

# ANNUAL REPORT **2014** TEM GEMINI CENTRE

Department of Physics, NTNU, Department of Materials Science and Engineering,  
NTNU, Department of Synthesis and Properties, SINTEF Materials and Chemistry

*Graphic design: Ingebjørg Fyrileiv Guldvik, Sigurd Wenner  
Photo: Terje Trobe, Ole Morten Melgård  
TEM images: Scientists at the TEM Gemini Centre*

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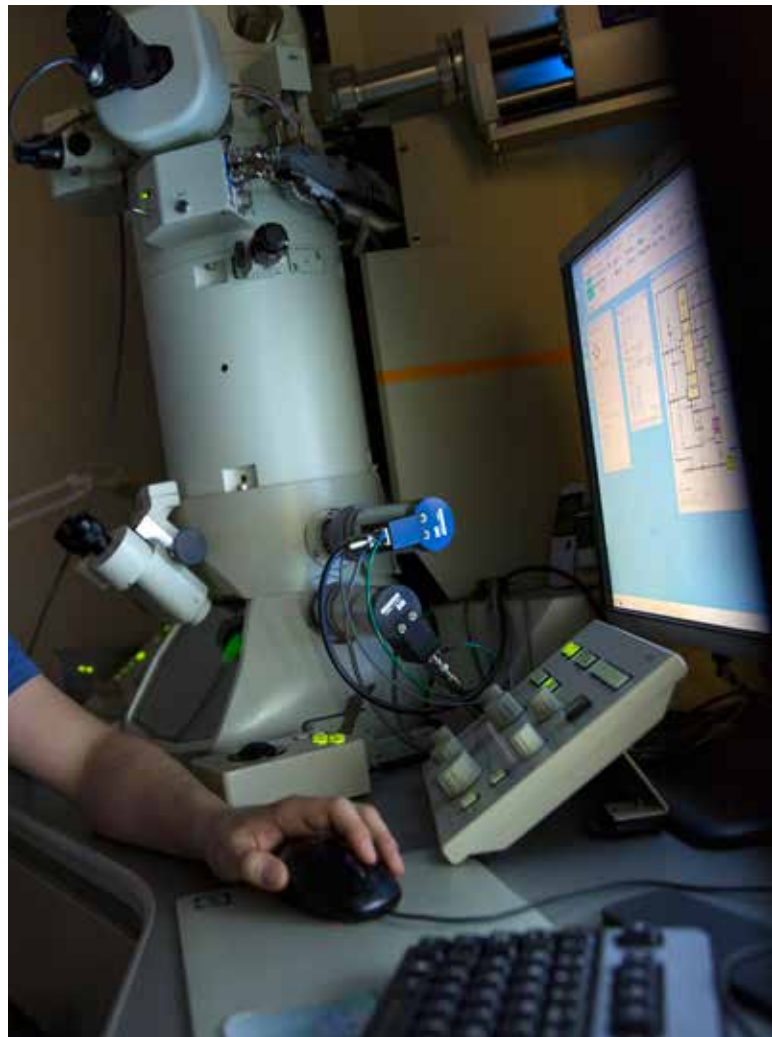


## THE TEM GEMINI CENTRE

The TEM (Transmission Electron Microscopy) Gemini Centre was established in March 2006 and consisted of professors, postdocs, students and engineers from Department of Physics (DP), NTNU and researchers from the Material Physics research group, a part of the Department of Synthesis and Properties in SINTEF Materials and Chemistry.

In June 2009 the TEM Gemini 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. As a consequence all TEM facilities related to physical sciences in Trondheim are under one umbrella and the access is more open to groups outside the Centre. The same constellation was renominated in November 2012 for another period of 3 years.

The TEM Gemini 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. 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. A large infrastructure project, NORTEM, has given a national identity to TEM, and the discussion about how to run the Gemini Centre in the future has started.



## THE GEMINI CENTRE CONCEPT

The 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 vision: To be an international joint force at the leading edge of research.

## INTRODUCTION

2014 has been the first year with the new NORTEM infrastructure up running and marks the transition to use of the new facilities. The NORTEM project described in the further is a partnership between NTNU, UiO, SINTEF and the Research Council of Norway. The Gemini Centre is establishing routines and its role in the NTNU/SINTEF system. The need for development of the national role, and strategic objectives, such as increased activity at the European level, should be addressed. The instruments cover a wide range of activities within teaching and research. Instrument use has increased over the last year, showing that the new equipment is being utilized quickly. This has been a great achievement and required a great deal of hard work from everyone involved within the group.

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 is gratefully acknowledged.

In November 2012 we were re-nominated for 3 new years as a Gemini Centre. We got a positive evaluation and feedback on how the Centre has functioned in the last years. Since the nomination is for 3 years, a new re-nomination is due fall 2015. Looking forward we are discussing the role of the TEM Gemini Centre and the relation to the national NORTEM project.

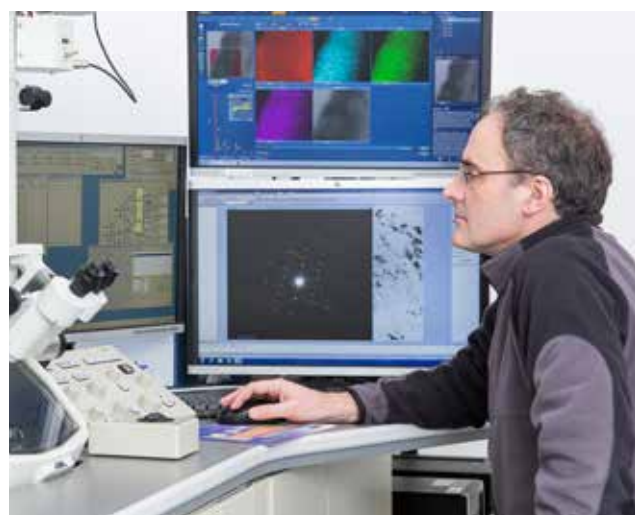
The Gemini Centre participates in a range of internal and external projects, including national public and industrial and EU funding. At the end of 2014 we got the good news that the TEM Gemini Centre is participating in three new long-term SFI projects – Centre of advanced structural studies (CASA), Sustainable Innovations for Automated Manufacturing of Multi-Material Products (Manufacturing) and Industrial Catalysis Science and Innovation for a competitive and sustainable process industry (iCSI). These are projects starting up in 2015, and the Centre will participate with PhD students in all of them.

The Centre had about 34 operators of TEM and produced more than 40 journal publications in 2014. 16 of these publications have international co-authors. In addition, 5 Masters and 3 PhD candidates were educated with TEM as a substantial part of their theses. Three courses, with a total of approximately 130 students, used the facilities as part of their education at NTNU. We organized a TEM introduction course with 15 participants in November. We have organized group meetings almost every week, and have given many guided tours for high school students to the microscopes. As seen from the publica-

tion list at the end, most members of the TEM group participated in international conferences and meetings in 2014. The Centre hosted an international STEM workshop in January, which is described in detail later in the report.

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 2014. 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  
April 2015.



## BOARD AND MANAGEMENT

**During 2014 the TEM Gemini Centre board has consisted of:**

- **Ragnar Fagerberg**, Research director, SINTEF Materials and Chemistry
- **Erik Wahlström (Mikael Lindgren until 31/07)**, Department head, Department of Physics, NTNU
- **Jostein Mårdalen**, Department head, Department of Materials Science and Engineering, NTNU

**The daily 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
- **Yingda Yu**, Materials Science, NTNU

The Centre had one board meeting, and a one-day strategy meeting in June 2014.

### **Key staff**



*Randi Holmestad, Professor, Physics, NTNU, leader, John C Walmsley, Senior scientist/Professor II, SINTEF Materials and Chemistry, NTNU, Ton van Helvoort, Professor, Physics, NTNU, Bjørn Gunnar Soleim, Senior engineer, Physics, NTNU, Ragnhild Sæterli, Senior engineer, Physics, NTNU, Yingda Yu, Senior engineer, Materials Science and Engineering, NTNU.*

# VISION OF NORTEM

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.

## 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. In 2012 NORTEM signed contracts with the TEM vendors. In Trondheim we signed a contract with JEOL. It included three TEM instruments on three different user levels (state-of-the-art, advanced and routine). In the contract we also agreed that JEOL should be responsible for the rebuilding of areas in the basement of Kjemiblokk 1 (K1), close to NTNU NanoLab. The fact that JEOL has been actively involved in the planning and rebuilding phase has been crucial for obtaining the best possible solutions for the infrastructure. In addition, the TEM Gemini Centre and JEOL have agreed on a cooperation agreement (JEOL Competence Centre (JCC) to develop the facility and the users' competence in the coming years.

The aim of NORTEM is to realize and provide access to modern TEM equipment 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 will 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 fair and 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 new 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. Attention has been paid to addressing the challenge of establishing and getting the best out of the facilities. From 2014 Ragnhild Sæterli has been recruited within the project to work alongside Bjørn Soleim in building-up and supporting maintenance, training, competence and techniques. In addition, Per Erik Vullum has been recruited into an Adjunct Professor position, which will particularly contribute to developing spectroscopy and reciprocal interaction between NTNU and SINTEF.

Rebuilding, installation and training on the new equipment was the prime focus and required a lot of the Centre's resources in 2013 and 2014. The transition period from the old to the new instruments has gone smoothly, and we are gradually building up routines and competence on the new instruments.

For more information on NORTEM see the webpages at [www.nortem.no](http://www.nortem.no).









Group picture TEM Gemini Centre, February 2014

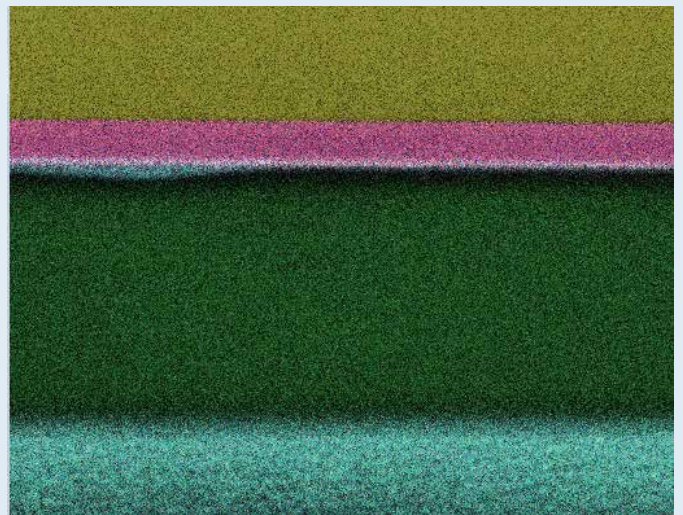
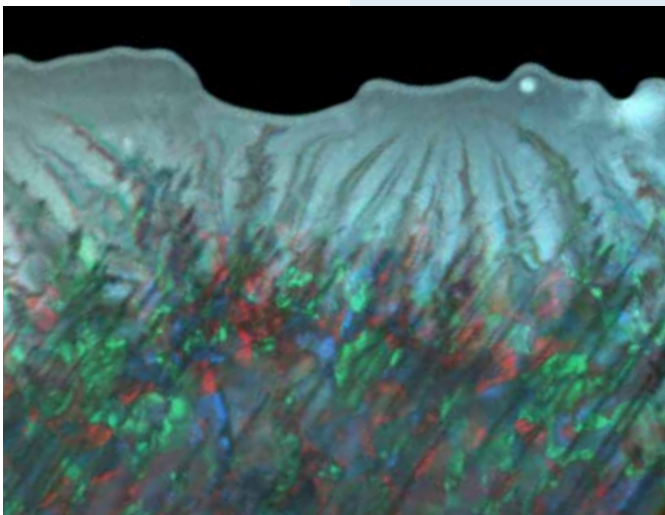
Photo: Ole Morten Melgård

Randi Holmestad (Professor, DP, NTNU / Leader TEM Gemini Centre)  
 John Walmsley (SINTEF senior scientist and Professor II, DP, NTNU)  
 Ton van Helvoort (Associate professor/Professor from Sept. 2014, DP, NTNU)  
 Knut Marthinsen (Professor, DMSE, NTNU)  
 Jan Ketil Solberg (Professor Emeritus, DMSE, NTNU)  
 Yanjun Li (Research scientist, SINTEF)  
 Per Erik Vullum (Research scientist, SINTEF and assoc. Prof II, NTNU)  
 Bjørn Gunnar Soleim (Senior engineer, DP, NTNU)  
 Ragnhild Sæterli (Senior engineer, DP, NTNU)  
 Yingda Yu (Senior engineer, DMSE, NTNU)  
 Sigmund J. Andersen (Senior scientist, SINTEF)  
 Jesper Friis (Research scientist, SINTEF)  
 Calin Marioara (Research scientist, SINTEF)  
 Ruben Bjørge (Research scientist, SINTEF)

