi Holmestad
February 2016.



BOARD AND MANAGEMENT

During 2015 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

Key staff

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 two years. The NORTEM project is ending in 2016, and it is important to start planning the medium and long-term running and development of the facilities.

The vision of NORTEM has been "**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 discuss how to establish a running model for the new infrastructure. As a part of this process, "seed project" funding has been included in the NORTEM budget, for proof-of-principle and training activities that will lead to new projects and activities in the years to come. In Trondheim, these have contributed to 10 journal publications, so far, 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 4 new users of the facilities. Very importantly, TEM resources have been incorporated in new research applications. SINTEF Materials and Chemistry has also funded two strategic projects (SEP) with a total of 3 MNOK, that finished during 2015, of which 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. From

Present at the inauguration were SINTEF director Unni Steinsmo, Professor Randi Holmestad and prorector for research at NTNU Kari Melby. Photo: Bård Ove Molberg/Adressa.

The FEI Titan at the University of Oslo (UiO) was inspected by SINTEF director Unni Steinsmo, rector of UiO Ole Petter Ottersen, state secretary in the ministry of education and research Bjørn Haugstad and department director in NFR Asbjørn Mo. Associate professor at UiO Øystein Prytz explains the capabilities of the microscope. Photo: Werner Juvik/Teknisk Ukeblad.

2014 Ragnhild Sæterli has been working within the project alongside Bjørn Soleim in building-up and supporting maintenance, training, competence and techniques. In addition, Per Erik Vullum has been hired as an adjunct associate professor, which particularly contributes to developing interaction between NTNU and SINTEF. This comes as an addition to John Walmsley, who has had an adjunct professorship for 8 years.

In September 2015 the official opening of the NORTEM facility took place in Oslo, with the secretary of the state Bjørn Haugstad, and prominent guests from the three host institutions (NTNU, UiO, SINTEF) in addition to representatives from Norwegian industry and guests from abroad.

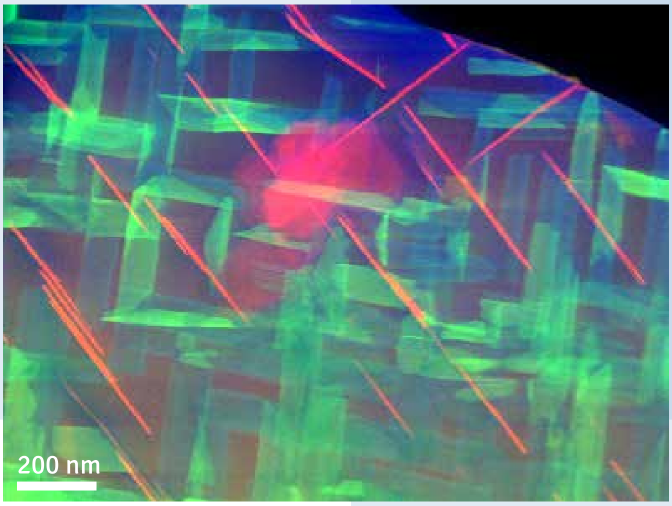
For more information on NORTEM see the webpages: www.nortem.no

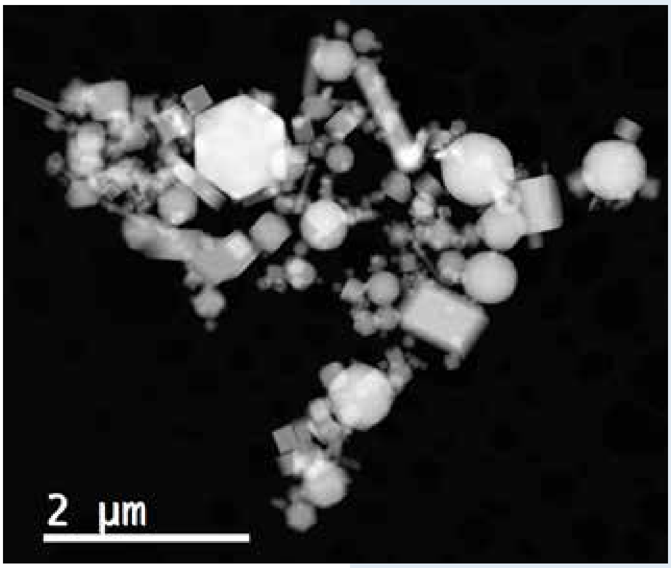
PEOPLE WORKING IN THE TEM GEMINI CENTRE DURING 2015

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)
Jan Ketil Solberg (Professor Emeritus, 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)
Yanjie Gan (Postdoc, DP, NTNU)
Sigurd Wenner (Postdoc, DP, NTNU)
Jon Holmestad (PhD student, DP, NTNU)
Vidar Tonaas Fauske (PhD student, DP, NTNU)
Magnus Nord (PhD student, DP, NTNU)
Eva Anne Mørtsell (PhD student, DP, NTNU)
Martin Ervik (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 (Master / PhD student, DP, NTNU)
Emil Christiansen (Master / PhD student, DP, NTNU)
Joakim Larsen (Master student, DP, NTNU)
Tina Bergh (Project student, DP, NTNU)
Andreas Garmannslund (Project student, DP, NTNU)
Theodor Secanell Holstad (Project student, DP, NTNU)
Jonas Sunde (Project student, DP, NTNU)
Philip Østli (Master student, DP, NTNU)

INSTRUMENTATION





TEM INSTRUMENTS

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). These instruments are described on the next pages. In addition there are two older TEMs that are still in use.

Microscopes in the TEM Gemini Centre today.

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*	*Aug. 2013*
JEOL ARM-200F	*DP, Chemistry building I*	*Cold FEG,image and probe corrected, Quantum GIF, Centurio EDS, CCD cameras*	*Oct. 2013*
JEOL 2100F	*DP, Chemistry building I*	*FEG, 200 kV, EDS, US camera, ASTAR, gas reaction and tomography holders*	*Dec. 2013*

THE NORTEM INSTRUMENTS IN TRONDHEIM

The following gives a short technical description of the three new microscopes that have been installed in the completely refurbished basement of K1.

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)*

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 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*

SPECIMEN HOLDERS

Each TEM has its own set of single and double tilt (±30 degrees) holders. A broad range of holders is available for use on all three microscopes. This includes backup double tilt holders, a cold stage holder, a conventional heating holder, an environmental cell/transfer 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 and is extremely stable at high temperatures and when changing temperature (see e.g. the cover image). Investment in additional holders for dedicated advanced studies is continuously evaluated.

OLDER TEM INSTRUMENTS

The CM30 is now 27 years old, and is still central to some research projects, mainly related to aluminium alloys. This microscope is used for conventional TEM, especially for diffraction and bright field/dark field studies. The microscope has an electron loss spectrometer for thickness measurements which is essential in quantifying precipitation density in alloys. We expect to continue to use the CM30 for some more years.

The JEOL 2010 at DMSE is 23 years old and provides similar analysis to the CM30. It has its main application areas in metallurgy and nanotechnology and is mainly used by undergraduate and post-graduate research and teaching.

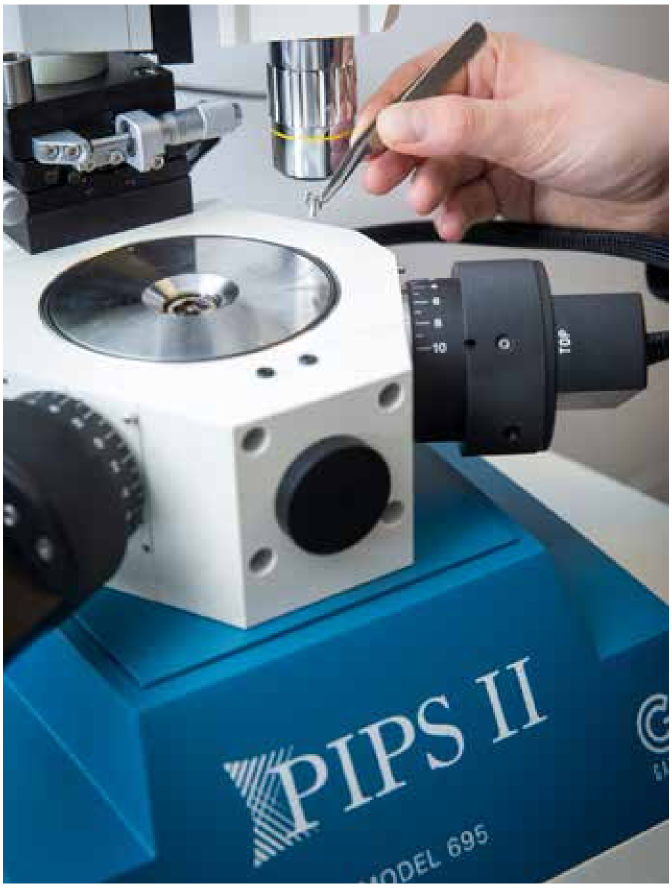
Maintaining the performance of these two older machines is increasingly demanding and cannot be guaranteed. This illustrates the importance of experienced local technical support, continuous evaluation and re-investment in large-scale facilities.

The Philips CM30.

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.

In 2015 a PIPSII was acquired, the third PIPS in the Centre, to make more accurately and reproducible specimens. A routine has been developed to polish FIB made lamellas to obtain the highest specimen quality and best possible TEM results. 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. 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. The planned acquisition of a second FIB in 2016 is very welcome.



The Gatan Precision ion polishing system (PIPS II), acquired in 2015.

SUPPORTING FACILITIES

Near the TEMs there is a well-equipped supporting room. With the new microscope technologies the cleanness requirements of the specimen and the holders increase. In 2015 investments were made in an ozone cleaner and a dedicated pumping station to store holders and delicate specimens. The room has general equipment as a plasma cleaner, a stereomicroscope, user specimen storage and 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.

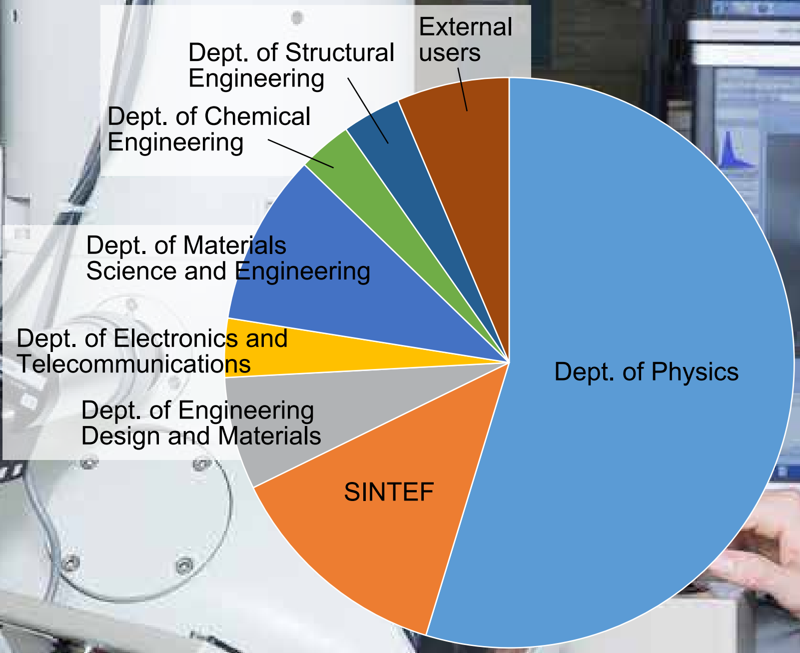
The specimen preparation room and the computer room are located in Realfagsbygget in B4, near where most Gemini Centre members have offices. All acquisition software is accessible via offline licenses in the computer room. For completeness, the Centre has a darkroom for use of negatives (mainly used for the CM30) and access to an image plate reader since December 2015.

Ragnhild Sæterli (left) and Bjørn Gunnar Soleim work as senior engineers at the Department of physics, NTNU and have done a tremendous job the last years to set up and organise the the activities around the new TEM infrastructure.

USER STATISTICS IN 2015

The total registered used time for the four instruments in 2015 was 3480 hours, including 360 non-paid hours used for testing, competence development, demonstrations and guided tours. Of the 3120 paid hours, the use by NTNU corresponds to 62,2% SINTEF 36,3 % and external 1,4%. NTNUs use is divided on six departments, where the main use is from Department of Physics (80% of NTNUs paid hours).

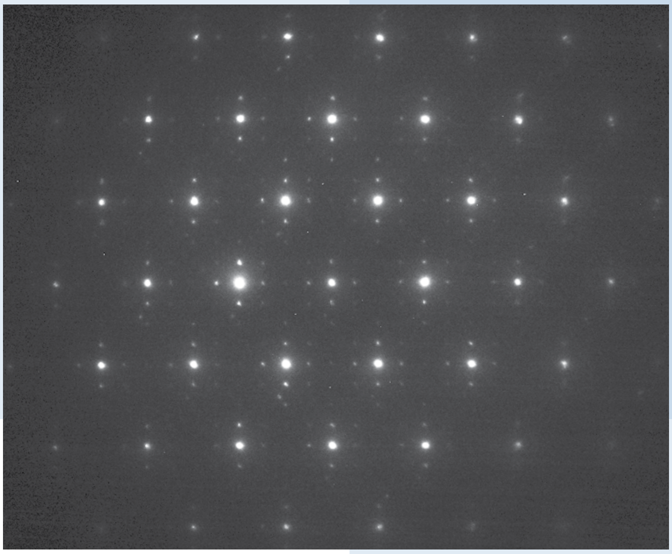
The infrastructure had 30 users in 2015, where 4 were from SINTEF. 14 were PhD students and 6 Master students. The use from outside the Physics department has experienced an increase. 7 PhD students from other departments used the infrastructure in 2015, as seen from the figure below.

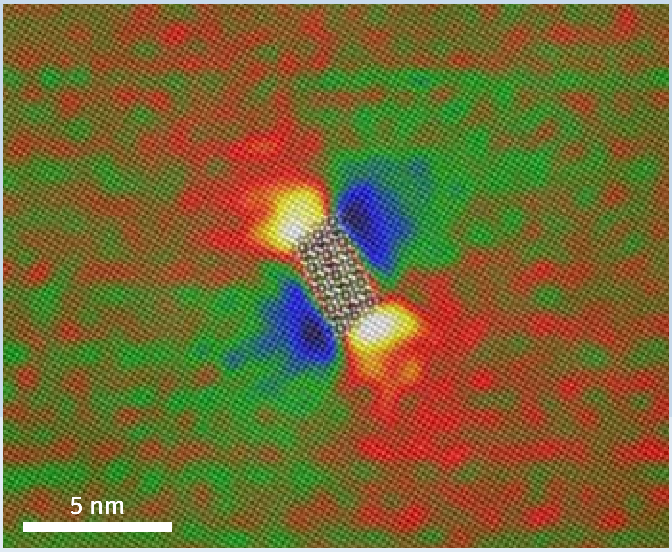


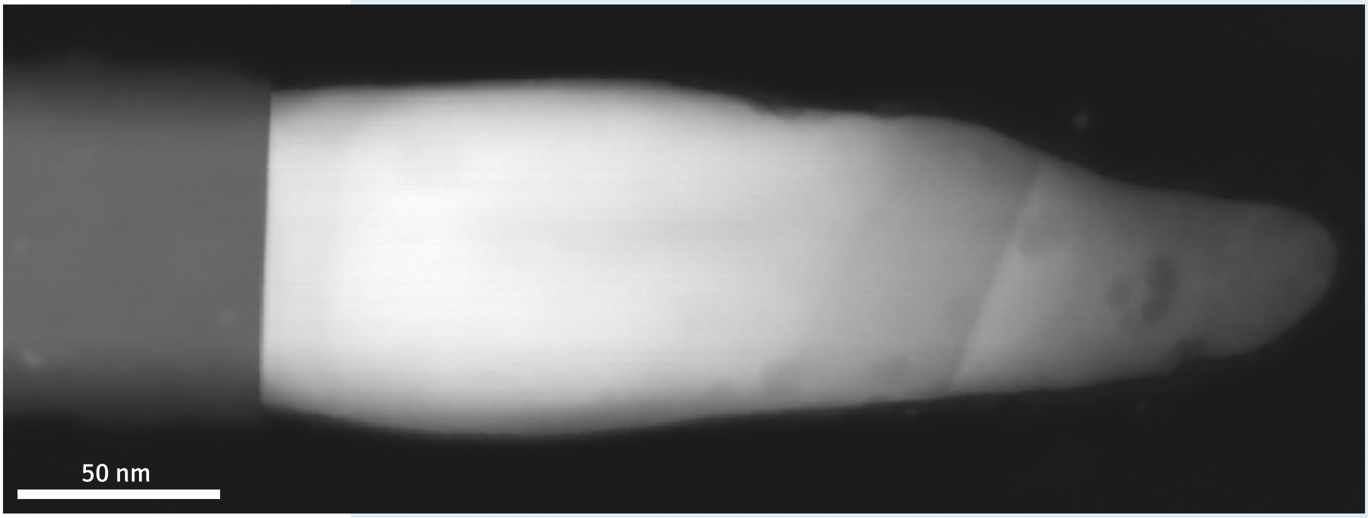
Microscope use in hours	ARM-200F	2100F	2100	CM30
SINTEF	*639*	*350*	*40*	*105*
NTNU – Physics	*579*	*322*	*413*	*77*
NTNU – IMT	*0*	*91*	*83*	*0*
NTNU – Other departments	*0*	*14*	*199*	*0*
NTNU – Visitors from abroad	*10*	*50*	*28*	*0*
NTNU – Teaching lab	*0*	*10*	*69*	*0*
External	*0*	*7*	*38*	*0*
NTNU – Set-up/testing/training/demonstations	*158*	*137*	*65*	*1*
Total use	*1385*	*980*	*934*	*182*

ACTIVITIES IN 2015

RESEARCH AND EVENTS







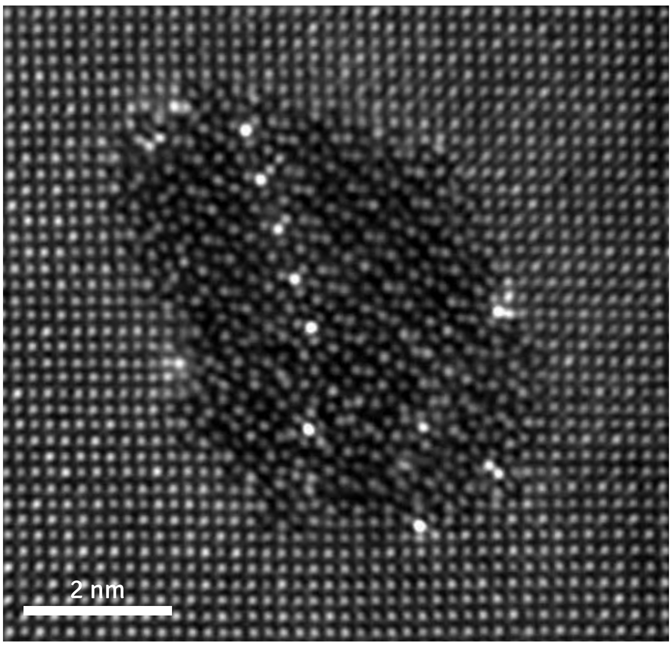
FOCUS AREAS

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 2015. 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 two 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.

In the industrially initiated project RolEx (Smart 6xxx Alloy Development for Rolling and Extrusion) which is a collaboration between NTNU, SINTEF and Hydro Aluminium, Eva Anne Mørtsell is doing her PhD. As the name indicates, this project focuses 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 is the FRINATEK Research Council project called 'Fundamental investigations of precipitation in the solid state with focus on Al-based alloys'. Martin Ervik worked for two years on this project. Sigurd Wenner is doing a 3 year postdoc, mostly working on HAADF STEM and precipitate-matrix strain measurements. 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.



In addition, 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. Emil Christiansen started his PhD on this project in August 2015. In the Manufacturing SFI project, headed by Sverre Guldbrandsen-Dahl from SINTEF RSM, joining of aluminium with other materials in multimaterial products will be a central topic, and one PhD student will be hired in 2016 to study the microstructure of these materials.

Two new KPN projects were awarded and started in 2015 towards aluminium research. One is the project 'Funda-

mentals 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 using advanced laboratory infrastructure, such as TEM and Focussed Ion Beam (FIB). 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. One PhD student will be hired for TEM studies and will start in 2016. 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 is hired as a PhD student on this project and will start in August 2016.

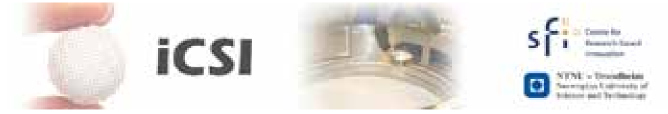
Jon Holmestad finished his PhD within aluminium research in 2015. He studied grain boundary segregation and connection to precipitates and corrosion properties within the project "Nucleation control for improved properties". These topics will be followed up in the FICAL project.

The collaboration within aluminium and TEM between the TEM Gemin Centre and Japanese research groups will continue and be expanded in the years to come. In 2015 we were awarded a new 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" (NJALC). 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 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 will be signed at the Norwegian Embassy in Tokyo in March, with participation from the Norwegian Ambassador in Japan.

ENERGY MATERIALS / SOLAR CELLS

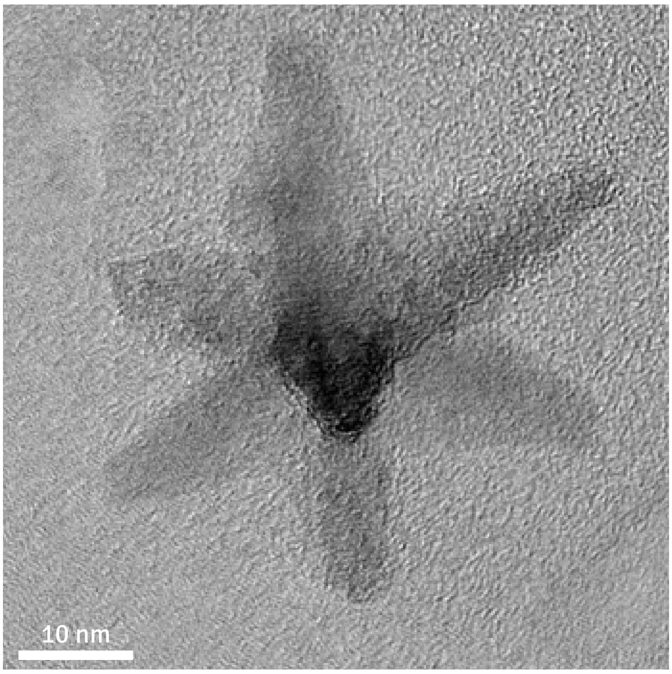
TEM has proven to be a crucial characterization tool to understand and improve the efficiency of both conventional and novel types of solar cells. The TEM Gemini Centre activities within solar cells include both studies of conventional silicon solar cell materials and new materials. Maryam Vatanparast is a PhD student working on TEM characterization of InAs quantum dots in GaAs, here we are working on measuring band gaps with electron energy loss spectroscopy. In 2015 we also had one MSc student (Joakim Larsen) working on thin film based solar cells in ZnS. The activities within intermediate band solar cell materials are in collaboration with Associate professor Turid W. Reenaas at DP. In February 2015 Julie Stene Nilsen started her PhD on 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 will be central. In November Marina Jorge started a PhD 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.

During the last four years, SINTEF has cooperated with a small Trondheim-based company, Integrated Solar, in an IPN project called "Hybrid Dual Junction Silicon Based Solar Cell". The aim of this project was to develop a prototype high efficiency solar cell based on epitaxial growth of a III-V cell on top of a Si cell. 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. This project is now followed up by another IPN project starting in the beginning of 2016, where advanced TEM will play a major role.



During the last 3 years, SINTEF has cooperated with ELKEM and IFE in an IPN project, "SiNODE", where the aim has been 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. "SiNODE" is now followed up by another IPN project owned by ELKEM, and where TEM has been dedicated an even more important role than in the ended "SiNODE".

SINTEF is also partner in two recently started 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.