Flemming Ehlers (Postdoc, DP, NTNU)  
 Yanjie Gan (Postdoc, DP, NTNU)  
 Jon Holmestad (PhD student, DP, NTNU)  
 Astrid Marie F. Muggerud (PhD student, DP, NTNU)  
 Takeshi Saito (PhD student, DP, NTNU)  
 Sigurd Wenner (PhD student, DP, NTNU)  
 Vidar Tonaas Fauske (PhD student, DP, NTNU)  
 Magnus Nord (PhD student, DP, NTNU)  
 Eva Anne Mørtzell (PhD student, DP, NTNU)  
 Martin Ervik (PhD student, DP, NTNU)  
 Maryam Vatanparast (PhD student, DP, NTNU)  
 Mami Mihara (PhD student from Tokyo Tech, DP, NTNU)  
 Espen Undheim (Master student, DP, NTNU)  
 Eivind Seim (Master student, DP, NTNU)  
 Toshiki Omi (Master student from Tokyo Tech, DP, NTNU)  
 Emil Christiansen (Master student, DP, NTNU)  
 Maximillian Erlbeck (Project student, DP, NTNU)  
 Julie Stene Nilsen (Project student, DP, NTNU)  
 Ørjan Berntsen (Master student, DP, NTNU)  
 Alexander Mosberg (Project student, DP, NTNU)  
 Joakim Larsen (Project student, DP, NTNU)

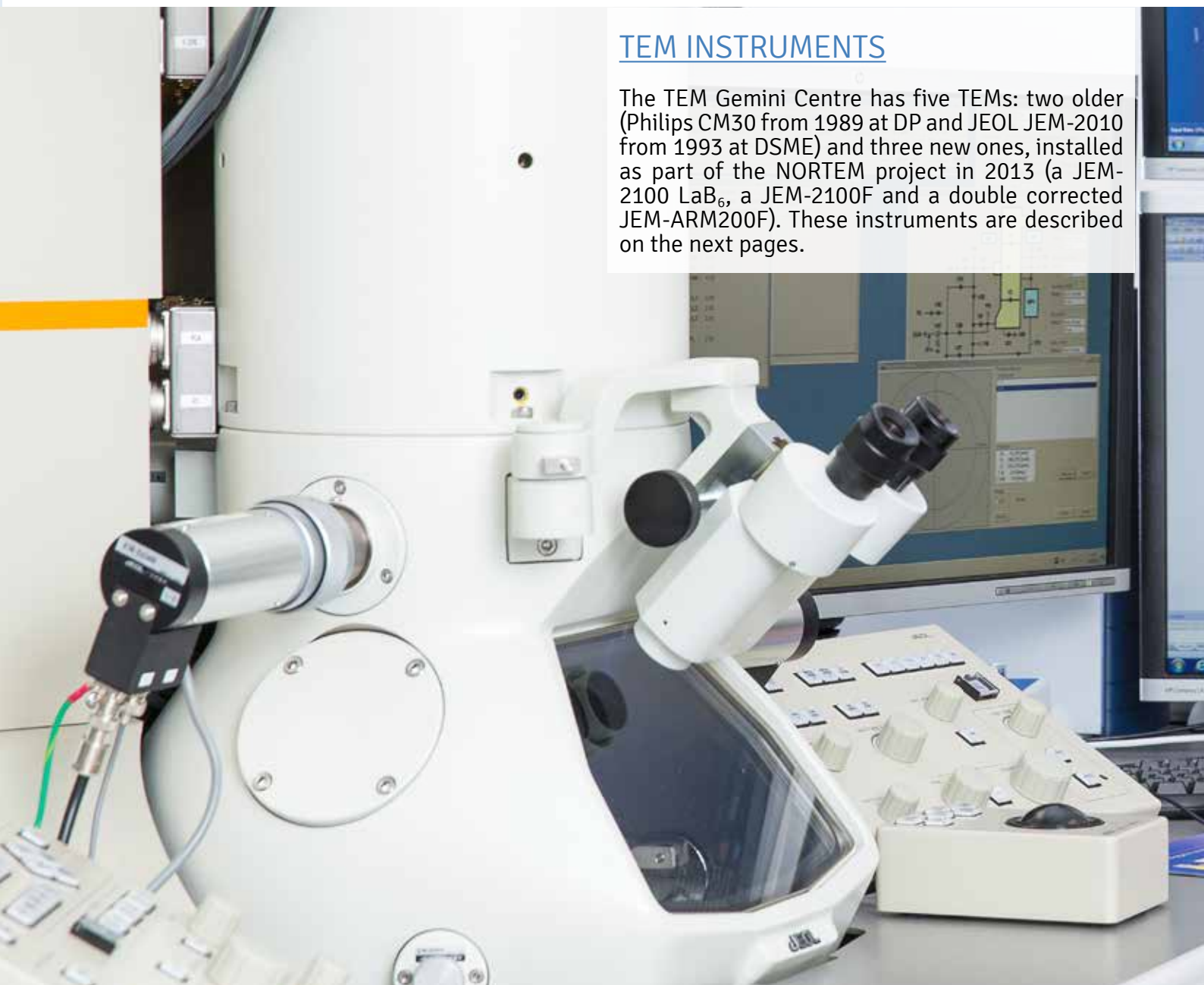


# INSTRUMENTATION



## TEM INSTRUMENTS

The TEM Gemini Centre has five TEMs: two older (Philips CM30 from 1989 at DP and JEOL JEM-2010 from 1993 at DSME) and three new ones, installed as part of the NORTEM project in 2013 (a JEM-2100 LaB<sub>6</sub>, a JEM-2100F and a double corrected JEM-ARM200F). These instruments are described on the next pages.



### Microscopes in the TEM Gemini Centre today.

Instrument	Location	Configuration	Installed
<i>Philips CM30</i>	<i>DP, Natural Science building</i>	<i>LaB<sub>6</sub>, 300 kV, PEELS, EDS, 1k camera</i>	<i>1989</i>
<i>JEOL 2010</i>	<i>DMSE, Alfred Getz vei 2</i>	<i>LaB<sub>6</sub>, 200 kV, GIF, EDS, 2k camera</i>	<i>1993</i>
<i>JEOL 2100</i>	<i>DP, Chemistry block I</i>	<i>LaB<sub>6</sub>, 200 kV, STEM, GIF, EDS, Orius camera</i>	<i>Aug. 2013</i>
<i>JEOL ARM-200F</i>	<i>DP, Chemistry block I</i>	<i>Cold FEG, image and probe corrected, Quantum GIF, Centurio EDS, CCD cameras</i>	<i>Oct. 2013</i>
<i>JEOL 2100F</i>	<i>DP, Chemistry block I</i>	<i>FEG, 200 kV, EDS, US camera, ASTAR, gas reaction and tomography holders</i>	<i>Dec. 2013</i>



# 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, along with a support/preparation area and a computer room.



## **JEOL JEM-2100**

*The 2100 LaB<sub>6</sub> 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

A broad range of holders is available for use on all three microscopes. This includes single tilt holders, double tilt holders ( $\pm 30$  degrees), a cold stage holder, a heating holder, an environmental cell/transfer holder and tomography holders. A tilt-rotation holder and a new double tilt holder have been purchased in 2014. In addition, investments in a new heating holder will be done early in 2015.

## OLDER TEM INSTRUMENTS

The CM30 is now 26 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. We expect to continue to use the CM30 for some more years.

The JEOL 2010 at DMSE is 22 years old and provides similar analysis to the CM30. It has its main application areas in metallurgy and nanotechnology. It is used in undergraduate and post-graduate research and teaching.

Maintaining the performance of these two older machines is proving more demanding. This illustrates the importance of experienced local technical support, continuous evaluation and re-investment in large-scale facilities.



*The Philips CM30.*

## SPECIMEN PREPARATION



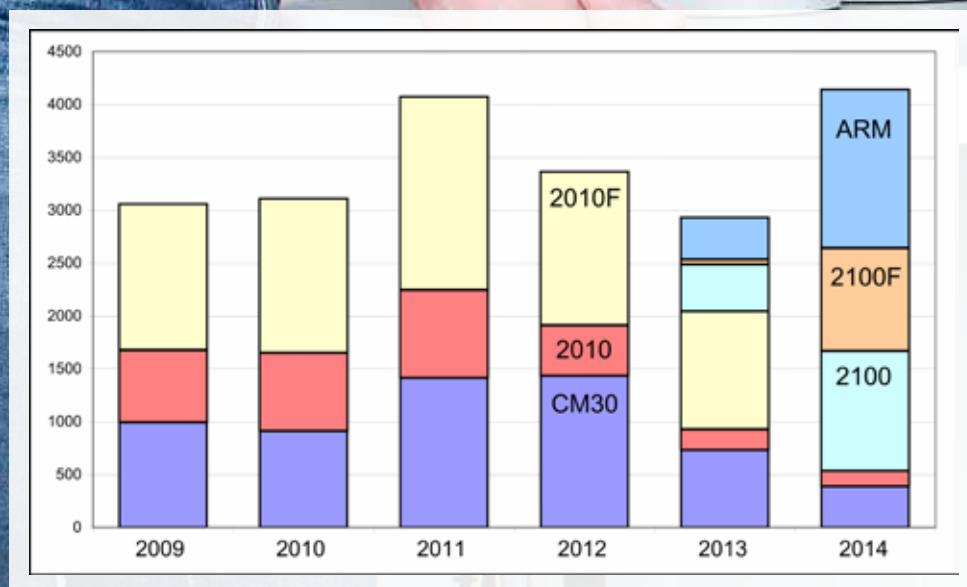
*PhD student Takeshi Saito, who graduated in 2014, preparing Al specimens by electropolishing.*

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 ion mills, dimplers and other tools for TEM specimen preparation of metal and ceramic cross sectional specimens. 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 attractive preparation technique. It 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 limiting factor in a (S)TEM study is often the specimen quality rather than the microscope. We see that this is particularly true for imaging and analysis in aberration corrected instruments. The new instruments have highlighted the need to upgrade the specimen preparation facilities, which has started in 2014 and is also continuing in 2015.

In particular, specimen contamination has been one of the limiting factors of high-quality (S)TEM work. The Centre has therefore invested in an ozone cleaner. A new generation ion miller and a dedicated pumping station for cleaning and storing holders and delicate specimens will be installed early 2015.





Historical microscope usage in hours per year. This shows the transition to the three NORTEM instruments in 2013.



## USER STATISTICS IN 2014

There were in total 34 operators of the four DP microscopes in 2014. Of these are 4 SINTEF users, who can give access to industry/third parties. There were 26 users from DP, 2 users from DMSE and 2 users from other departments.

The first table below shows hours which are written down in the log book ("real hours spent"), for the four microscopes. The second table below shows an overview of days the microscopes were used during the year. Because of all the work done to build up competence on the new TEMs and build up a broader user base, the overall use was high throughout the year, and is expected to increase further.