CATALYSIS / MEMBRANE MATERIALS

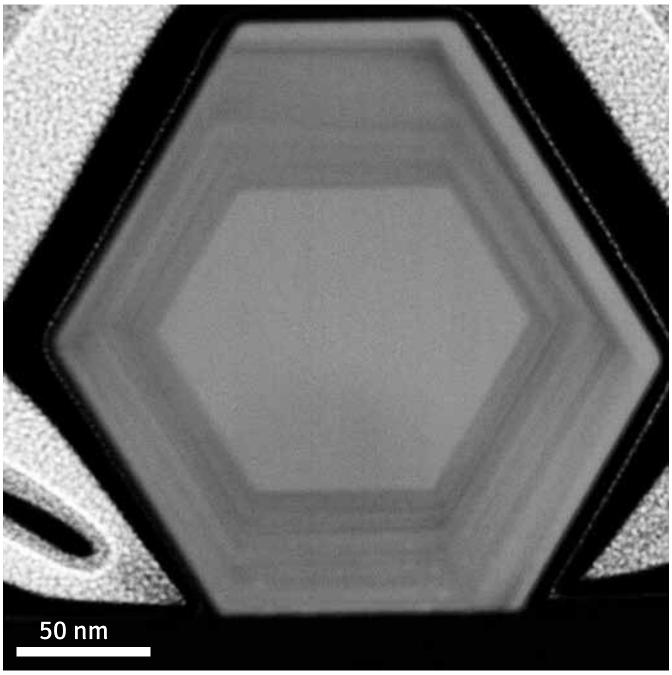
Catalysis is an important area of application for TEM and the Centre has strong interaction with the national catalysis environment, including NTNU Chemical Engineering department. The inGAP SFI, Innovative natural Gas Processes and Products finished in 2015, but the new SFI, Industrial Catalysis Science and Innovation for a Competitive and Sustainable process Industry (iCSI), headed by Hilde Johnsen Venvik, will provide a platform, including industrial contact, for applying TEM in catalysis.

We have continued our cooperation with Profs. De Chen, Hilde Venvik and Magnus Rønning at the Department of Chemical Engineering at NTNU. A Gassmaks programme project to develop catalysts and materials for a compact steam reformer is ongoing and we will address both catalyst and materials issues, such as degradation of steels by metal dusting corrosion during exposure to synthesis gas. During 2015 we have also contributed to studies of carbon supported alloy systems, supercapacitors and doped carbon nanofibers. There has been an increased cooperation with the SINTEF Materials and Chemistry Oil and Gas Process Technology Department in internal, EU funded and industrial projects. These include the EU FP7 project FASTCARD which aims at rational development of catalyst systems for biomass conversion.

Yanjie Gan left the group in 2015 after working for two years as a postdoc contributing TEM analysis to a researcher project in the CLIMIT programme, led by Thijs Peters at SINTEF, on Pd-based hydrogen storage membranes. Other membrane activity include the BIGCCS FEM centre on CO2 capture, transport and storage.

NANOTECHNOLOGY

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 the secured funding for Norfab-II is important for the TEM Gemini Centre activities within nanotechnology. PhD student Vidar Fauske, and since August 2015 Aleksander Mosberg, are hired by NTNU NanoLab and based in the TEM Gemini Centre. In his PhD Fauske combines advanced TEM techniques such as MEMS heating and precession diffraction with the complementary advanced techniques at NTNU NanoLab. Mosberg's PhD is focused on using FIB for large scale nanostructuring and TEM will be 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 2015. The Network has also been funding PhD student Magnus Nord, who is working in the field of oxide electronics. Magnus is 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 is a project in collaboration with Prof. Thomas Tybell in the Electronics department. In 2015 we also had a Master student, Emil Christiansen, who did his Master within $LaFeO_3$ thin films, studying domains in the films. We also work close together with Profs. Helge Weman and Bjørn-Ove Fimland in the Electronics department. 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 TEM work dedicated to Nanotechnology will be growing in the coming years.

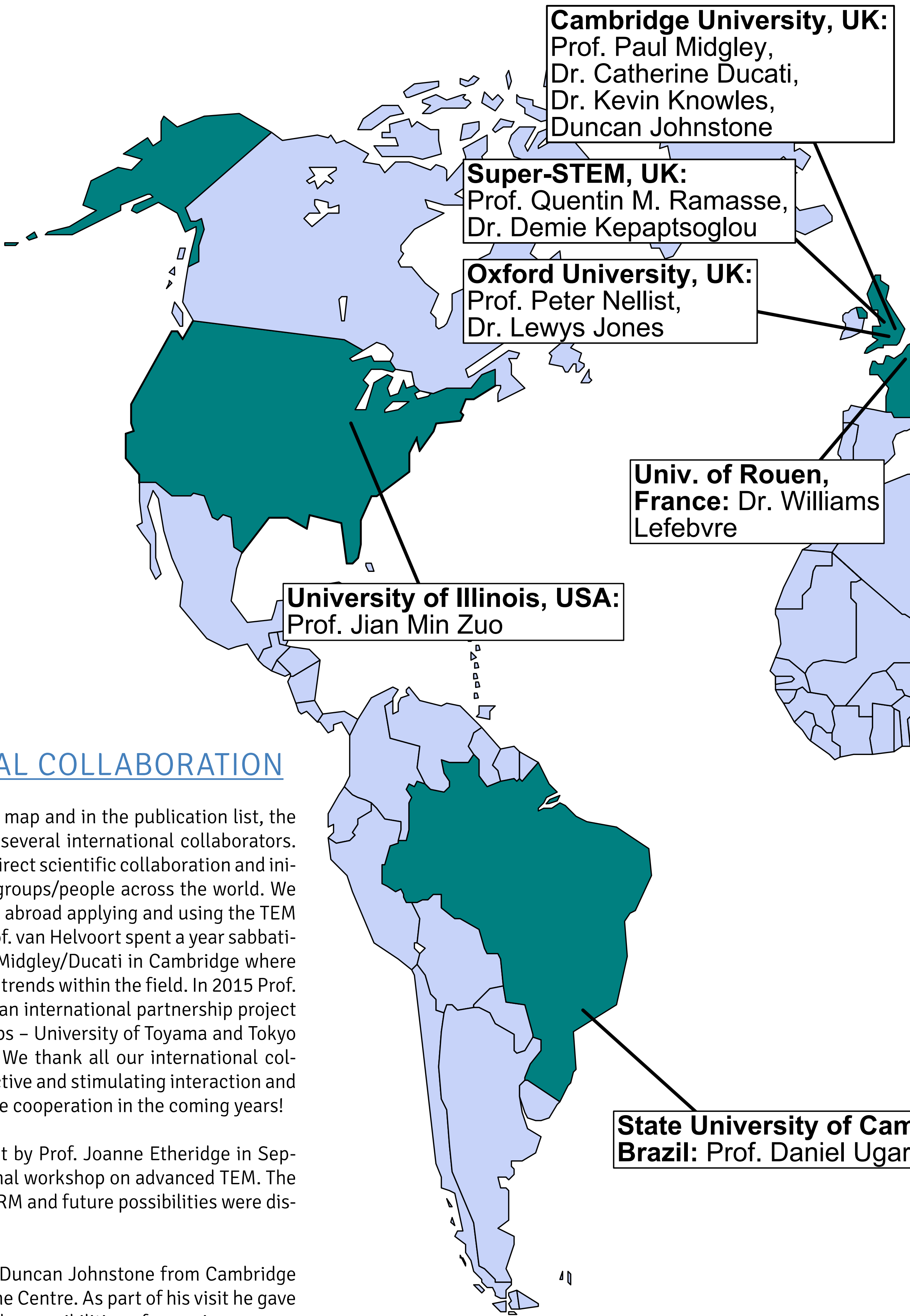


HYPERSPY / DATA PROCESSING

In 2015, beside weekly group meetings often with presentations, the Centre hosted several workshops. In September we had a day long workshop on HyperSpy, a Python-based open access platform on TEM data handling tools. The course was given by our own PhD students Vidar Fauske and Magnus Nord, who are in the development group for the package. The workshop was a tutorial with real TEM data collected within the Centre and there were 15 participants. The Centre had also one MSc project on developing a routine within HyperSpy related to quantitative energy dispersive spectroscopy and will support Fauske in 2016 to finalize a GUI for the package. We consider repeating the workshop in 2016.

CONFERENCES

As can be seen from the list of conference proceedings at the end of this report, the TEM Gemini group members are regularly participating in national and international conferences on various topics. They are often invited to international scientific meetings related to aluminium research. In July 2015 Sigmund Andersen gave an invited talk in South Africa at the 7th Light Metals Technology Conference, Jesper Friis gave an invited talk at the conference of Solid-Solid Phase Transformations in Inorganic Materials in Canada in July, and Randi Holmestad was invited to the American Microscopy & Microanalysis 2015 Meeting in August, in addition to the TEM inauguration workshop in Chongqing, China in November. Furthermore, Eva Mørtsell won the best poster Award in the EMAG conference in Manchester, UK in June.

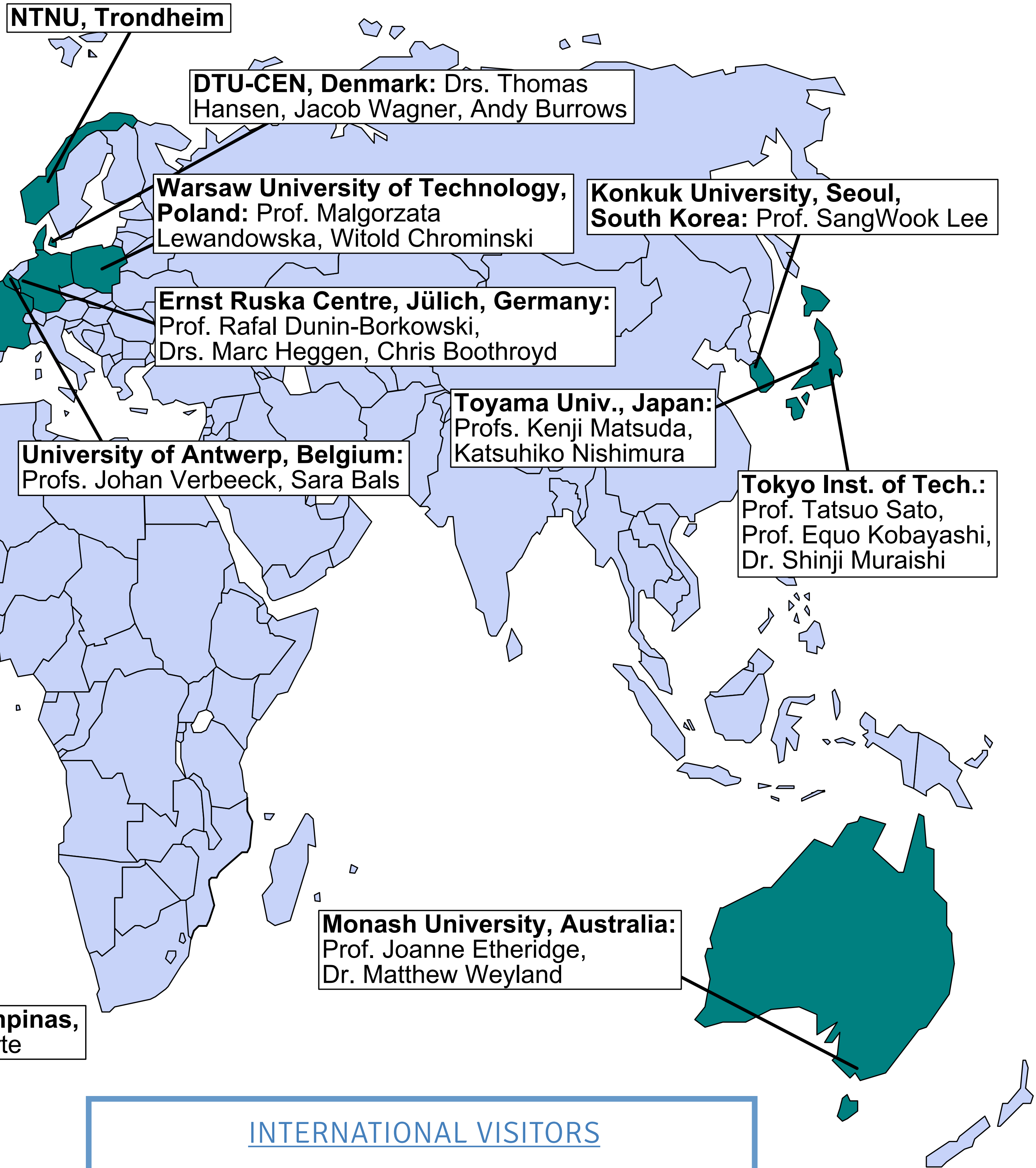


INTERNATIONAL COLLABORATION

As can be seen from the map and in the publication list, the TEM Gemini Centre has several international collaborators. The map illustrates the direct scientific collaboration and initiated contact with the groups/people across the world. We had several visitors from abroad applying and using the TEM facility in Trondheim. Prof. van Helvoort spent a year sabbatical in the group of Prof Midgley/Ducati in Cambridge where he focused on upcoming trends within the field. In 2015 Prof. Holmestad was granted an international partnership project with two Japanese groups – University of Toyama and Tokyo Institute of Technology. We thank all our international collaborators for the productive and stimulating interaction and hope we can continue the cooperation in the coming years!

In connection to the visit by Prof. Joanne Etheridge in September we had an informal workshop on advanced TEM. The latest results from the ARM and future possibilities were discussed.

In November we hosted Duncan Johnstone from Cambridge University one week in the Centre. As part of his visit he gave an inspiring lecture on the possibilities of scanning precession electron diffraction (SPED) and he trained 5 users in the technique on the 2100F. Since then we have several new SPED users and the group in general has advanced in the processing of SPED data. Especially for the activities within aluminium alloys and thin film oxides, the technique has potential to grow further.



INTERNATIONAL VISITORS

- Witold Chrominski, Poland, 7th of May -9th of June.
- Paulo Longo, Gatan Inc., USA, 18-22 May
- Protima Rauwel, University of Tartu, Estonia, 25-26 May
- Kenji Matsuda, Japan, 19-20 May
- Jo Etheridge, Monash, Austrialia, 16-17 September
- Duncan Johnstone, Cambridge University, UK, 14-21 November

ACTIVE PROJECTS IN 2015

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.

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, Aker Solutions, Audi, Benteler, BMW, DNV GL, Gassco, Honda, Hydro, Sapa, SSAB, Statoil, Toyota			
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			
IPN/BIA	***Smart 6xxx Alloy Development for Rolling and Extrusion (RolEx)***	*2 PhDs, SINTEF*	*2012–2016*
Partners: Hydro, NTNU, SINTEF			
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, Gränges			
KPN/BIA	***Fundamentals of Intergranular Corrosion in Aluminum Alloys (FICAL)***	*1 PhD, SINTEF*	*2015–2020*
Partners: NTNU, SINTEF, Hydro, Benteler, Steertec, SAPA, Gränges			
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/ENERGIX	***High Efficiency Quantum Dot Intermediate Band Solar Cells (HighQ-IB)***	*1 PhD, SINTEF*	*2012–2017*
Partners: NTNU, SINTEF			
FP/FRINATEK	***Oxide Intermediate Band Photovoltaics (Ox-IB)***	*1 PhD, SINTEF*	*2015–2020*
Partners: NTNU			
IFP/Nano2021	***GRANASOL – Low Cost, Ultra-High Efficiency Graphene Nanowire Solar Cells***	*1 PhD*	*2014–2018*
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			
HyF-Lex	***Fatigue and hydrogen degradation of steels***	*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			

DISSERTATION – JON HOLMESTAD

Jon Holmestad defended his PhD thesis on the 30th of January. The thesis title is “(Scanning) Transmission Electron Microscopy Studies of Grain Boundary Segregation relevant to Intergranular Corrosion in Al-Mg-Si-Cu Alloys”. Jon undertook his PhD research on secondment from his position at the Hydro Research Centre at Sunndalsøra. The main part of the thesis concerned relating intergranular corrosion susceptibility to grain boundary microstructure and chemistry observations made at the nanoscale in the TEM. This provided some new insights, and raised new questions, into the segregation behaviour of solute elements, such as copper, magnesium and silicon, in industrial aluminium alloys and the consequences with respect to corrosion. The prescribed trial lecture title was “3D microstructure imaging across length-scales – possibilities and application areas”. Jon is now working as a R&D Scientist at Swix Sport AS in Lillehammer.

From the left, Associate Prof Ida Westermann (administrator), NTNU,PhD candidate Jon Holmestad, NTNU, Prof Vidar Hansen (opponent) University of Stavanger, Prof Roy Faulkner, Loughborough University, UK, Prof II John Walmsley (main supervisor), NTNU/SINTEF

MARTIN ERVIK (1987–2015)

Martin Ervik wrote his Master thesis in the TEM group at Department of Physics in 2011. He worked on an aluminium project - learned to use the TEM, and studied two aluminium alloys with respect to corrosion on grain boundaries. Two years after, in the fall of 2013, he returned to start a PhD in the same field, hired in the FRINATEK project “Fundamental investigations of precipitation in the solid state with focus on Al-based alloys”. Martin worked for two years on his PhD, attended all required courses, measured hardness and characterized alloys using TEM. As a part of the study, he did duty work for the Department of Physics, supervising younger students. Here he showed his high pedagogical skills, and was very much liked. Martin was trained on the ARM during the last weeks he lived. He progressed very fast, and obtained impressive results for a beginner. He eagerly planned to start using this microscope to study the alloys from his project.

Martin was modest and a little shy, but he always had a smile for everyone, and was not afraid to ask for advice. He was eager to help new students in the group and was always well prepared. Just before he died Martin was in Stuttgart at an ARM user meeting. He was going to present what he had learned there on our Friday lunch meeting the day after his return trip. However, in an early morning e-mail he said he had taken ill. He died in his home that weekend, as a consequence of his diabetes. Our thoughts go to Martin’s parents and brother. In the TEM group we miss Martin as a good colleague and friend.

PHILIPS 400T: SECTIONED

The first TEM within physical sciences in Trondheim, the Philips 400T microscope, was acquired in 1981. It was decided that this TEM is valuable as an educational exhibit object. With aid from the mechanical workshop at the Factulty of Natural Sciences, we sectioned a 90 degree wedge out of the column so that the inner workings of the microscope are visible from the outside. The setup is portable and will be used in teaching, presentations and during school visits and conferences. It is depicted here with the second 400T in the background, which originates from the University of Oslo.

Nordic Microscopy Society

SCANDEM 2016

JUNE 7–10

NTNU
Trondheim

Photo: Mentz Indergaard

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

SCANDEM 2016 is held in Trondheim, Norway, hosted by NTNU, the Norwegian University of Science and Technology and organized both by the Natural Science- and Medical- Faculties. Several departments, including the Kavli Institute for Systems Neuroscience, SINTEF, NTNU NanoLab and NORTEM are involved.

The program includes plenary lectures, parallel sessions in physics/material sciences and life sciences, a large microscopy exhibition with more than 20 companies, poster session, pre-conference workshops, and lab visits.

Parallel sessions on Life Science

– ***Molecular scales***, chair: Magnus Lilledahl, Catharina Davies
– ***Bio-nano***, chair: Bjørn Stokke, Pawel Sikorski
– ***Neuroscience***, chair: Menno Witter
– ***Correlative microscopy***, chair: Johannes Van der Want
– ***Cellular imaging***, chair: Trude Flo

Parallel sessions on Materials science

– ***Nanomaterials***, chair: Per Erik Vullum, Kay Gastinger
– ***Geology***, chair: Suzanne McEnroe, Nathan Church
– ***Instrumentation***, chair: John Walmsley, Ragnhild Sæterli
– ***Functional materials***, chair: Ton van Helvoort, Jostein Grepstad
– ***Structural materials***, chair: Randi Holmestad, Yanjun Li

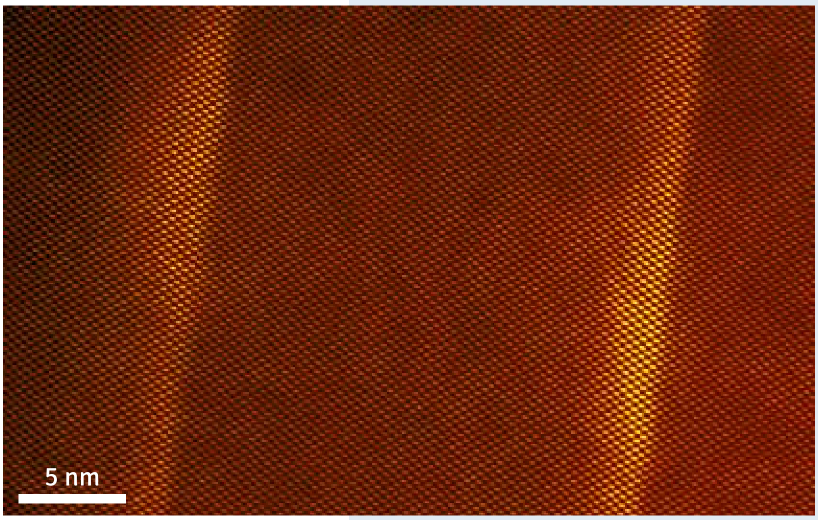
Confirmed invited speakers:

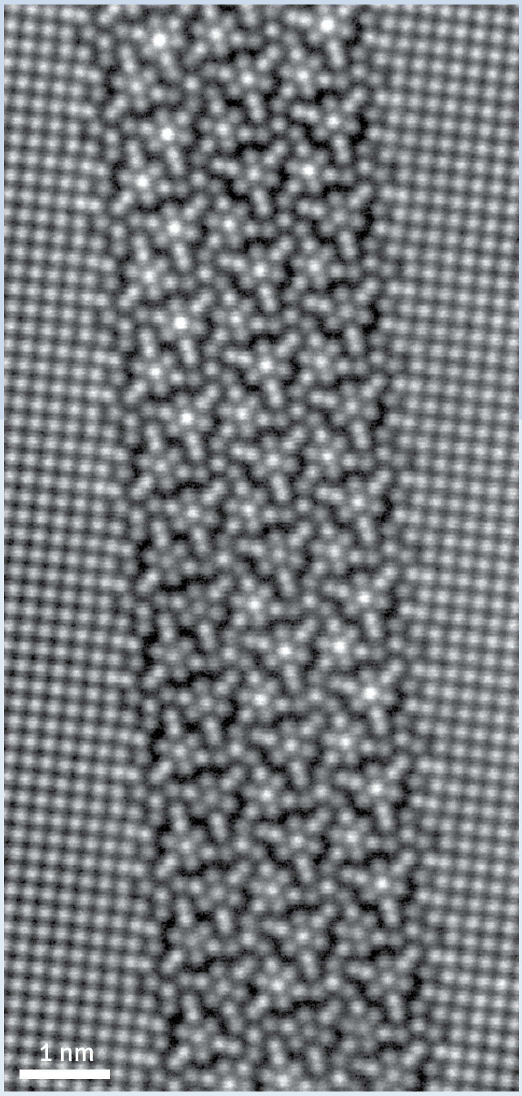
Sara Bals, Antwerp
Paul Midgley, Cambridge
Peter J. Peters, Maastricht
Andreas Brecht, Oslo
Lucy Collinson, London
Stephan Hofmann, Cambridge
Quentin Ramasse, Daresbury
Stefan Zaeffrer, Düsseldorf
Alice Bastos da Silva Fanta, Copenhagen
Simon Scheuring, Marseille
Julian Moger, Exeter
Lewys Jones, Oxford
Kenji Matsuda, Toyama

Sponsored by: Nano@NTNU
TEM Gemini Centre
The Research Council of Norway

SELECTED
SCIENTIFIC PAPERS

CONTAINING RESULTS FROM NORTEM INSTRUMENTS





L-DOPA-Coated Manganese Oxide Nanoparticles as Dual MRI Contrast Agents and Drug-Delivery Vehicles

Birgitte Hjelmeland McDonagh, Gurvinder Singh, Sjoerd Hak, Sulalit Bandyopadhyay, Ingrid Lovise Augestad, Davide Peddis, Ioanna Sandvig, Axel Sandvig, and Wilhelm Robert Glomm*

Manganese oxide nanoparticles (MONPs) are capable of time-dependent magnetic resonance imaging contrast switching as well as releasing a surface-bound drug. MONPs give T2/T2* contrast, but dissolve and release T1-active Mn^{2+} and L-3,4-dihydroxyphenylalanine. Complementary images are acquired with a single contrast agent, and applications toward Parkinson's disease are suggested.