### Use in hours

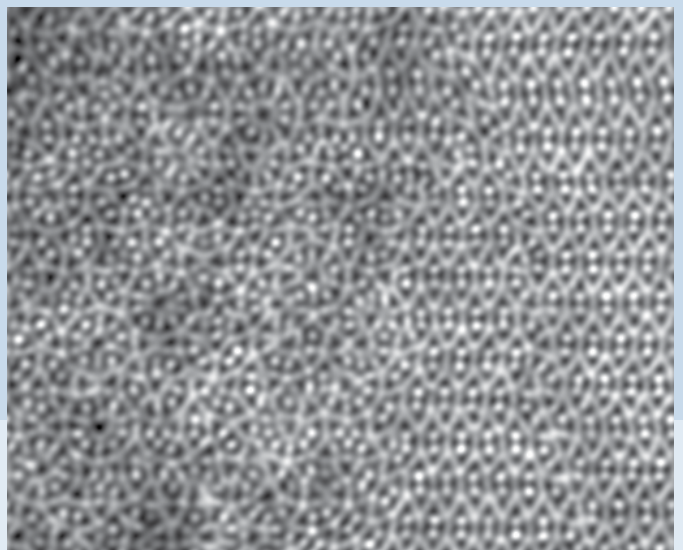
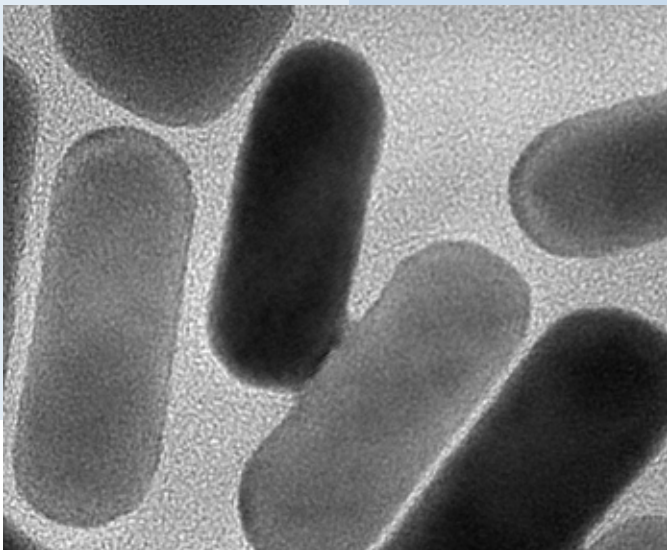
User	ARM-200F	2100F	2100	CM30	2010
	<i>hours</i>	<i>hours</i>	<i>hours</i>	<i>hours</i>	<i>hours</i>
SINTEF	<b>545</b>	<b>237</b>	<b>62</b>	<b>132</b>	<b>0</b>
NTNU	<b>523</b>	<b>573</b>	<b>774</b>	<b>253</b>	<b>113</b>
Physics	523	563	661	253	0
IMT	0	0	29	0	113
Other departments	0	10	84	0	0
External	0	0	0	0	0
NTNU Teaching lab	0	20	101	0	40
NorTEMnet workshop	27	41	37	0	0
NTNU, Set-up/testing/training/ demonstrations	406	101	162	2	0
<b>Total use</b>	<b>1501</b>	<b>972</b>	<b>1136</b>	<b>387</b>	<b>153</b>

### Use in days

	ARM-200F	2100F	2100	CM30	2010
Use on work days	194	128	182	68	36
Use in weekends	37	23	23	7	1
<b>Total days in use</b>	<b>231</b>	<b>151</b>	<b>205</b>	<b>75</b>	<b>37</b>
Downtime	6	35	1	18	211
Work days unused	50	87	67	164	3

# ACTIVITIES IN 2014

## 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, solar cell research and nanotechnology. TEM plays an important role in these research areas, which will be strategically important for Norway in the future. The TEM Gemini Centre had activities in all these four areas in 2014. The next sections describe these activities. Particularly strong were the activities in aluminium alloys and light metals. The study of aluminium alloy systems using TEM has been a pillar in the Trondheim TEM environment for many years.

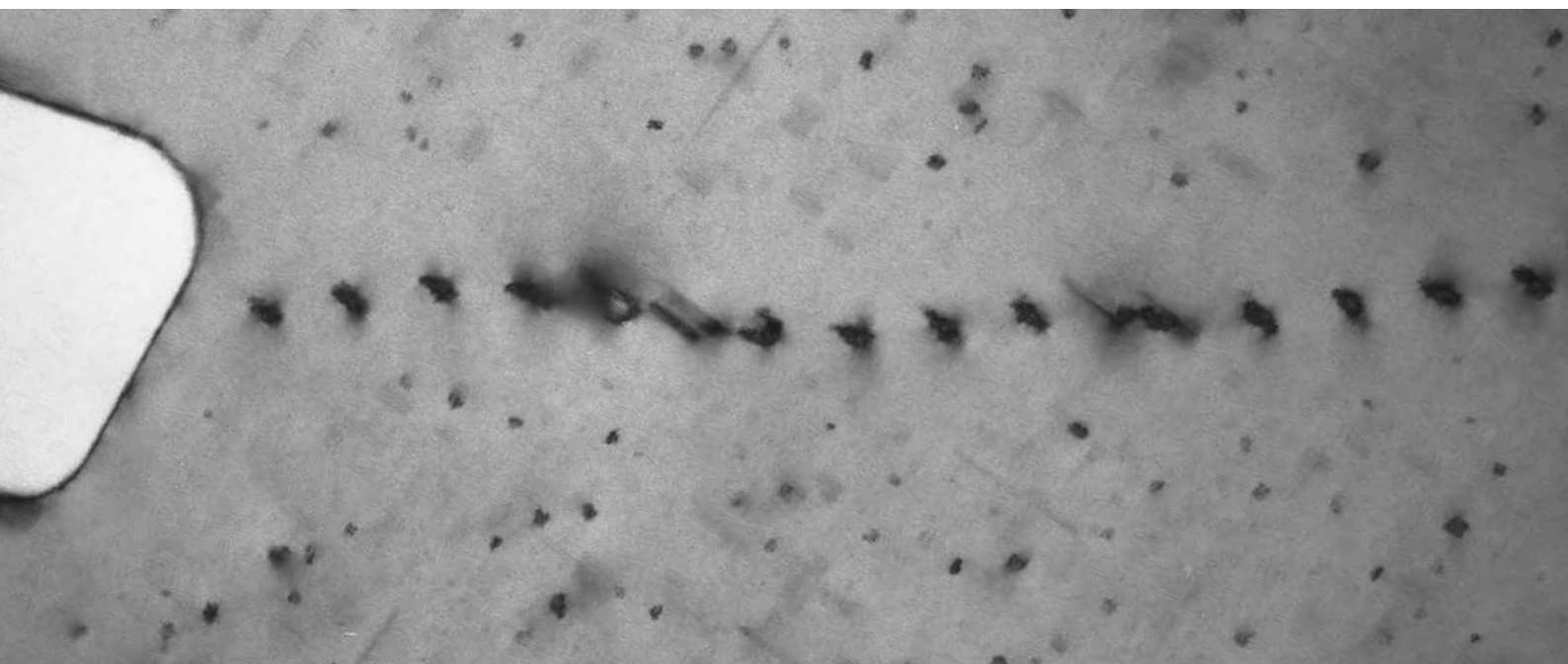
## ALUMINIUM ACTIVITIES (LIGHT METALS)

The KK (Nucleation Control) project ended in 2012, although Jon Holmestad has been working on this project in 2014, as he started his PhD work late in the KK project. Jon studies grain boundary segregation and connection to precipitates and corrosion properties. The Hydro supported Research council funded project SALSA (Sustainable Aluminium Surface Applications) project ended in 2014. In this project TEM has been one of several characterisation tools which have been used in interdisciplinary studies to relate nano-scale understanding of microstructure and chemistry to macroscopic electrochemical and corrosion properties. The Research council eVITA project Multimodal (Multiscale modelling in Aluminium) also ended in 2013. In this project Flemming Ehlers worked as a postdoc on atomistic modelling of precipitates and interfaces. In addition, SINTEF researchers were involved, combining several types of modelling to get more insight into precipitate-matrix interfaces. Several papers have been published in this project in 2014. The competence project MoReAl, which focused mainly on 3xxx alloys, ended in 2014. SAPA and Hydro was involved and contributed financially, and several PhD students have worked on this project, using SEM

and TEM. PhD student Astrid Marie F. Muggerud focused on different TEM techniques used on dispersoids in her study, which finished in September 2014.

The bilateral competence building project 'The Japanese-Norwegian Al-Mg-Si precipitation project' ended in 2014. Here Sigurd Wenner and Takeshi Saito both finished their PhD theses in 2014, both working together with SINTEF scientists. The scientific output from this project measured in refereed journal publications has been high in 2014. The main topic has been to study the effects of small additions of trace elements on precipitation in the context of recycling by TEM. In addition to TEM, we have also used muon spin relaxation to get more information on vacancies and defects. Two visitors from Tokyo Institute of Technology stayed in the TEM Gemini Centre in 2014 as a result of the Japanese collaboration (see under).

We have now two ongoing aluminium projects. The first one is 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 Mørtzell is doing a PhD within this project which, as the name indicates, 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. In addition to SINTEF activity, also Master student Toshiki Omi from Tokyo Institute of Technology has been working on this project up to June 2014. The second is the FRINATEK Research Council project called ‘Fundamental investigations of precipitation in the solid state with focus on Al-based alloys’. Here Martin Ervik started his PhD in the fall of 2013. Sigurd Wenner started as a postdoc in this project in April 2014, dedicated to HAADF STEM. In collaboration with SINTEF scientists, Martin and Sigurd are 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. Mami Mihara from Tokyo Institute of Technology visited our group for 4 months in 2014, connected to the FRINATEK project. The two last projects will keep our Aluminium TEM activities high in the coming 4 years period.

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 will be involved in the “lowest scale” of the multiscale activities, including TEM of precipitates, dislocations and interactions between them, mostly in industrial 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 the TEM Gemini Centre will be studying the microstructure of these materials.

TEM Gemini group members actively participate in international aluminium scientific meetings. In June 2014 the international ICAA conference (<http://www.ntnu.edu/icaa14>) was held in Trondheim, with many contributions from the TEM Gemini Centre. In addition to an invited keynote talk given by Randi Holmestad, our group gave 7 contributed talks and presented 3 posters.

## SOLAR CELLS

To improve the efficiency of conventional and novel types of solar cells, materials and devices are optimized at the length scale where TEM is one of the main characterization tools. TEM Gemini Centre activities within solar cells include both studies of conventional silicon solar cell materials and new materials, including InAs quantum dots in GaAs and GaAs nanowires for solar cell applications. Two 2-year Master students, Eivind Seim and Espen Undheim handed in their theses in spring 2014. The RENERGI project Nanosolar – Core-shell ended in 2013, in collaboration with Profs. Weman and Fimland at IET-NTNU, and a follow-up project was granted (Low Cost, Ultra-High Efficiency Graphene Nanowire Solar Cells (GRANASOL)) in the NRF program NANO2021. TEM on high-performance nanowire-based solar cells will be a crucial element of the project.

In 2013 we started up a new research council project within the Energix programme, called High Efficiency Quantum Dot Intermediate Band Solar Cells (HighQ-IB) where associate Prof. Turid W. Reenaas is the project leader. Maryam Vatanparast works as a PhD student on this project, and SINTEF has an active role. The new ARM microscope will be used heavily to understand the formation and performance of the quantum dots. SINTEF is a participant within this project.

The TEM Gemini Centre is participating in the FME (Centre of Environmental Friendly Energy) on solar cells, and has had some SINTEF activity in this project during 2014. Espen Undheim became a PhD student at IMT within this project, and plans to use TEM in his project on nucleation and early growth of silicon for solar cells.

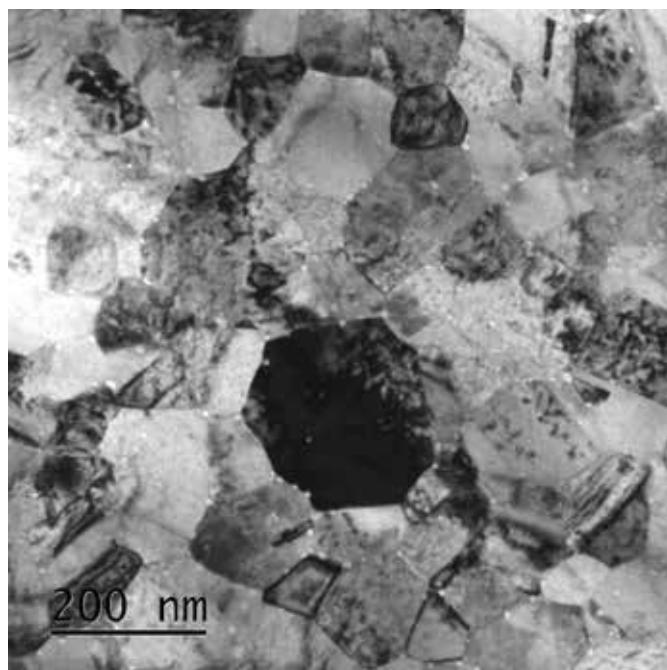
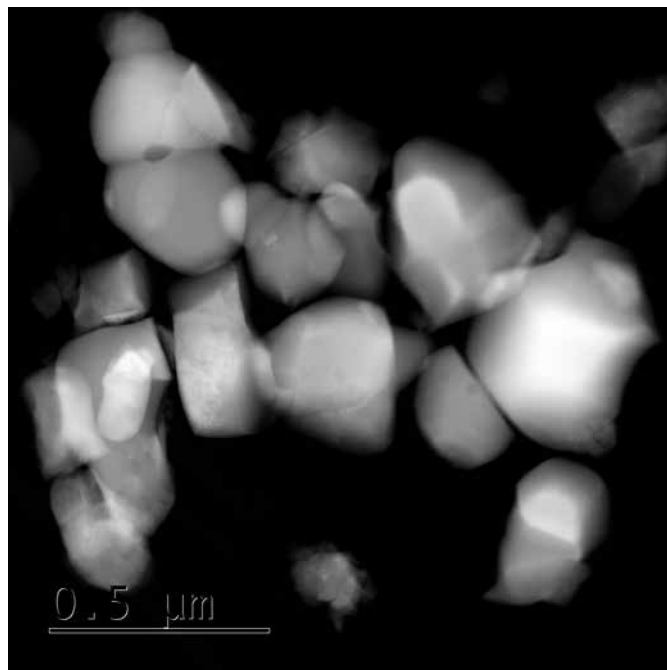