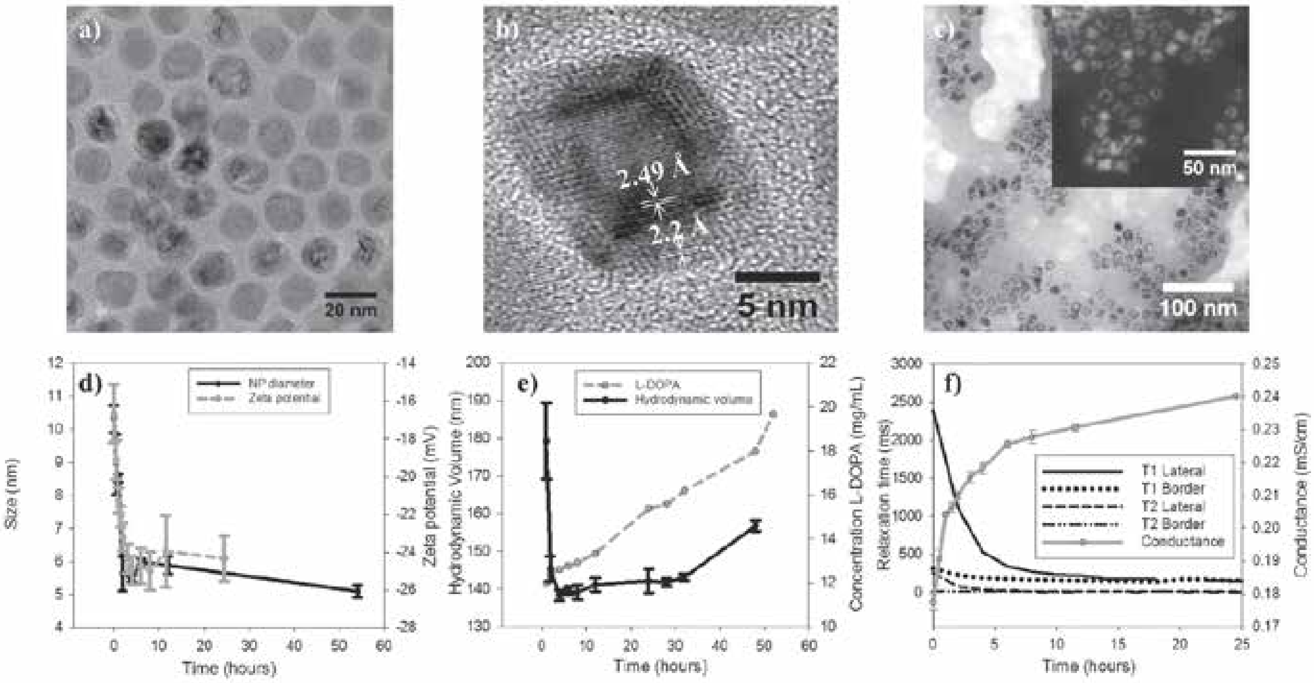


Figure 1. MONPs degrade in water and release L-DOPA. a) TEM of MONPs in organic phase. b) HRTEM image of MONPs before surface modification. c) TEM of L-DOPA-coated MONPs, 1 h in water (pH = 6.8). Inset shows dark field image of hollow MONPs after 1 h in water. d) Diameter of MONPs and ζ-potential of MONPs as a function of time. e) Hydrodynamic volume and concentration of L-DOPA as a function of time. f) T1 and T2 relaxation time in pig eyes upon injection of MONPs, as a function of time. "Lateral" refers to the opposite side of the injection site, while "Center" refers to the mid part of the pig eye. Conductance as a function of time is also shown.

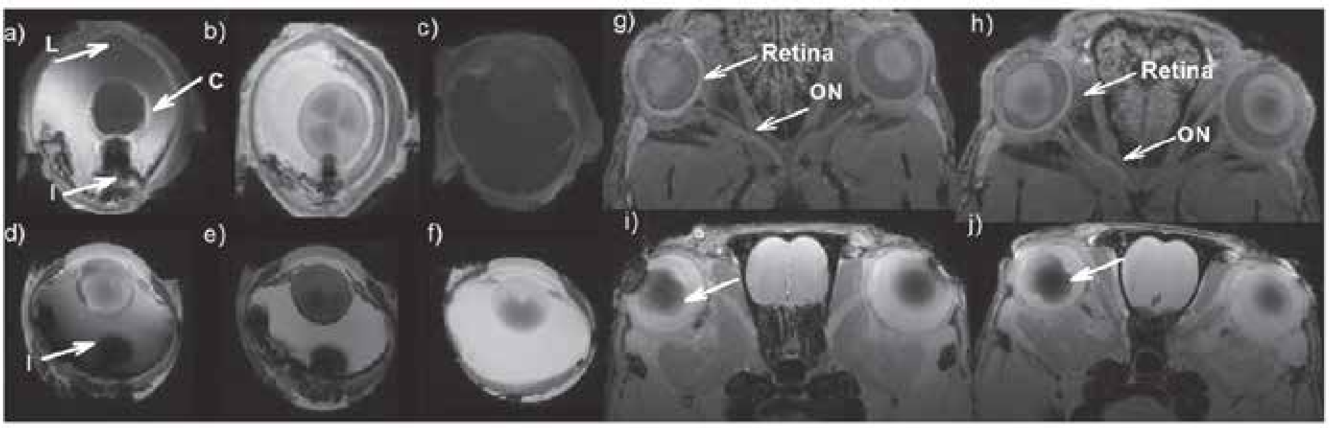


Figure 2. Ex vivo and in vivo degradation of MONPs. Images (a) and (b) show the T1w images of a pig eye injected with MONPs, acquired immediately after injection and after 34 h, respectively. "L" refers to the lateral side of the injection site "I", while "C" refers to the central part of the pig eye. Images (d) and (e) show the complementary T2*w images immediately after injection and 34 h after injection, respectively. Image (c) is the T1w and image (f) is the T2*w image of the control eye, immediately after injection and 34 h after injection, respectively. g) T1w and i) T2*w image of rat ON and retina, 1 h postinjection. h) T1w and j) T2*w image of rat ON and retina, 24 h postinjection.

Philosophical Magazine, 2015
Vol. 95, No. 7, 744–758, http://dx.doi.org/10.1080/14786435.2015.1006294

Combining HAADF STEM tomography and electron diffraction for studies of α-Al(Fe,Mn)Si dispersoids in 3xxx aluminium alloys

Astrid Marie F. Muggerud[a]* , John C. Walmsley[ab], Randi Holmestad[a] and Yanjun Li[c]

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(*Received 23 June 2014; accepted 26 December 2014*)

A new methodology to study the precipitation crystallography of dispersoids in an Al matrix is proposed. By combining high angle annular dark field tomography and electron diffraction studies, the three-dimensional morphology, orientation relationship (OR) with Al matrix and habit planes of the dispersoids can be achieved simultaneously. This approach has been applied to investigate the α-Al(Mn,Fe)Si dispersoids precipitated in an AA3xxx alloy. Most dispersoids have a plate-shaped morphology after low-temperature homogenization at 450°C. The largest proportion of the dispersoids follows the previously described OR with the Al matrix $\langle 1\,\bar{1}\,1\rangle_{Al}//\langle 1\,\bar{1}\,1\rangle_{\alpha}$, $\{0\,1\,1\}_{Al}//\{5\,\bar{2}\,\bar{7}\}_{\alpha}$. Two plate-shaped dispersoids have been studied in detail. The dispersoid following the commonly observed orientation had habit planes $(\bar{1}\,1\,1)_{Al}//(2\,\bar{5}\,0)_{\alpha}$. The dispersoid not following the commonly observed OR had habit planes $(4\,0\,\bar{2})_{Al}//(1\,3\,\bar{2})_{\alpha}$, with (OR) $[0\,1\,0]_{Al}//[4\,2\,5]_{\alpha}$, $(4\,0\,\bar{2})_{Al}//(1\,3\,\bar{2})_{\alpha}$.

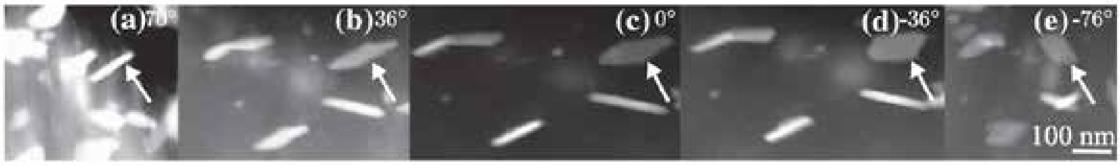

Figure 2. HAADF STEM tomography micrographs from tilt series at (a) 70°, (b) 36°, (c) 0°, (d) −36° and (e) −76° tilt angle. The arrow pints to dispersoid 1 in Figure 1(b) and (c).

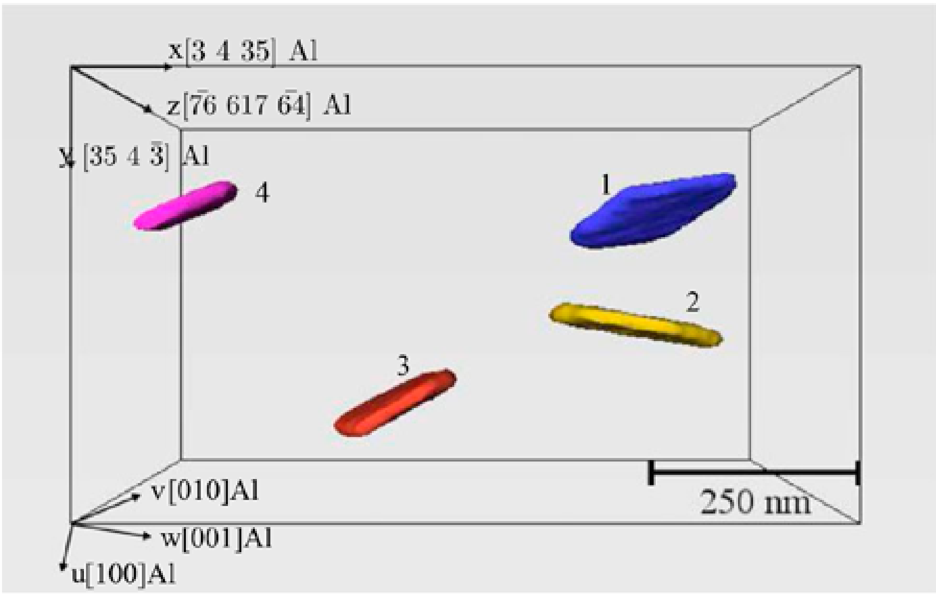

Figure 3. (colour online) 3D visualization of the reconstructed dispersoids viewed in the z-direction of the tomography series, close to the v [0 1 0] Al zone axes direction. The directions in Al are found from diffraction. The indices of the x, y and z axis in the Al coordinate system are given in the upper left corner. The dispersoids are marked 1–4 corresponding to Figure 1(c). See 'Movie I' (Online Supplemental data) for a full dynamical view of the reconstructed dispersoids.

Effects of Germanium, Copper, and Silver Substitutions on Hardness and Microstructure in Lean Al-Mg-Si Alloys

EVA ANNE MØRTSELL, CALIN D. MARIOARA, SIGMUND J. ANDERSEN, JOSTEIN RØYSET, ODDVIN REISO, and RANDI HOLMESTAD

It is shown that strength loss in a 6060 Al-Mg-Si alloy caused by reduction in solute can be compensated by adding back smaller quantities of Ag, Ge, and Cu. Nine alloys were investigated. Ge was found to be the most effective addition, strongly refining the precipitation. The hardness is discussed in terms of statistics of the precipitates near a T6 condition, as acquired by transmission electron microscopy (TEM). Precipitates in some conditions were also investigated by high-angle annular dark-field scanning TEM. The added elements have strong influence on the main hardening precipitate, β'', changing its structure and promoting disorder.

DOI: 10.1007/s11661-015-3039-5

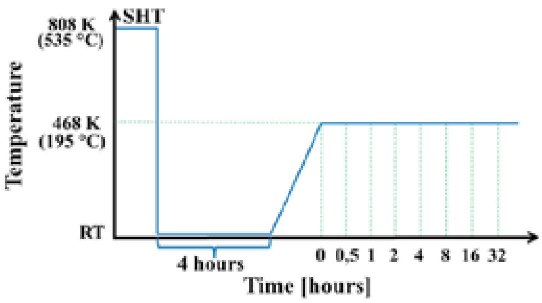

Fig. 1—Heat treatment overview. After quenching from solution temperature [626 K (353 °C)], samples were held 4 h at RT before taken to the 468 K (195 °C) aging temperature 26 K (°C)/h. Vertical dotted lines (with respective aging times) indicate water-quenching to RT.

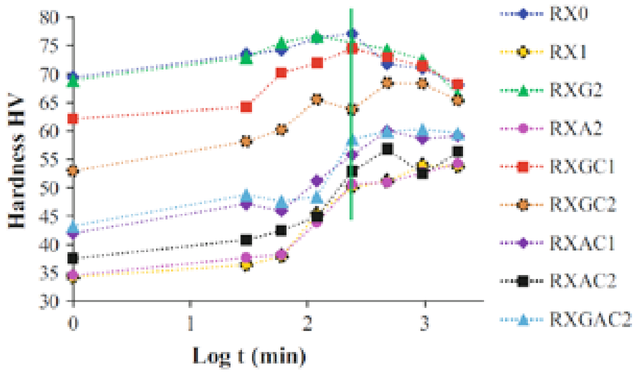

Fig. 2—Hardness (HV) as a function of time for the nine alloys, during an artificial aging (AA) at 468 K (195 °C) (see Fig. 1). The lean reference, RX1, exhibits the lowest HV, while the denser reference, RX0, has the highest HV values together with RXG2. The green vertical line indicates the conditions investigated by TEM, corresponding to 4 h of AA (Color figure online).