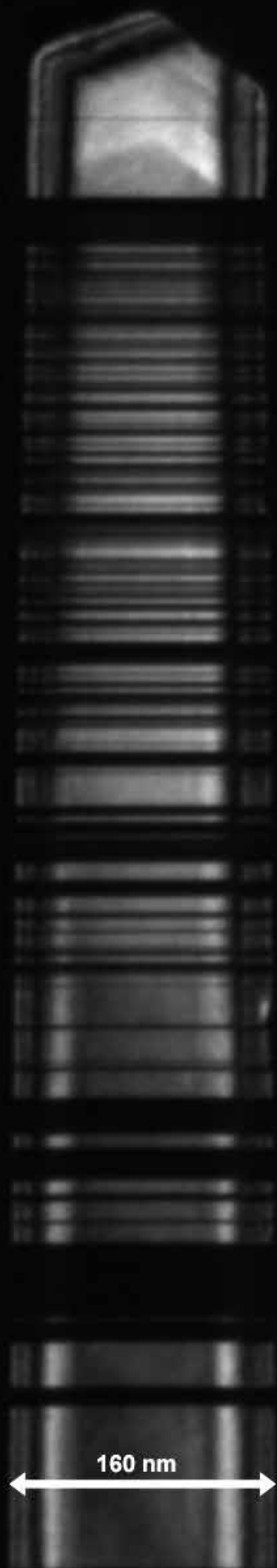
## CATALYSIS AND MEMBRANE MATERIALS

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 (IKP) and the inGAP SFI, Innovative natural Gas Processes and Products. During 2014 we have worked towards applying the new infrastructure to the study of catalyst systems, including supported metal nanoparticles, oxides and other systems such as doped carbon nanotubes.

A Gassmaks programme project lead by Profs. De Chen and Hilde Venvik at NTNU IKP to develop catalysts and materials for a compact steam reformer has now started. Here we will address both catalyst and materials issues, such as degradation of steels by metal dusting corrosion during exposure to synthesis gas. We also participated in an ongoing industrial project lead by the SINTEF Materials and Chemistry Oil and Gas Process Technology Department for SABIC. 2014 was the last full year of the inGAP SFI but the new SFI, Industrial Catalysis Science and Innovation for a Competitive and Sustainable process Industry (iCSI), headed by Hilde Johnsen Venvik, will be using TEM to study systems that are in direct relevance to the industrial participants in the project. There is activity in several other projects, both national and EU funded.

Yanjie Gan has worked in 2014 as a postdoc contributing TEM analysis to a researcher project in the CLIMIT programme, lead by Thijs Peters at SINTEF, on Pd-based hydrogen storage membranes. This continues a long-term cooperation in the study of these materials and allows us to contribute further with understanding the membrane microstructure and chemistry process and the changes that occur when they transport hydrogen and are exposed to other gases. Membranes with more complex alloy compositions have been studied before and after testing, paying attention to features such as cavity formation at grain boundaries and grain texture.





## NANOTECHNOLOGY

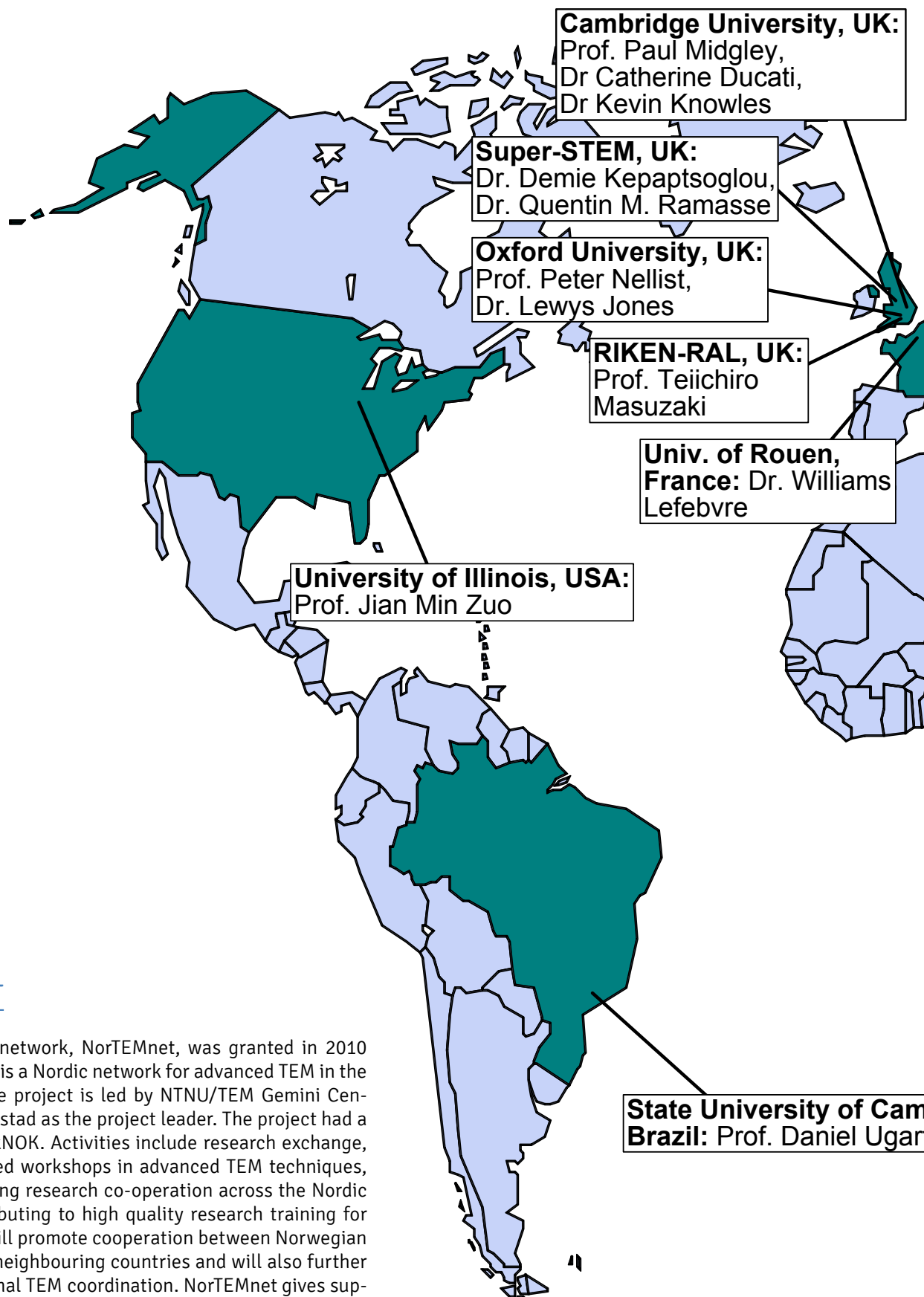
TEM is a crucial tool in the development of Nanotechnology. The TEM Gemini Centre has several small and larger projects related to studying nanoparticles, 1D-nanostructures (i.e. nanowires, carbon nanotubes), thin films and 2D-crystals such as graphene. NTNU NanoLab is an important partner, and now next door neighbour, for the TEM Gemini Centre. It coordinates the education and research of Nanotechnology at NTNU. PhD student Vidar Fauske is hired by NTNU NanoLab and based in the TEM Gemini Centre. In his PhD on the interfaces between nanowires and the substrates they grow from, Fauske can combine advanced TEM techniques with the complementary advanced techniques at NTNU NanoLab. Further, there is a growing interaction with the Nano Network, 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 2014. The Network is also funding PhD student Magnus Nord, who is working in the field of oxide electronics. Magnus is investigating epitaxial complex oxide thin films of  $\text{LaSrMnO}_3$  with advanced TEM, including quantitative EELS. This is a project in collaboration with Prof. Thomas Tybell in the Electronics department. Within the same department we work close together with Profs. Helge Weman and Bjørn-Ove Fimland. Their group is developing optoelectronic devices as solar cells and LEDs based on III-V nanowires. We expect that the TEM work dedicated to Nanotechnology will be growing and one of the new TEMs is dedicated to nanotechnology.



## ACTIVE PROJECTS IN 2014

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

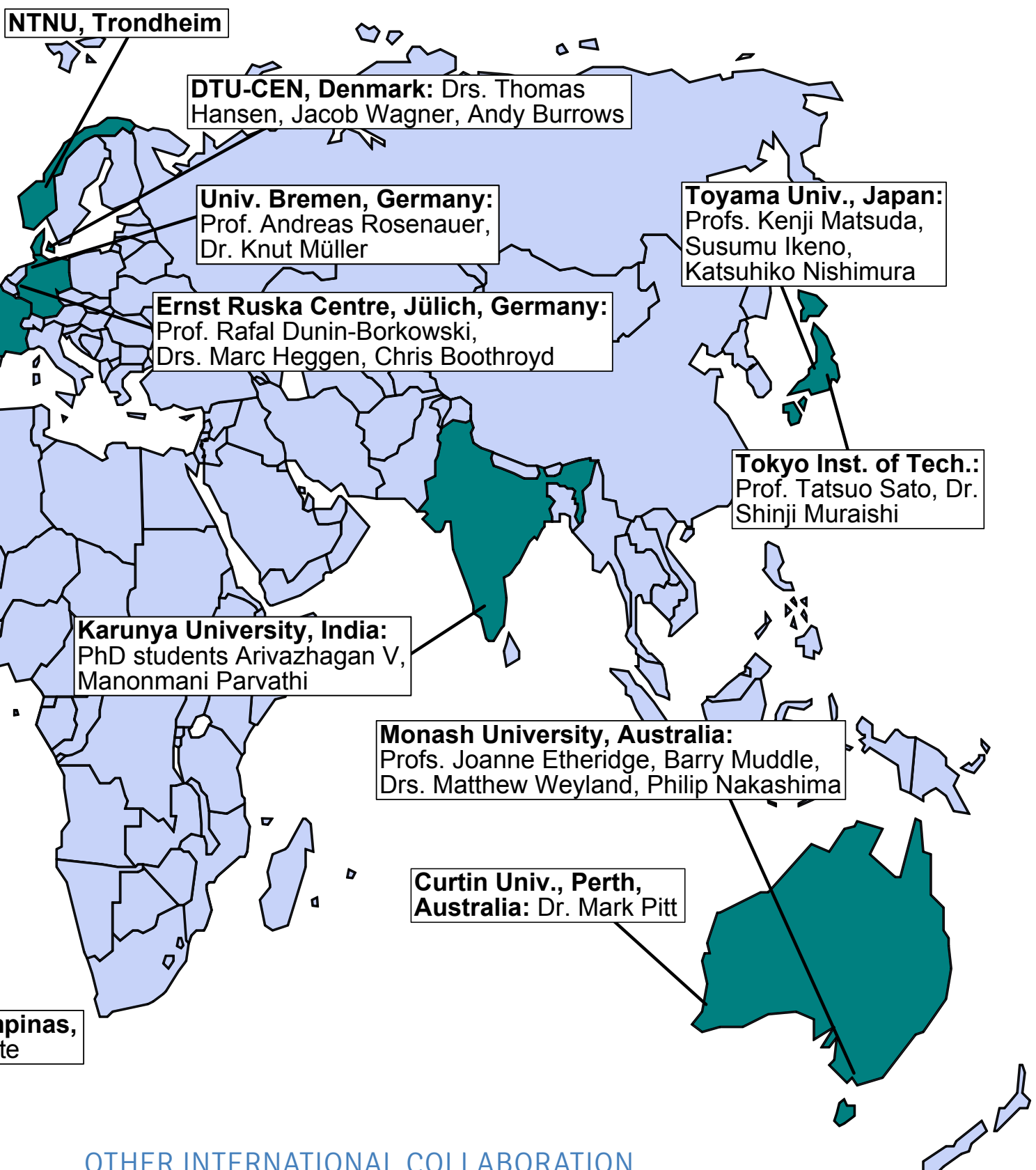
Project type	Project title	Duration	Partners
SFI	<i>inGAP – Metal dusting</i>	2006–2014	NTNU, SINTEF, UiO, Statoil, Haldor Topsøe
RENERGI	<i>Nanosolar – Development of semiconductor nanowire based solar cells</i>	2009–2013	NTNU
KMB	<i>Norwegian–Japanese Al–Mg–Si Alloy Precipitation project</i>	2009–2014	NTNU, SINTEF, Hydro
KMB	<i>MoreAl</i>	2009–2014	SINTEF, NTNU, Hydro, Gränges
Nordic network	<i>NorTEMnet</i>	2009–2014	NTNU, SINTEF
SUP	<i>Improvement (Innovation in light metals processing)</i>	2009–2014	NTNU, SINTEF
FME	<i>Solar United</i>	2009–2017	SINTEF, NTNU, IFE, several industry partners
eVITA	<i>MultiModal- Multiscale modelling in Al</i>	2011–2014	NTNU, SINTEF
Infrastructure	<i>NORTEM</i>	2011–2017	SINTEF, NTNU, UiO
Climit	<i>Pre-memCO2</i>	2012–2014	NTNU, SINTEF
KMB	<i>Co-catalysts for GTL</i>	2012–2014	Statoil, NTNU, SINTEF
FRINATEK	<i>BENTMAT</i>	2012–2015	NTNU, SINTEF
EU	<i>SMARTCat</i>	2012–2015	SINTEF, DTU, CNRS, CEA, Basic Membranes
BIP	<i>Hybrid Dual Junction Silicon Based Solar Cell</i>	2012–2015	Integrated Solar, SINTEF
BIP	<i>RoEx (Al Alloy development for Rolling and Extrusion)</i>	2012–2016	NTNU, SINTEF, Hydro
SEP	<i>Advanced TEM using NORTEM infrastructure</i>	2013–2015	NTNU, SINTEF
Industry	<i>Studies of FT catalysts</i>	2013–2015	SINTEF, SABIC, NTNU
Energix	<i>High Efficiency Quantum dot Intermediate Band Solar Cell</i>	2013–2017	NTNU, SINTEF
IPN	<i>SiNODE</i>	2013–2017	SINTEF, IFE, Elkem
FRINATEK	<i>Fundamental investigations of precipitation in the solid state</i>	2013–2017	NTNU, SINTEF
National Infrastructure	<i>NORTEM seed projects</i>	2014–2016	NTNU, SINTEF, UiO
GASSMAKS	<i>Development of Catalysts and Materials for Compact Steam Reformer</i>	2014–2017	NTNU, SINTEF



## NORTEMNET

The NordForsk TEM network, NorTEMnet, was granted in 2010 and ended in 2014. It is a Nordic network for advanced TEM in the Nordic countries. The project is led by NTNU/TEM Gemini Centre with Randi Holmestad as the project leader. The project had a total budget of 900 kNOK. Activities include research exchange, training and dedicated workshops in advanced TEM techniques, aimed at strengthening research co-operation across the Nordic countries and contributing to high quality research training for PhD students. This will promote cooperation between Norwegian groups and those in neighbouring countries and will also further strengthen the national TEM coordination. NorTEMnet gives support for students to visit other labs within the Nordic countries for doing research and using TEM equipment not available in their home labs. For details on the project, see NorTEMnet webpage (<http://www.ntnu.edu/physics/nortemnet>). The TEM Gemini Centre organised a workshop on advanced STEM in Trondheim in January 2014 (see page 24).





As can be seen in the publication list, the TEM Gemini Centre has several international collaborators. During 2014 we have had direct scientific collaboration/initiated contact with the groups/people illustrated in the figure above. This included several exchange research stays (2 – 6 months). Especially, since the new TEMs came in use we had several groups outside Norway visiting our labs. We thank all our international collaborators for the productive and pleasant interaction!

## STEM WORKSHOP January 2014

As a part of the NorTEMnet project the TEM Gemini Centre organized a STEM workshop in January 2014. with lectures from several high profile researchers in the STEM community - Peter Nellist and Lewys Jones from Oxford, Sara Bals from EMAT Antwerp, Miran C  h from Jozef Stefan Institute Ljubljana and Knut M  ller from University of Bremen. Topics included theory of HAADF image formation, image simulations, technical aspects of STEM at the atomic level, noise reduction by post-processing and STEM tomography. In total 40 people participated in the workshop, including presenters and participants from 8 countries. Labs involving the three NORTEM instruments and advanced computer labs related to post-processing were organized. With the improvement of the microscopes and especially probe correction, resulting in a exception growth in STEM use, this dedicated workshop was well timed and has already proven to contribute in the daily work of many of the participants.

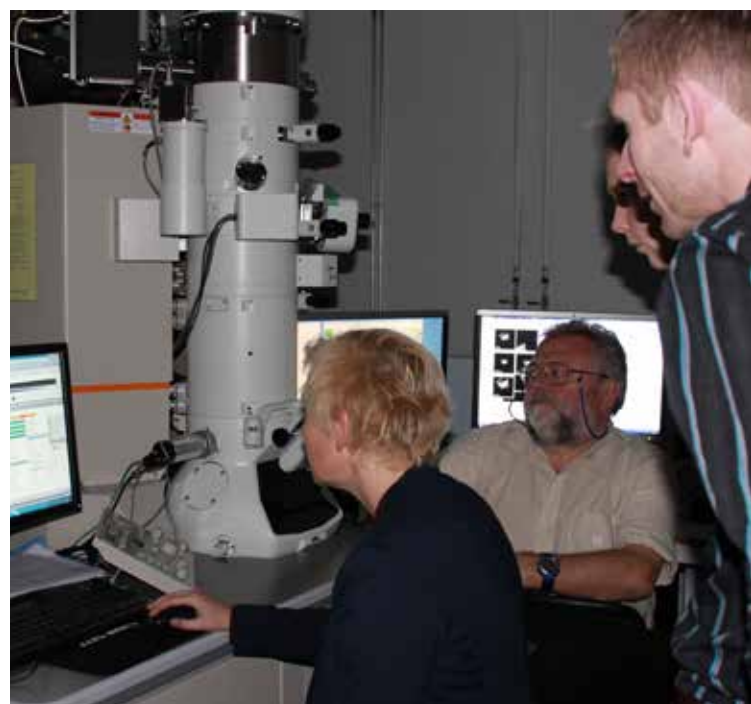


### **Invited talks**

- Pete Nellist, "Introduction to STEM"
- Knut M  ller, "Simulation of STEM images"
- Lewys Jones, "Quantitative analysis of HAADF STEM image data"
- Knut M  ller, "Practice of quantitative STEM"
- Sara Bals, "Electron tomography in materials science"
- Miran C  h, "STEM: Experimental considerations and applications in materials science"

### **Lab sessions**

- R  gis R. Chapuis, HRSTEM with Aberration correction on the ARM
- John Walmsley / Astrid Marie F. Muggerud, STEM tomography on the 2100F
- Antonius T. J. van Helvoort, setting-up STEM, low magnification HAADF-STEM
- Knut M  ller / Lewys Jones, STEM image simulation and post-processing of experimental data





## PHD DISSERTATIONS 2014

Sigurd Wenner defended his PhD thesis on the 29<sup>th</sup> of January. The thesis title was “Transmission electron microscopy and muon spin relaxation studies of precipitation in Al-Mg-Si alloys”. Most of the work was concerned with the role of Cu, Ag and Ca additions to Al-Mg-Si alloys, where TEM was the main investigative technique. Sigurd conducted aberration-corrected EELS elemental mapping on precipitates at the SuperSTEM facility in Daresbury, UK, enabling the distinction between Cu and Ag atomic columns. He also had several visits to the RIKEN-RAL muon facility in Oxfordshire, UK, investigating the distribution of vacancies in Al alloys by muon spin relaxation spectroscopy. The prescribed trial lecture topic was “Topological materials – A new state of matter transforming electronics?”.

Takeshi Saito defended his PhD thesis on the 13<sup>th</sup> of June. The thesis title was “The Effect of Trace Elements on Precipitation in Al-Mg-Si alloys – A Transmission Electron Microscopy Study”. Takeshi investigated alloys with small additions of Cu and Zn, which are present in recycled Al alloys. In addition to metallurgical properties such as corrosion resistance and tensile strength during hardening, the bonding environments of Cu and Zn in precipitates were studied. The candidate spent a few months at the University of Rouen in France for aberration-corrected STEM studies of precipitates, which by aid of simulations and theoretical calculations revealed incorporations of Cu and Zn at specific sites in common hardening Al-Mg-Si phases. The prescribed trial lecture topic was “The opportunities of new large area EDX detectors in electron microscopy”.

Astrid Marie Flattum Muggerud defended her PhD thesis on the 4<sup>th</sup> of September. The thesis title was “Transmission electron microscopy studies of dispersoids and constituent phases in Al-Mn-Fe-Si alloys”. The PhD work was centered around electron diffraction studies of constituent and dispersoid phases, which through comparison with simulations yielded their orientation relationship with the Al matrix. The relationship for dispersoids was explained by their cubic icosahedral quasi crystal approximant structure, with the icosahedrons having a fixed orientation relative to the Al matrix. The morphology of dispersoids was investigated by HAADF-STEM tomography. The prescribed trial lecture topic was “Ptychography; methods and examples”.



*From the left: Opponent Velimir Radmilovic, PhD candidate Sigurd Wenner, administrator Dag W. Breiby, opponent Isao Watanabe, supervisor Randi Holmestad.*



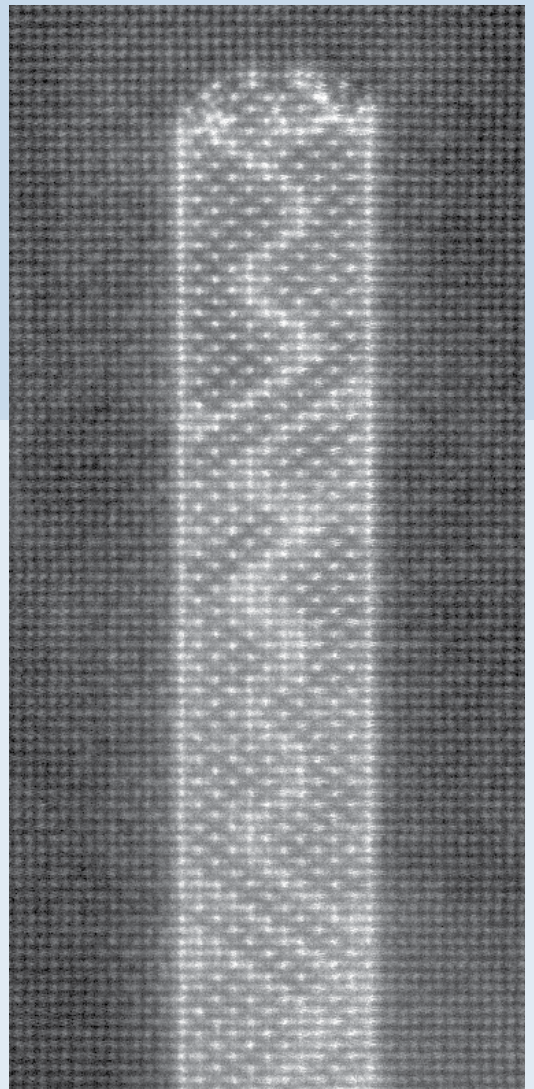
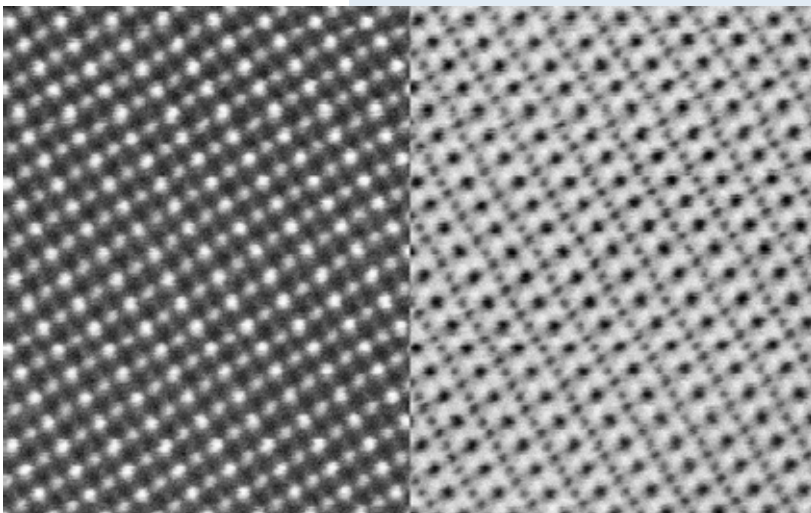
*From the left: Opponent Thierry Epicier, co-supervisor Calin D. Marioara, PhD candidate Takeshi Saito, administrator Øystein Grong, opponent Carmen Schäfer, co-supervisor Knut Martinsen, supervisor Randi Holmestad, co-supervisor Jostein Røyset.*