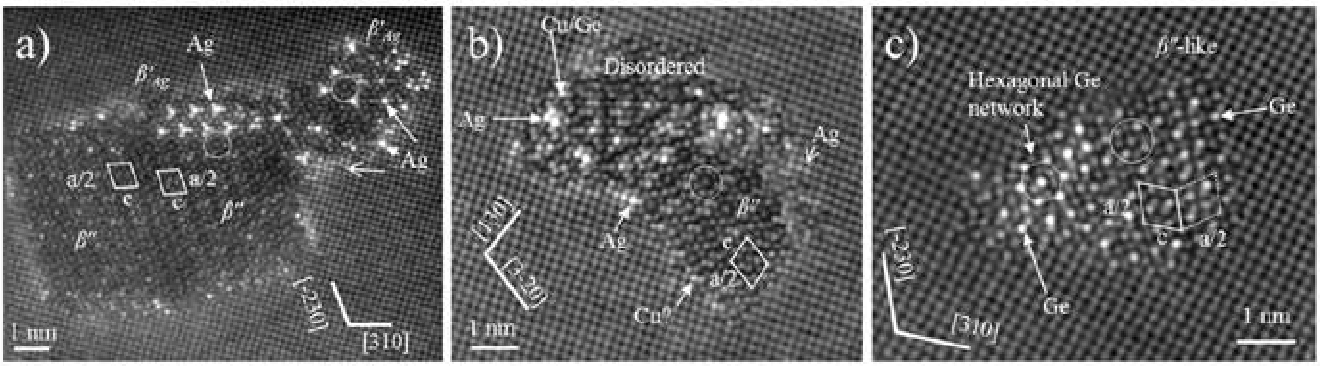

Fig. 6—HAADF images of typical precipitates in three of the investigated alloys showing that formation of β'' phase is strongly affected by the additions of Ge, Ag, and Cu. Circles indicate basic units of β''. Open/filled arrows point to single columns/areas. Half unit-cells of β'' are indicated: (*a*) Silver-containing alloy RXA2 with large Ag-free β'' particle with a central stacking fault along the *a*-axis. Ag is found at the interface as single columns, ordered in a β'_{Ag} phase, or in a low concentration in Al just outside the particle, (*b*) Mixed β''/disordered particle in alloy RXGAC2 with additions of Ge, Ag, and Cu. The added elements observed in the disordered part cannot be distinguished by the Z-contrast alone. (*c*) Alloy RXG2 with Ge addition. Ge integrates in the β'' structure, substituting in some of the Si-positions in the β'' phase unit, leading to disorder/restacking of the units (dotted rhomb). The left part shows a region where Ge arranges hexagonally.

Structural phases driven by oxygen vacancies at the $La_{0.7}Sr_{0.3}MnO_3/SrTiO_3$ hetero-interface

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An oxygen vacancy driven structural response at the epitaxial interface between $La_{0.7}Sr_{0.3}MnO_3$ films and $SrTiO_3$ substrates is reported. A combined scanning transmission electron microscopy and electron energy loss spectroscopy study reveal the presence of an elongated out-of-plane lattice parameter, coupled to oxygen vacancies and reduced manganese oxidation state at the $La_{0.7}Sr_{0.3}MnO_3$ side of the interface. Density functional theory calculations support that the measured interface structure is a disordered oxygen deficient brownmillerite structure. The effect of oxygen vacancy mobility is assessed, revealing an ordering of the vacancies with time. © *2015 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4906920]

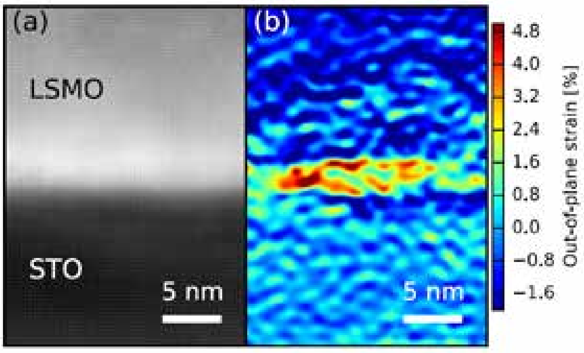

FIG. 1. (a) Cross-sectional STEM-HAADF image of the LSMO/STO heterostructure, showing a coherent interface. (b) Map of the out-of-plane strain in (a), using the STO-substrate (3.905 Å) as a reference.

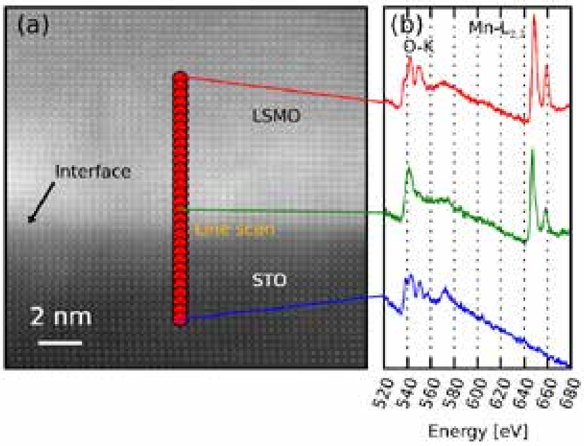

FIG. 2. (a) Cross-sectional STEM-HAADF image of the LSMO/STO heterostructure. (b) Results of an EELS line scan across the LSMO/STO interface.

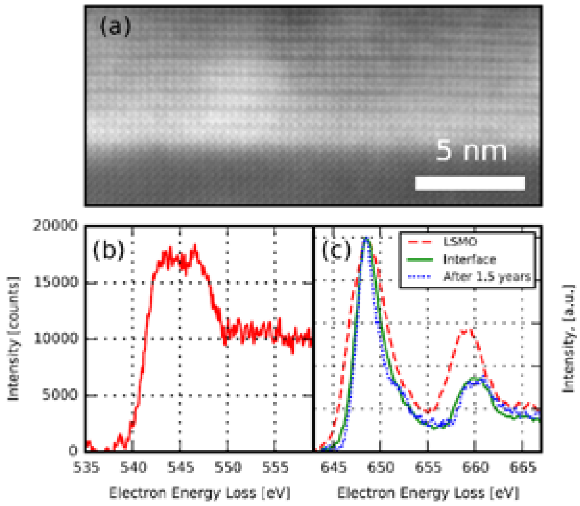

FIG. 4. (a) Cross sectional STEM-HAADF image from the same TEM lamella as Figs. 1(a) and 2(a), after approximately 1.5 years showing an ordered brownmillerite superstructure at the film side of the interface. (b) Representative STEM-EELS data from the superstructure in (a), showing an oxygen K-edge consistent with oxygen vacancies. (c) The same as (b) but for the Mn-$L_{2,3}$ edge, with the spectra from Fig. 3(a) as comparison.

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Structural modifications and electron beam damage in aluminium alloy precipitate θ'–Al_2Cu

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The θ'–Al_2Cu phase in an Al–4Zn–2Cu–1Mg–0.7Si (wt.%) alloy was investigated by means of scanning transmission electron microscopy. With our specific alloy composition, the phase is often formed with stacking faults on $\{1\,0\,1\}_{\theta'}$ and $\{0\,0\,1\}_{\theta'}$ planes. The stacking faults on $\{1\,0\,1\}_{\theta'}$ planes are often regularly spaced and create a previously unreported superstructure. Structural damage by electron irradiation is observed, even at a low acceleration voltage of 80 kV. The damage is more pronounced in the θ' precipitates with stacking faults, which agrees with theoretical calculations of knock-on scattering cross-sections. These two very different forms of disruptions of the θ' structure are linked to its spacious interstitial sites and the ease at which Cu atoms diffuse into and between them.

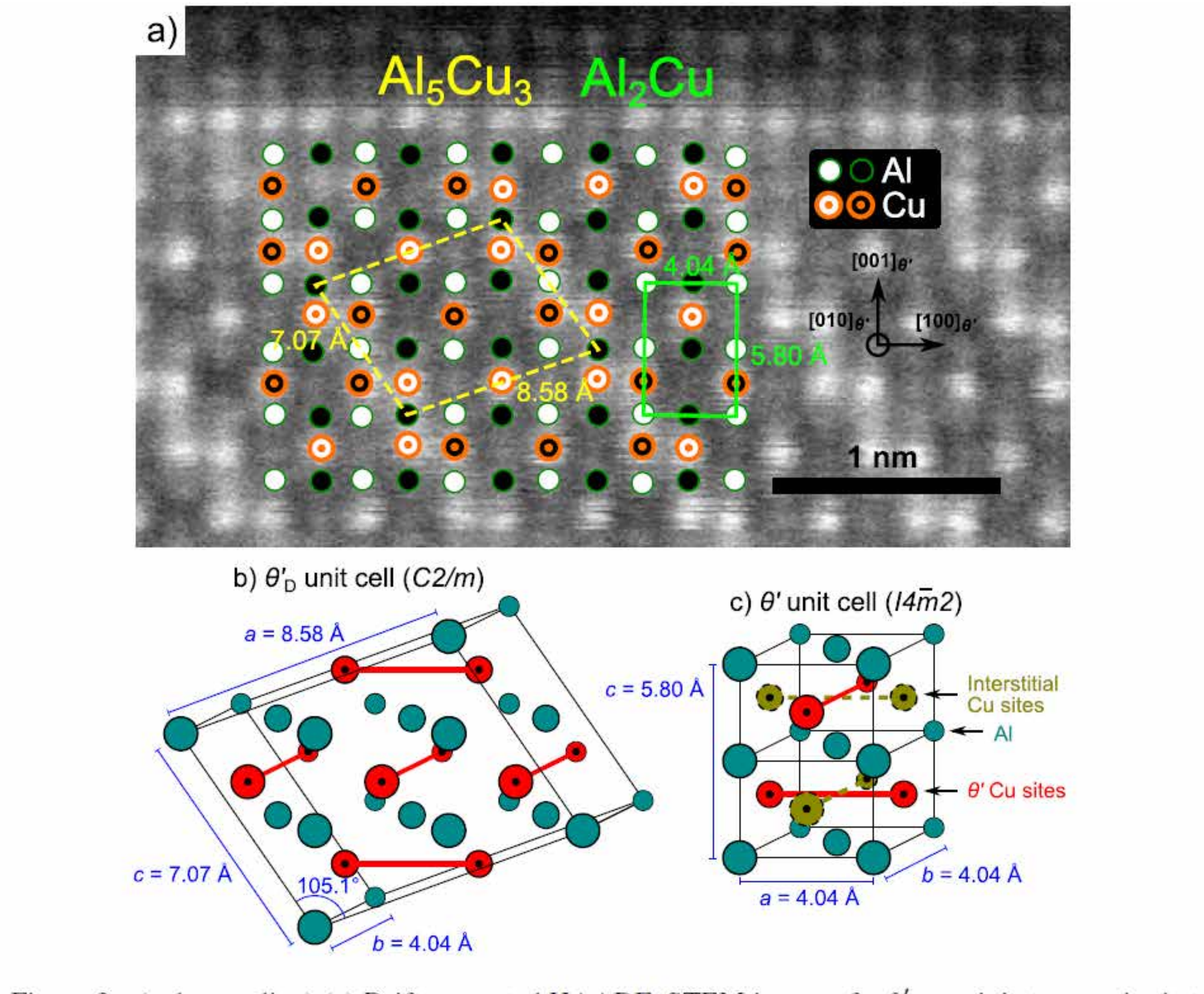

Figure 3. (colour online) (a) Drift-corrected HAADF–STEM image of a θ'_D precipitate, acquired at 80 kV. The overlay shows the atomic structure and unit cell of the pure and modified θ' structures. White/black fill distinguishes atoms that are spaced 202 pm in the viewing direction. (b) Three-dimensional model of the θ'_D unit cell. The structure is identical to θ' except that some Cu atoms are removed and others are added to the interstitial sites shown in (c).

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CHEMCATCHEM Full Papers

ZnO–Carbon-Nanotube Composite Supported Nickel Catalysts for Selective Conversion of Cellulose into Vicinal Diols

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A ZnO-nanolayer-coated carbon nanotube (CNT) composite (ZnO–CNT) was employed as a support to immobilise Ni catalysts. Well-mixed oxides, formed as a result of enhanced interfaces between the two oxides on CNTs, resulted in the formation of Ni–Zn alloys in reduced catalysts. The Ni/ZnO–CNT catalyst exhibited much higher yields of vicinal diols, for example, ethylene glycol and 1,2-propylene glycol, from the conversion of cellulose than the reference Ni/CNT catalyst. A plausible reaction network on the Ni/ZnO–CNT catalyst has been proposed. Moreover, the effects of Ni loading and catalyst reduction temperature on the catalytic performance were probed, and the relationship between the catalyst structure and the yields of vicinal diols was correlated. The Ni-rich alloy, relative to the Zn-rich alloy, was suggested as the dominating active phase for the selective conversion of cellulose into vicinal diols. This might shed new light on the design and optimisation of Ni-based catalysts for the catalytic conversion of cellulose with the Ni–Zn interaction.