*From the left: Opponent Olaf Engler, PhD candidate Astrid Marie Flattum Muggerud, opponent Laure Bourgeois, administrator Justin W. Wells.*

# SELECTED SCIENTIFIC PAPERS

FROM 2014





# Titanium uptake and incorporation into silica nanostructures by the diatom *Pinnularia* sp. (Bacillariophyceae)

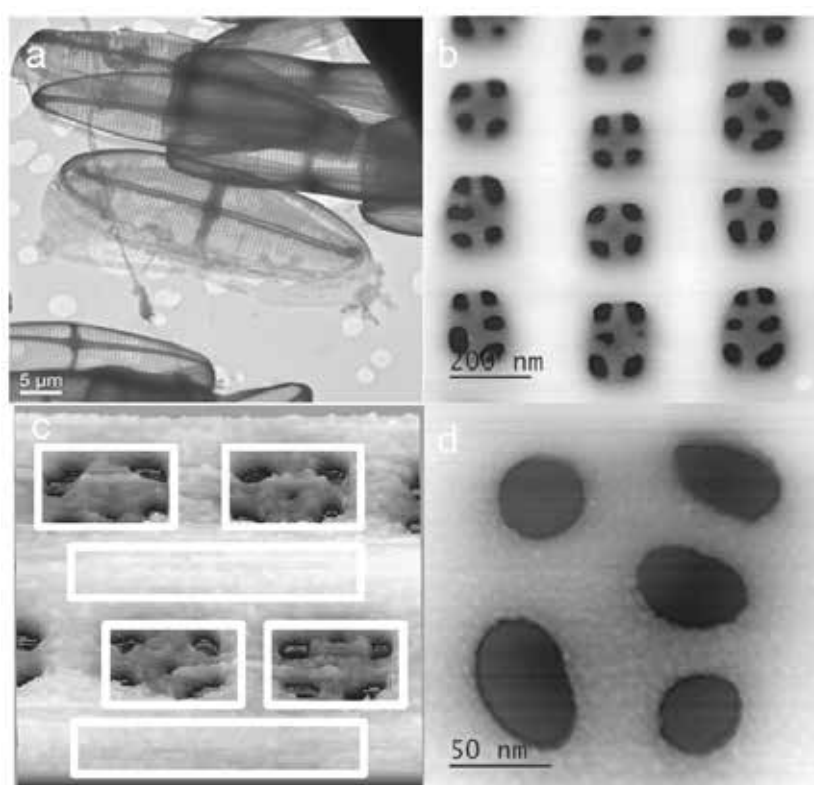
Matilde Skogen Chauton · Lotte M. B. Skolem ·  
Lasse Mork Olsen · Per Erik Vullum · John Walmsley ·  
Olav Vadstein

Received: 3 June 2014 / Revised and accepted: 3 July 2014  
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**Abstract** Diatoms are an ecologically successful group within the phytoplankton, and their special feature is a biofabricated silica cell encasement called a frustule. These frustules attract interest in material technology, and one potential application is to use them in solar cell technology. The silica frustule with its nanoscaled pattern is interesting per se, but the utility is enhanced if we succeed in incorporating other elements. Titanium is an interesting element because its oxide is a semi-conductor with a high band gap. However, doping with relevant elements through bioincorporation is challenging, and it is necessary to understand the biology involved in element uptake and incorporation. Here we present data on

bioincorporation of Ti into the silica frustules of the pennate diatom *Pinnularia* sp. (Ehrenberg) and show that the distribution of the incorporated Ti is inhomogeneous both between and within valves. More than a tenfold increase of Ti in newly synthesised valves was achieved, and increased Ti around the pores was confirmed by both EDS and EELS analyses. HAADF STEM spectroscopy revealed a grainy surface with amorphous silica particles of 4 to 5 nm in size. These observations are explained by what is known from the physico-chemical processes involved in biosilification and frustule formation, looking into it from a biological point of view.

**Fig. 2** **a** Low-resolution, bright-field TEM image of cleaned *Pinnularia* frustules from one of the Ti uptake experiments. **b** Section of a *Pinnularia* sp. valve with pores. **c** High annular dark field (HAADF) scanning transmission electron spectroscopy (STEM) image of an area which includes four pore openings and surrounding SiO<sub>2</sub>. The boxes indicate where EDS/EELS measurements were performed, and in the four pore areas the average Ti was 1.06 ( $\pm 0.101$ ) at.%. In the two areas between the pores, the Ti was 0.21 ( $\pm 0.050$ ) at.%. **d** High annular dark field (HAADF) scanning transmission electron spectroscopy (STEM) image of one of the pores showing the granular appearance of the amorphous SiO<sub>2</sub>



# Phase stabilization principle and precipitate-host lattice influences for Al–Mg–Si–Cu alloy precipitates

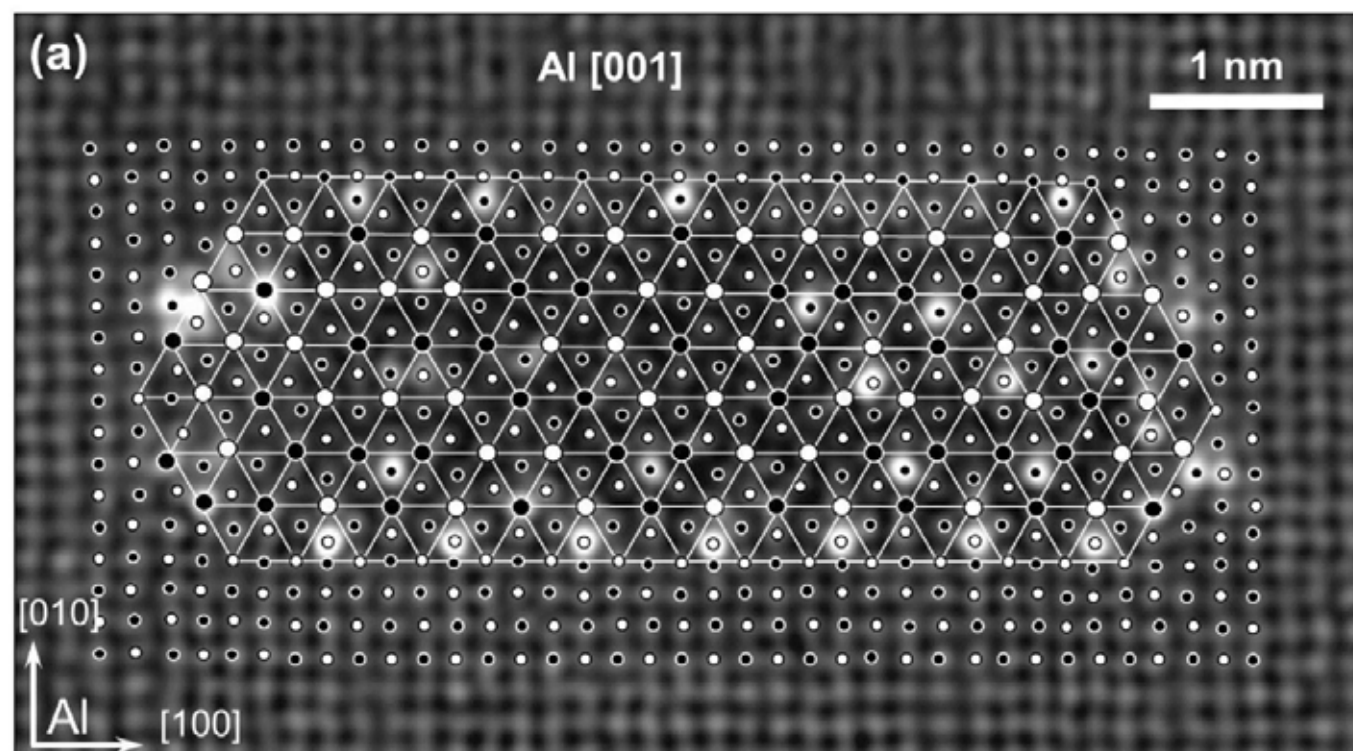
F. J. H. Ehlers · S. Wenner · S. J. Andersen ·  
 C. D. Marioara · W. Lefebvre · C. B. Boothroyd ·  
 R. Holmestad

Received: 21 February 2014 / Accepted: 28 May 2014 / Published online: 17 June 2014  
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**Abstract** In this work, we seek to elucidate a common stabilization principle for the metastable and equilibrium phases of the Al–Mg–Si–Cu alloy system, through combined experimental and theoretical studies. We examine the structurally known well-ordered Al–Mg–Si–Cu alloy metastable precipitates along with experimentally observed disordered phases, using high angle annular dark field scanning transmission electron microscopy. A small set of local geometries is found to fully explain all structures. Density functional theory based calculations have been carried out on a larger set of structures, all fully constructed by the same local geometries. The results reveal that experimentally reported and hypothetical Cu-free phases from the set are practically indistinguishable with regard to formation enthalpy and composition. This strongly supports a connection of the geometries with a bulk phase stabilization principle. We relate our findings to the Si

network substructure commonly observed in all Mg–Al–Si(–Cu) metastable precipitates, showing how this structure can be regarded as a direct consequence of the local geometries. Further, our proposed phase stabilization principle clearly rests on the importance of metal–Si interactions. Close links to the Al–Mg–Si precipitation sequence are proposed.

**Fig. 5 a** The L precipitate from Fig. 1c, with all atom heights (including those of adjacent host lattice columns) highlighted. Atoms within the precipitate are distinguished in a manner corresponding Figs. 2 and 3, with the network atom grid terminating at the approximate boundary to the host lattice. For the host lattice atoms, the non-network atom nomenclature has been used.





# Rapid Estimation of Catalyst Nanoparticle Morphology and Atomic Coordination by High-Resolution Z-Contrast Electron Microscopy

Lewys Jones,<sup>\*,†</sup> Katherine E. MacArthur,<sup>†</sup> Vidar T. Fauske,<sup>‡</sup> Antonius T. J. van Helvoort,<sup>‡</sup> and Peter D. Nellist<sup>†</sup>

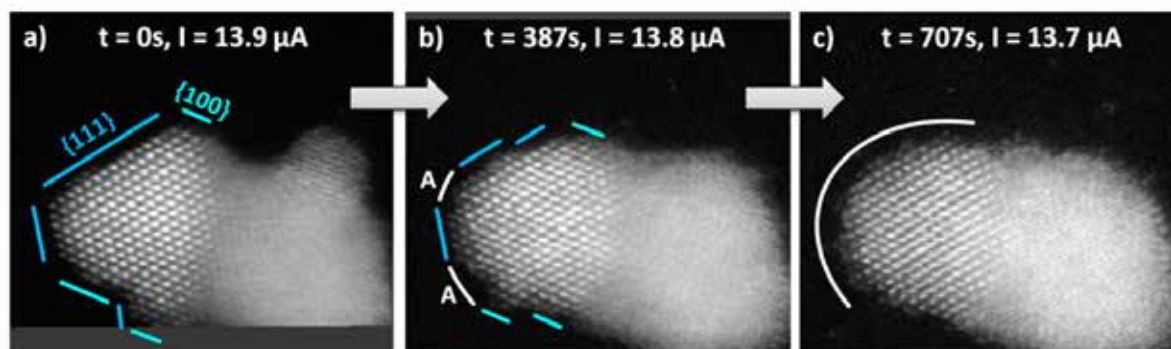
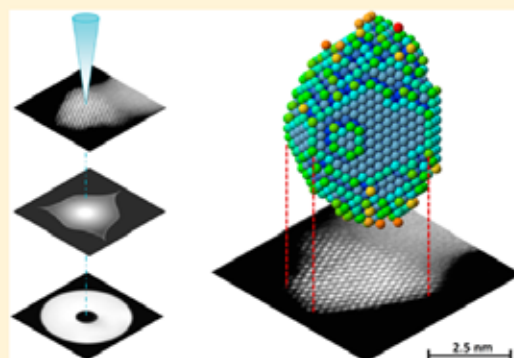
<sup>†</sup>Department of Materials, University of Oxford, OX13PH Oxford, United Kingdom

<sup>‡</sup>Department of Physics, Norwegian University of Science and Technology (NTNU), NO-7491 Trondheim, Norway

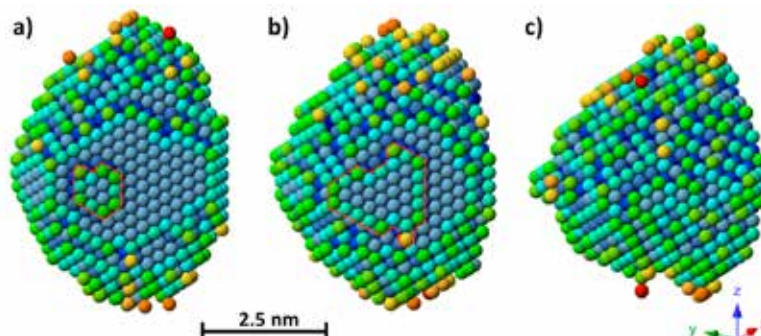
**S** Supporting Information

**ABSTRACT:** Heterogeneous nanoparticle catalyst development relies on an understanding of their structure–property relationships, ideally at atomic resolution and in three-dimensions. Current transmission electron microscopy techniques such as discrete tomography can provide this but require multiple images of each nanoparticle and are incompatible with samples that change under electron irradiation or with surveying large numbers of particles to gain significant statistics. Here, we make use of recent advances in quantitative dark-field scanning transmission electron microscopy to count the number atoms in each atomic column of a single image from a platinum nanoparticle. These atom-counts, along with the prior knowledge of the face-centered cubic geometry, are used to create atomistic models. An energy minimization is then used to relax the nanoparticle's 3D structure. This rapid approach enables high-throughput statistical studies or the analysis of dynamic processes such as facet-restructuring or particle damage.

**KEYWORDS:** catalysis, nanometrology, aberration corrected STEM, quantitative ADF, platinum nanoparticles



**Figure 2.** Selected frames from the (a) start, (b) middle, and (c) end of the time-series data. Frame times and indicated emission currents are as shown. Line annotations indicate the terminating facets where visible. As the series progresses further, parts that indicate the amorphous-type sections of the perimeter are labeled “A”. Field of view is  $\approx 10$  nm.



**Figure 4.** Energetically relaxed models corresponding to the frames shown in Figure 2. The coloring of the Pt atoms indicates the determined nearest-neighbor coordination, from 1 in red to 12 in dark blue. In the orientation shown here, the {111} facet (labeled with the red dashed line) facing front-right corresponds to the one labeled in the upper left of Figure 2a, and the unresolved grain would be behind these 3D models. The sputtering (mass loss) and rounding resulting from increasing beam heating/damage can be seen from left to right.

## Near-surface depletion of antimony during the growth of GaAsSb and GaAs/GaAsSb nanowires

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<sup>1</sup>Department of Physics, Norwegian University of Science and Technology (NTNU), Trondheim, Norway

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The near-surface reduction of the Sb mole fraction during the growth of GaAsSb nanowires (NWs) and GaAs NWs with GaAsSb inserts has been studied using quantitative high-angle annular dark field scanning transmission electron microscopy (STEM). A model for diffusion of Sb in the hexagonal NWs was developed and employed in combination with the quantitative STEM analysis. GaAsSb NWs grown by Ga-assisted molecular beam epitaxy (MBE) and GaAs/GaAsSb NWs grown by Ga- and Au-assisted MBE were investigated. At the high temperatures employed in the NW growth, As-Sb exchange at and outward diffusion of Sb towards the surface take place, resulting in reduction of the Sb concentration at and near the surface in the GaAsSb NWs and the GaAsSb inserts. In GaAsSb NWs, an increasing near-surface depletion of Sb was observed towards the bottom of the NW due to longer exposure to the As beam flux. In GaAsSb inserts, an increasing change in the Sb concentration profile was observed with increasing post-insert axial GaAs growth time, resulting from a combined effect of radial GaAs overgrowth and diffusion of Sb. The effect of growth temperature on the diffusion of Sb in the GaAsSb inserts was identified. The consequences of these findings for growth optimization and the optoelectronic properties of GaAsSb are discussed. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4896904>]

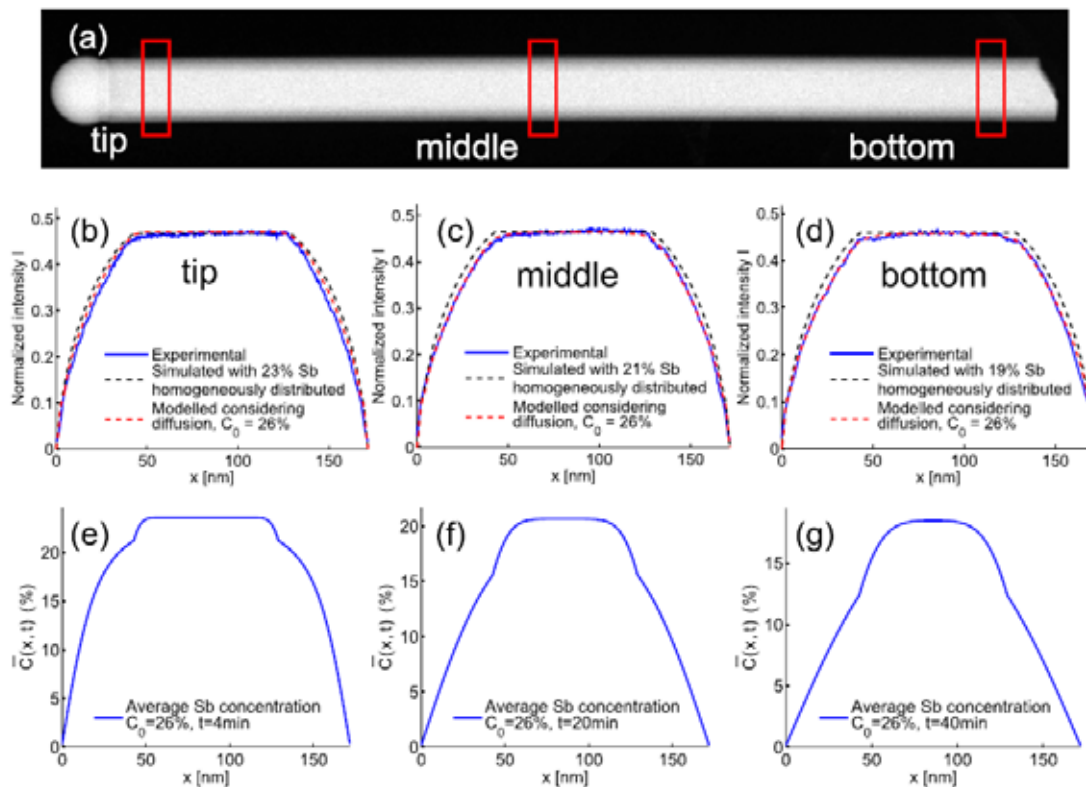
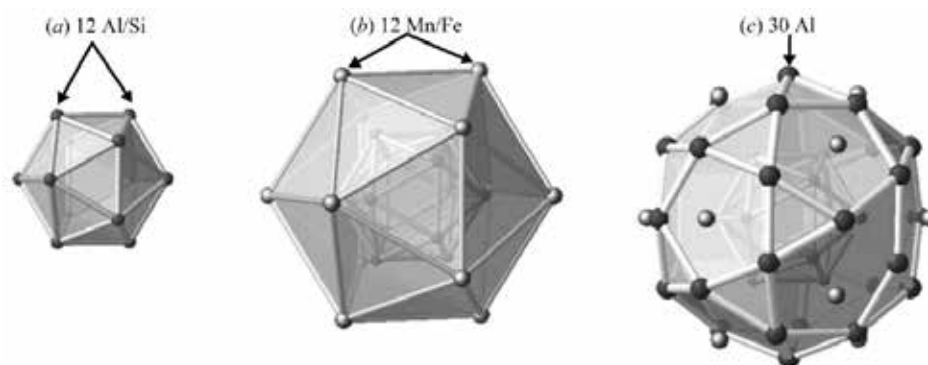


FIG. 4. (a) HAADF STEM image of a GaAsSb NW (batch A) and experimental, simulated, and modelled HAADF STEM intensity profiles across the NW at the different areas indicated in (a): (b) tip, (c) middle, and (d) bottom. The experimental intensity profiles were not taken from (a), but from higher magnification images taken from the respective regions. (e)–(f) The corresponding profiles for the average Sb concentration,  $C_{\text{shell}}(x, t)$ , according to Eqs. (3) and (4), employed in constructing the modelled intensity profiles (red dashed lines in (b)–(d)).

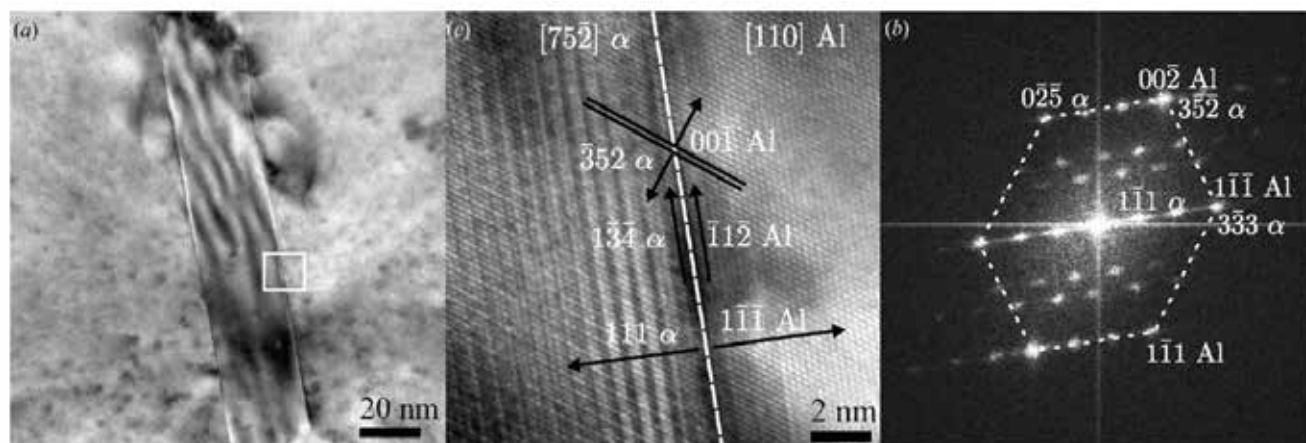


Mackay icosahedron explaining orientation  
relationship of dispersoids in aluminium alloysAstrid Marie F. Muggerud,<sup>a\*</sup>  
Yanjun Li,<sup>b</sup> Randi Holmestad<sup>a</sup>  
and Sigmund J. Andersen<sup>c</sup><sup>a</sup>Department of Physics, NTNU, N-7491  
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astrid.muggerud@ntnu.noReceived 24 June 2014  
Accepted 2 August 2014

The orientation relations (ORs) of the cubic icosahedral quasicrystal approximant phase  $\alpha$ -Al(Fe,Mn)Si have been studied after low temperature annealing of a 3xxx wrought aluminium alloy by transmission electron microscopy. From diffraction studies it was verified that the most commonly observed OR for the  $\alpha$ -Al(Fe,Mn)Si dispersoids is  $[1\bar{1}1]\alpha // [1\bar{1}1]\text{Al}$ ,  $(5\bar{2}\bar{7})\alpha // (011)\text{Al}$ . This orientation could be explained by assuming that the internal Mackay icosahedron (MI) in the  $\alpha$ -phase has a fixed orientation in relation to Al, similar to that of the icosahedral quasi-crystals existing in this alloy system. It is shown that mirroring of the normal-to-high-symmetry icosahedral directions of the MI explains the alternative orientations, which are therefore likely to be caused by twinning of the fixed MI. Only one exception was found, which was related to the Bergman icosahedron internal to the T-phase of the Al-Mg-Zn system.

**Figure 1**

The three-layered structure of the 54-atom MI in the  $\alpha$ -phase viewed along a threefold axis, coinciding with a  $\langle 111 \rangle \alpha$ -direction. The MI contains three concentric shells: (a) the inner icosahedron of 12 Al/Si atoms; (b) the surrounding icosahedron consisting of 12 Mn/Fe atoms; (c) Al atoms outside each of the 30 twofold edges of the Mn/Fe icosahedron constitute an icosidodecahedron. Similar figures can be found in Jarić & Gratias (1989).

**Figure 4**

(a) Plate-like  $\alpha$ -dispersoid viewed edge on. (b) Higher resolution of the area marked with a square in (a). The directions along and perpendicular to the habit plane trace are shown. (c) FFT of the micrograph in (b), indicating OR  $[752]\alpha // [110]\text{Al}$ ,  $(1\bar{1}1)\alpha // (1\bar{1}1)\text{Al}$ .