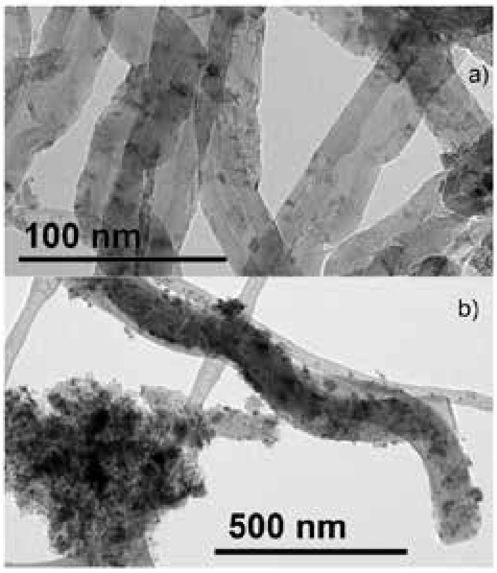

Figure 2. Typical bright-field TEM images of the reduced and passivated a) 20Ni/CNT and b) 20Ni/26ZnO–CNT catalysts.

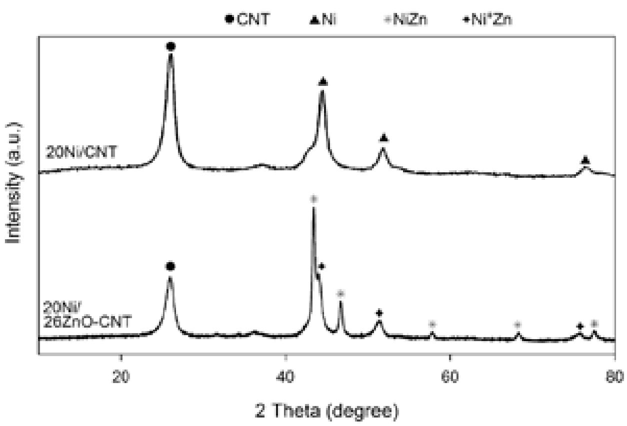

Figure 4. A comparison of the XRD spectra of the reduced and passivated 20Ni/CNT and 20Ni/26ZnO–CNT catalysts. ●: CNT; ▲: nickel; *: nickel–zinc alloy; ♦: Ni-rich nickel–zinc alloy.

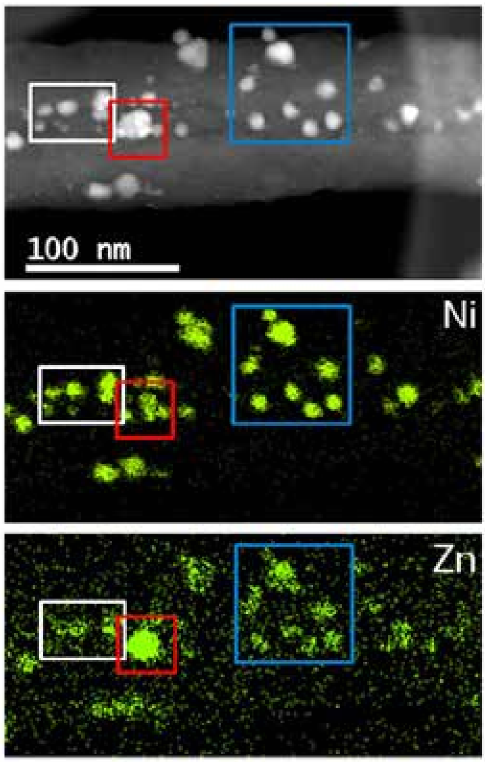

Figure 5. Dark-field TEM image (top) and EDS mapping of nickel (middle) and zinc (bottom) of the particles on the 20Ni/26ZnO–CNT catalyst. The white square indicates Ni-rich alloy particles, the blue square indicates nickel–zinc alloy particles and the red square indicates a Zn-rich particle.

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Size-Dependent Reaction Mechanism and Kinetics for Propane Dehydrogenation over Pt Catalysts

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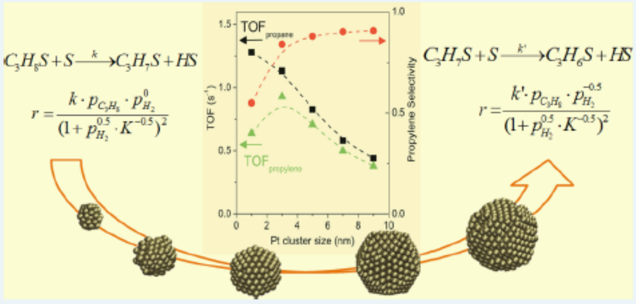
Supporting Information

ABSTRACT: Platinum cluster size has a significant influence on the activity, selectivity, and stability as well as the reaction mechanism during propane dehydrogenation (PDH). Well-controlled platinum catalysts of different cluster sizes are prepared by a seed growth method and supported on calcined hydrotalcite. The Pt catalysts show strong structure-sensitive behavior both in the C−H bond activation of propane and in the C−C bond activation to yield ethylene, methane, and coke. The Pt clusters of small cluster sizes, with (211) dominating on the surface, have a lower dehydrogenation energy barrier and thus higher activity. However, large Pt clusters with Pt(111) dominating result in a weakened binding strength of propylene and an increased energy barrier for the activation of C−H bonds in propylene, which leads to higher selectivity toward propylene by lowering the possibility of deep dehydrogenation. Kinetic analysis illustrates that the reaction order in hydrogen decreases and activation energy increases with an increasing Pt cluster size. Combined with density functional theory calculations and isotope effect experiments, it gives strong evidence of the change in reaction mechanism with Pt cluster size. It suggests that on small Pt clusters that are mostly surrounded by undercoordinated surface sites, the first C−H bond activation is likely to be the rate-determining step, while the second C−H bond activation is kinetically relevant on large Pt particles with terrace sites dominating.



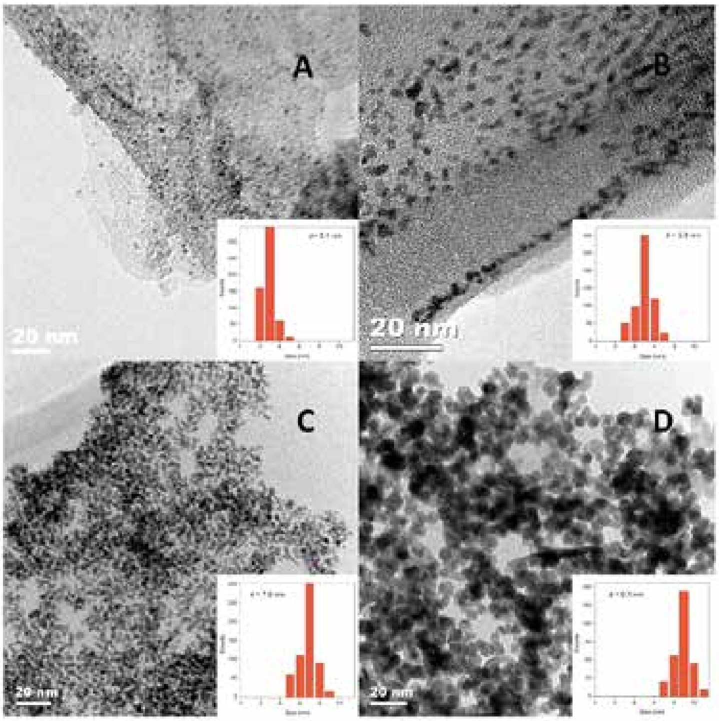


Figure 1. TEM images of Pt nanocolloids: (A) 3.1, (B) 5.3, (C) 7.0, and (D) 9.1 nm.

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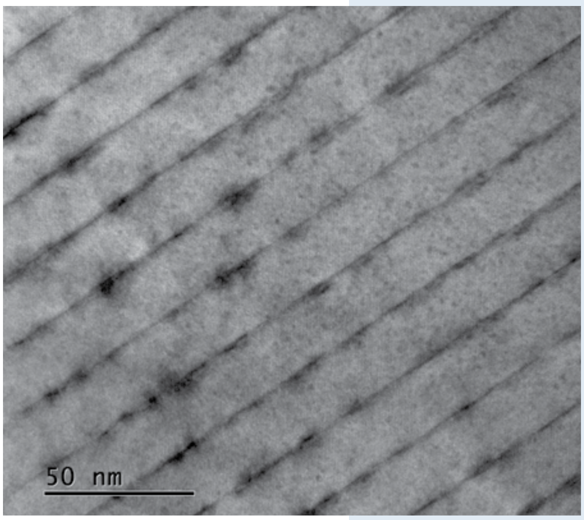

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S.J. **Andersen**, C. **Marioara**, R. **Holmestad**, J. Røyset, O. Reiso, U. Tundal, T. E. Nicolaisen and I. E. Opheim: "Advanced precipitate characterization and development of Al-Mg-Si alloys", 7th Light Metals Technology Conference, Port Elisabeth, South Africa, 27-29 July, 2015. [Invited talk]

E. **Christiansen**, M. **Nord**, I. Hallsteinsen, P. E. **Vullum**, T. Tybell and R. Holmestad, "Structural investigation of epitaxial (111) LaFeO3/SrTiO3 by transmission electron microscopy", MMC/EMAG2015, Manchester, UK., 29 June - 2 July 2015. [Poster]

P.I. Dahl, M. S. Thomassen, L.C.C. Rausseo, A. Barnet, P.E. **Vullum**, S.M. Hanetho, J.R. Tolchard, T. Mokkelbost, N.P. Wagner, A.M. Svensson, F. Vullum-Bruer, "Tailoring of Electrode Materials by Flame Spray Pyrolysis", 5th national meeting on inorganic and materials chemistry, Hell, Norway; October 15-16, 2015. [Poster]

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V. T. **Fauske**, M. **Erlbeck**, D. C. Kim, A. M. Munshi, D. L. Dheeraj, H. Weman, B.-O. Fimland and A. T. J. **van Helvoort**, "In-situ electronic probing of semiconducting nanowires in an electron microscope", 19th Microscopy of Semiconducting Materials, Cambridge, UK, 29 March - 2 April 2015. [Talk]

J. **Friis**, S. **Wenner**, C. **Marioara**, W. Lefebvre, S.J. **Andersen** and R. **Holmestad**, "Detailed structure analysis of precipitates combining TEM and DFT", International conference on Solid-Solid Phase transformations in Inorganic Materials (PTM-2015), Whistler, Canada, 28 June - 3 July, 2015. [Invited talk]

I. Hallsteinsen, M. Moreau, E. Folven, J. Grepstad, M.K. **Nord**, R. **Holmestad**, P.T.M. Tybell, "Magnetic coupling in (111)-oriented LSMO and LFO epitaxial heterostructures. Towards oxide based electronics (To-Be)", TO-BE Spring Meeting 2015 University of Aveiro, Aveiro, Portugal, 29 March - 4 April 2015. [Talk]

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A. T. J. **van Helvoort**, "Compositional Analysis of III-V Nanowires by Quantitative HAADF-STEM", EM seminar HREM group Cambridge, Cambridge, UK, 2 February 2015. [Talk]

A. T. J. **van Helvoort**, "III-V Nanowire-graphene hybrids", Hofmann Group meeting, Cambridge, UK, 15 April 2015. [Talk]

A. T. J. **van Helvoort**, "The study of III-V Nanowires by Quantitative HAADF-STEM" Seminar Department Materials Science Oxford, Oxford, UK, 7 April 2015. [Talk]

R. **Holmestad**, S. **Wenner**, T. **Saito**, E.A. **Mørtsell**, C. **Marioara** and S.J. **Andersen**, "TEM studies of precipitation in age hardenable aluminium alloys", Progress of Light Metals for the future - Retirement seminar for Prof. T. Sato, Tokyo Institute of Technology, Tokyo, Japan, 13 March 2015. [Invited talk]

R. **Holmestad**, A. T. J. **van Helvoort**, P. E. **Vullum**, R. **Sæterli**, B. G. **Soleim** and J. C. **Walmsley**, "NorTEM the national infrastructure for Transmission Electron Microscopy ", Nano-MatchMaking Seminar -NTNU NanoLab , Trondheim, Norway, 2 June, 2015. [Talk]

R. **Holmestad**, A. T. J. **van Helvoort**, C. **Marioara**, P. E. **Vullum**, R. **Bjørge**, S. **Wenner**, M. **Nord**, V. T. **Fauske**, R. **Sæterli**, B. G. **Soleim** and J. C. **Walmsley**, "Results from the first double corrected coldFEG TEM/STEM in Scandinavia", Scandem2015, Jyväskylä, Finland, 9-11 June 2015. [Talk]

R. **Holmestad**, S. **Wenner**, M. **Nord**, E.A. **Mørtsell**, P. E. **Vullum** and C. **Marioara**, "Materials development aided by atomic-resolution electron microscopy", Microscopy & Microanalysis 2015 Meeting, Portland, OR, USA, 2-6 August 2015. [Invited talk]

R. **Holmestad**, A. T. J. **van Helvoort**, P. E. **Vullum**, S. **Wenner**, M. **Nord**, V. T. **Fauske**, E.A. **Mørtsell**, M. **Vatanparast**, JS. **Nilsen**, A. **Mosberg**, E. **Christiansen** R. **Sæterli**, B. G. **Soleim**, C. **Marioara**, R. **Bjørge**,and J. C. **Walmsley**, " Instrumentation and activity at the Trondheim NORTEM – node (NTNU)", NORTEM opening seminar, Oslo, Norway, 10.-11.. September 2015. [Talk]

R. **Holmestad**, S. **Wenner**, E.A. **Mørtsell**, C. **Marioara** and S.J. **Andersen**, "(S)TEM studies of precipitation in age hardenable aluminium alloys", Talk given at Department of Materials Science and Engineering, Chongqing University,China, 19 November 2015. [Inviited talk]

R. **Holmestad**, S. **Wenner**, E.A. **Mørtsell**, C. **Marioara** and S.J. **Andersen**, "Precipitates in aluminium alloys – studied by HAADF-STEM", Workshop on Advanced Electron Microscopy and Characterisation, Inauguration of EM Centre at Chongqing University, Chongqing, China, 19-20 November 2015. [Invited talk]

R. **Holmestad** "Precipitation in 6xxx Aluminum Alloys", Guest lecture at Department of Materials Science and Engineering, Toyama University, Japan, 24 November 2015. [Lecture]

D. Johnstone, A. S. Eggeman, A. T. J. **van Helvoort** and P. A. Midgley, "Twinning & Polymorphism in GaAs Nanowires: A Scanning Precession Electron Diffraction Study", 19th Microscopy of Semiconducting Materials, Cambridge, UK, 29 March - 2 April 2015. [Talk]

D. Johnstone, A. S. Eggeman, A. T. J. **van Helvoort** and P. A. Midgley, "Mapping the structure of nanowires by scanning precession electron diffraction", Scandem2015, Jyväskylä, Finland, 9-11 June 2015. [Talk]

D. Johnstone, A. S. Eggeman, A. T. J. **van Helvoort** and P. A. Midgley, "Strain Mapping in III-V Semiconductor Nanowires by Scanning Precession Electron Diffraction (SPED)", Armourers and Brasiers' Cambridge Forum, Cambridge, UK, 16 June 2015. [Poster]

D. Johnstone, A. S. Eggeman, A. T. J. **van Helvoort** and P. A. Midgley, "Cartography by scanning precession electron diffraction", MMC/EMAG2015, Manchester, UK, 29 June - 2 July 2015. [Talk]

D. Johnstone, A. Eggeman, P. M. Midgley and A. T. J. **van Helvoort**, "Crystal phase mapping on the nanoscale", Nano@NTNU, Trondheim, 11 September 2015. [Talk]

B. Hjelmeland McDonagh, G. Singh, S. Hak, S. Bandyopadhyay, I. L. Augestad, L. Sandvig, A. Sandvig, , and W. Glomm, "L-DOPA-coated manganese oxide nanoparticles as dual MRI contrast agents and potential drug delivery vehicles", Nordic Neuroscience, Trondheim, Norway 10-12 June 2015. [Talk]

T. Mokkelbost, A. Fossdal, K. Jayasayee, J.R. Tolchard, P.E. **Vullum**, N.P. Wagner, F. Vullum-Bruer, "Study of $Li_2Fe_{1-x}Mn_xSiO_4$ Cathode Materials Synthesized Using Abundant Materials By Conventional Methods", 227th ECS Meeting, Chicago, USA, 24-28 May 2015. [Poster]

A. B. **Mosberg**, V. T. **Fauske**, and A. T. J. **van Helvoort,** "Construction of a prototype FIB heating stage", Nano@ NTNU, Trondheim, 11 September 2015. [Poster]

A. M. Munshi, D. Ren, D. L. Dheeraj, V. T. **Fauske**, L. M. S. Aas, A. T. J. **van Helvoort**, H. Weman, and B.O. Fimland, "On the growth of self-catalyzed GaAs nanowires on Si substrates: Opportunities and limitations", NAMBE2015, Mayan Riviera, Mexico, 4-7 October 2015. [Poster]

E. A. **Mørtsell**, C. D. **Marioara**, S. J. **Andersen**, J. **Friis**, O. Reiso, J. Røyset and R. **Holmestad**, "The Effect of Cu and Ge Additions on Strength and Precipitation in a lean 6xxx Aluminium Alloy ", MMC/EMAG2015, Manchester, UK, 29 June - 2 July 2015. [Poster, first prize EMAG]

M. Nematollahi, X. Yang, E. **Seim**, P.E. **Vullum**, R. **Holmestad**, U. Gibson, T.W. Reenaas, "Highly Cr-doped ZnS for intermediate band solar cells", 17th International Conference on II-VI Compounds and Related Materials, Paris, France, 13-18 September 2015. [Poster]

J. S. **Nilsen**, J. F. Reinertsen, A. B. **Mosberg**, V. T. **Fauske**, A. M. Munshi, D. L. Dheeraj, B. O. Fimland, H. Weman, and A. T. J. **van Helvoort**, "Radial composition variations in the shells of GaAs/AlGaAs core-shell nanowires", Nano@ NTNU, Trondheim, 11 September 2015. [Poster]

J. S. **Nilsen**, J. F. Reinertsen, A. B. **Mosberg**, V. T. **Fauske**, A. M. Munshi, D. L. Dheeraj, B.-O. Fimland, H. Weman and A. T. J. **van Helvoort**, "Self-Induced Formation of Quantum Structures in the Shell of GaAs/AlGaAs Core-Shell Nanowires", Myfab/NorFab user meeting 2015, Lund, Sweden, 21-22 April 2015. [Poster]

J. S. **Nilsen**, J. F. Reinertsen, A. B. **Mosberg**, V. T. **Fauske**, A. M. Munshi, D. L. Dheeraj, B.-O. Fimland, H. Weman and A. T. J. **van Helvoort**, "Compositional variations in the shells of GaAs/AlGaAs core-shell nanowires", MMC/EMAG2015, Manchester, UK, 29 June -2 July 2015. [Talk]

M. **Nord**, I. Hallsteinsen, P. E. **Vullum**, P. T. M. Tybell and R. **Holmestad**, "Analysing the electronic structure of perovskite oxides using Transmission Electron Microscopy ", Nano Network Annual Workshop, Oslo, Norway, 15 June 2015. [Poster]

M. **Nord**, I. Hallsteinsen, P. E. **Vullum**, P. T. M. Tybell and R. **Holmestad**, "Advanced quantitative fine structure analyzes of perovskite oxides using electron energy loss spectroscopy", MMC/EMAG2015, Manchester, UK, 29 June - 2 July 2015. [Poster]

J. F. Reinertsen, J. S. **Nilsen**, A. B. **Mosberg**, V. T. **Fauske**, A. M. Munshi, D. L. Dheeraj, B.-O. Fimland, A. T. J. **van Helvoort** and H. Weman, "Self-Induced Formation of Quantum Well Tubes in Self-Catalyzed GaAs/AlGaAs Core-Shell Nanowires", Heraeus-Seminar on III-V Nanowire Photonics, Bad Honnef, Germany, 22-25 March 2015. [Poster]

D. Ren, D.L. Dheeraj, J. Huh, J. F. Reinertsen, A. M. Munshi, D-C. Kim, A. T. J. **van Helvoort**, H. Weman, and B.-O. Fimland, "High Yield Self-catalyzed GaAsSb/GaAs Heterostructured Nanowire Array on Si (111) by Molecular Beam Epitaxy", Manowire growth workshop, Barcelona, Spain, 26-30 October 2015. [Talk]

D. Ren, D. L. Dheeraj, J. Huh, J. F. Reinertsen, A. M. Munshi, D.-C. Kim, A. T. J. **van Helvoort**, H. Weman, and B.-O. Fimland, "High-yield self-catalyzed GaAsSb/GaAs heterostructured nanowire array on Si(111) by molecular beam epitaxy", Nano@NTNU, 11 November 2015. [Talk]

G. Singh, "Design of Magnetic Nanoparticles and Their Self-assembly into Exotic Superstructures", International Workshop on Complex Magnetic Nanostructures, Agenia, Greece, 2-5 June 2015. [Talk]

G. Singh, F. Seland, and S. Sunde, "Design of nanoporous nanoparticles and their electrocatalytic activity", KIFEE International Symposium on Environment, Energy and Materials; Trondheim, Norway, 20-23 September, 2015. [Talk]

G. Singh, "Self-assembling colloidal nanomaterials at different length scale for nanobiotechnology applications", Colloids and Surfaces in Biology and Biomaterials Symposium, Uppsala, Sweden, 4-6 November 2015. [Talk]

S. **Wenner**, M. **Ervik**, C. **Marioara**, S. J. **Andersen** and R. **Holmestad**, "Atomic Resolution Electron Microscopy of a Hybrid Aluminium Alloy and All Its Phases", Scandem2015, Jyväskylä, Finland, 7-11 June 2015. [Talk]

S. **Wenner**, E. **Christiansen**, T.S. **Holstad**, R. **Holmestad**, "Krystaller på atomnivå" / "Se innsiden av et elektronmikroskop", Researchers' Night, NTNU, Trondheim, 25 September 2015. [Stand / Demo]

PhD theses

Jon **Holmestad**, "(Scanning) Transmission Electron Microscopy Studies of Grain Boundary Segregation relevant to Intergranular Corrosion in Al-Mg-Si-Cu Alloys", PhD Thesis NTNU, 2015.

Master projects/theses

Tina **Bergh**, "TEM characterization of SiC powders" (Project, December 2015).

Emil **Christiansen**, "TEM Characterization of $LaFeO_3$ Thin Films on $SrTiO_3$ Substrates" (Diploma, June 2015).

Andreas **Garmannslund**, "Processing multidimensional transmission electron microscopy data sets" (Project, December 2015).

Theodor Secanell **Holstad**, "TEM Characterization of $BaTiO_3$ Thin Films on $SrTiO_3$ (111) Substrates" (Project, December 2015).

Joakim **Larsen**, "TEM of Chromium doped Zinc Sulfide Thin Films for Solar Cell Applications" (Diploma, June 2015).

Aleksander B. **Mosberg**, "Characterization of AlGaAs shell structure in GaAs/AlGaAs Core-shell Nanowires" (Diploma, June 2015).

Jonas **Sunde**, "Precipitation of Several Coexisting Strengthening Phases in Aluminium Alloys", (Project, December 2015).

Workshops

Half day seminar on Advanced TEM with Joanne Etheridge (Monash, Australia), 13 September 2015.

HyperSpy user workshop by V. T. Fauske and M. Nord, 17 September, 2015.

Popular science contributions/Media

F. M. Lindboe, "TEM: 7. forsker i huset - The 7th Researcher of the house", https://www.youtube.com/watch?v=BuLqv4_cIMU [Video]

F. M. Lindboe, "TEM: Hvordan identifisere stoffer? EDX!", https://www.youtube.com/watch?v=9YKjTKE56Uo [Video]

F. M. Lindboe, "TEM: The Old Generation!", https://www.youtube.com/watch?v=L7ig4hNZ7-Q [Video]

"Norge betaler 120 millioner for fem helt spesielle mikroskoper", Tekniske Ukeblad, 11 September, 2015.

R. Sæterli and A.T.J. van Helvoort, "Mikroskoper til 120 millioner kroner", Her og nå hovedsending, 11 September, 2015. [Radio interview] Link: https://radio.nrk.no/serie/her-og-naa-hovedsending/DMNH01018015/11-09-2015#t=18m42s

"Supermikroskop gir trønderforskere tilgang til en helt ny verden", Adressa, 12 September, 2015.

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