# HAADF-STEM and DFT investigations of the Zn-containing $\beta''$ phase in Al–Mg–Si alloys

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David Hernandez-Maldonado<sup>b</sup>, Ruben Bjørge<sup>a,1</sup>, Calin D. Marioara<sup>c</sup>,  
Sigmund J. Andersen<sup>c</sup>, Randi Holmestad<sup>a</sup>

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Received 13 May 2014; accepted 23 June 2014

Available online 24 July 2014

## Abstract

The Zn-containing  $\beta''$  phase in Al–Mg–Si alloys was investigated by aberration corrected high angle annular dark-field scanning transmission electron microscopy (HAADF-STEM), combined with density functional theory (DFT) calculations. The mean intensity of one Si site of the  $\beta''$  phase is higher than that of the other Si sites, suggesting partial Zn occupancy. DFT studies support that this Si site is competitive for Zn incorporation. While HAADF-STEM image simulations show an influence of the Zn distribution along the  $\beta''$  main growth direction, total energy calculations predict a weak Zn–Zn interaction. This suggests that Zn atoms are not clustering, but uniformly distributed along the atomic columns. The Zn incorporation has a weak influence on the  $\beta''$  phase, where Zn is admitted as a “defect” according to the DFT studies.

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**Keywords:** Aluminium alloys; Precipitate; High angle annular dark-field scanning transmission electron microscopy; Density functional theory; Atomic structure

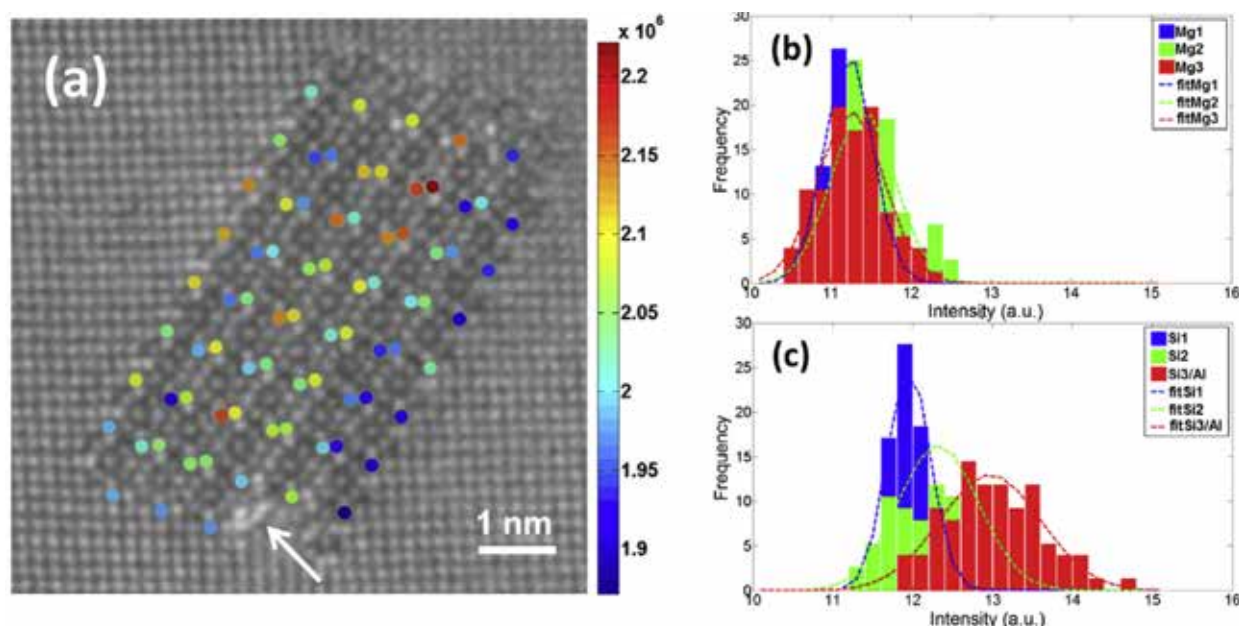


Fig. 4. (a) Colour mapping of the integrated intensity on the Si3/Al sites of the Zn-containing  $\beta''$  precipitate. The HAADF-STEM intensity is displayed in arbitrary units. In (b) and (c), the respective intensity distributions for Mg and Si sites in the precipitate are shown. The columns at the bottom part of the precipitate had non-systematically higher intensity (shown by white arrow in (a)) and were excluded in the statistics of intensity distributions. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

# Atomic-resolution electron energy loss studies of precipitates in an Al–Mg–Si–Cu–Ag alloy

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Despoina-Maria Kepaptsoglou,<sup>c</sup> Fredrik S. Hage<sup>c</sup> and Randi Holmestad<sup>a</sup>

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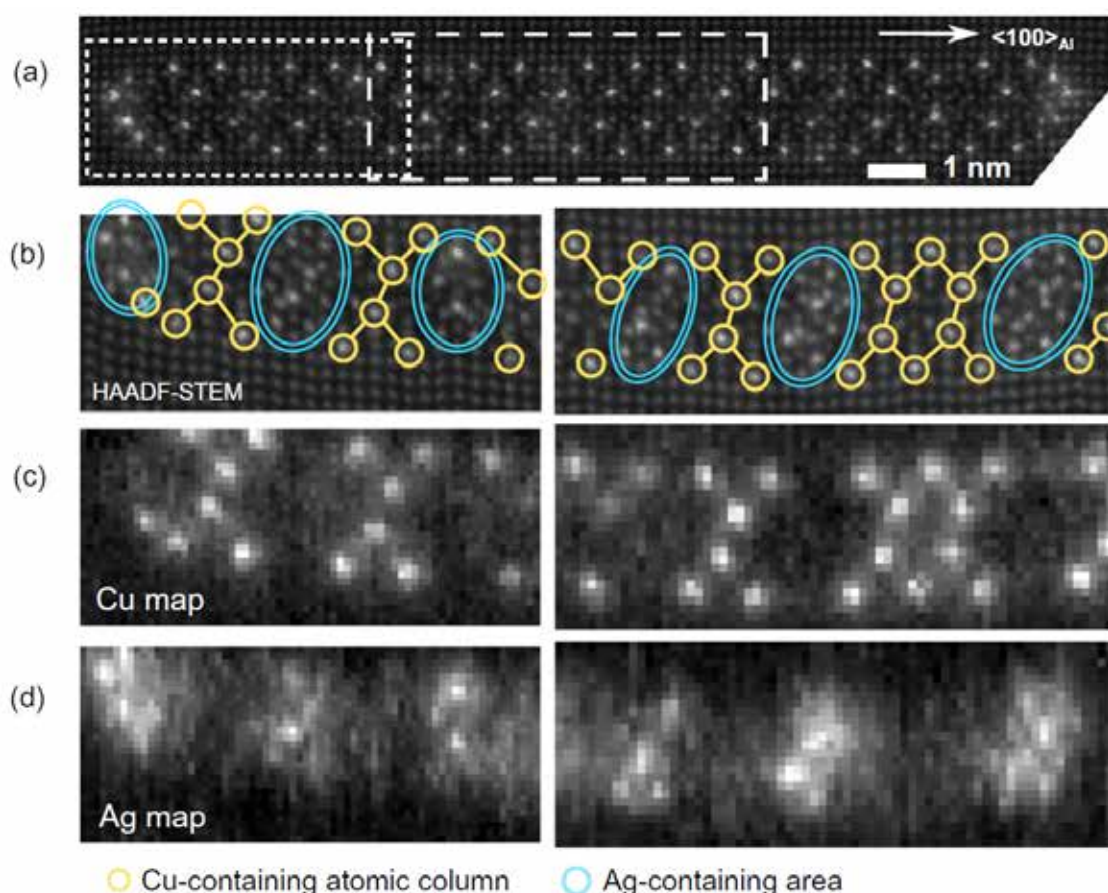
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Aberration-corrected scanning transmission electron microscopy combined with electron energy loss spectroscopy has been used to determine the distribution of Cu and Ag atomic columns of precipitates in an Al–Mg–Si–Cu–Ag alloy. Cu columns were commonly part of C and Q' phases, with the atomic columns having large projected separations. Columns containing Ag were more tightly spaced, in areas lacking repeating unit cells and at incoherent precipitate–host lattice interfaces. Cu-rich and Ag-rich areas were not found to intermix.

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**Keywords:** Aluminum alloys; Precipitation; Scanning transmission electron microscopy; Electron energy loss spectroscopy

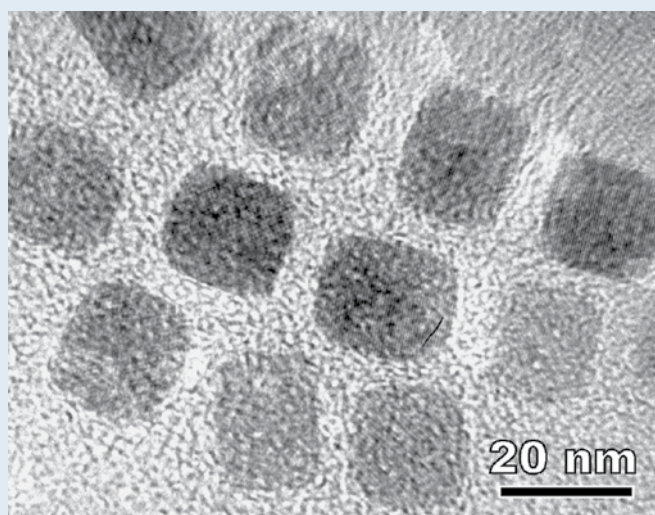
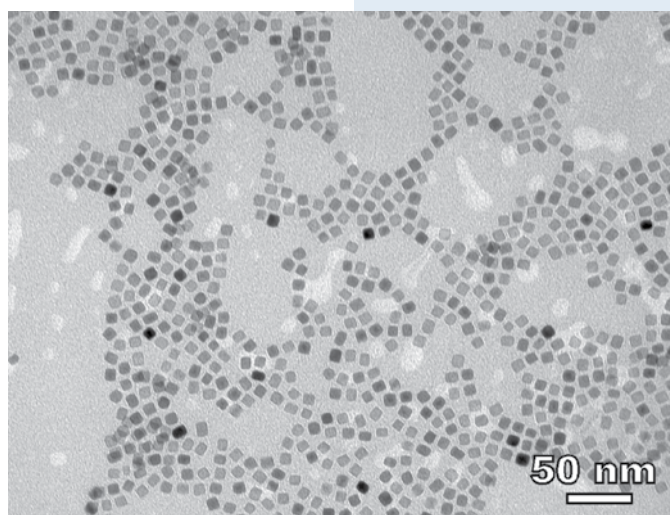
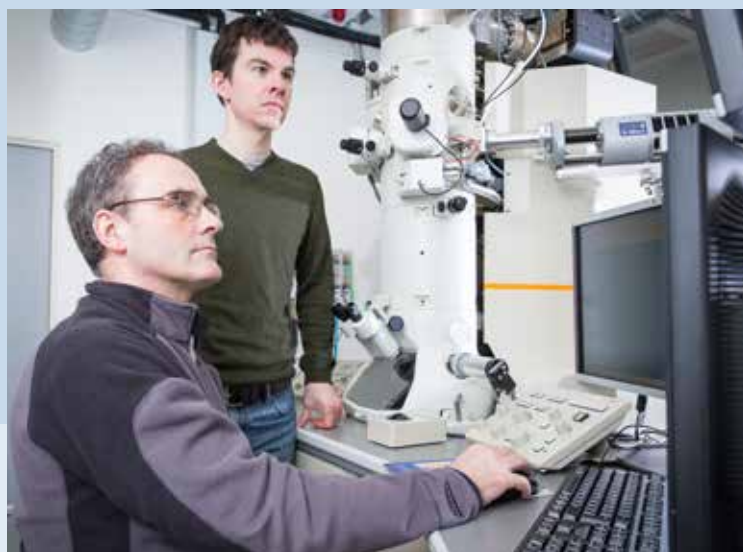
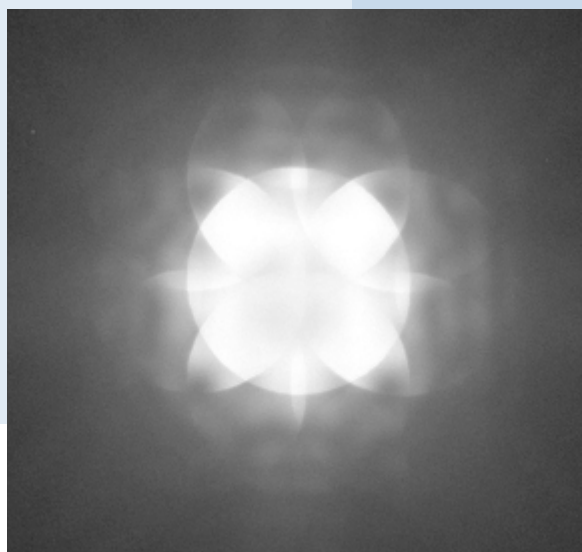


**Figure 3.** (a and b) Raw HAADF-STEM images of a precipitate cross-section, taken respectively before and during the STEM-EELS acquisition. (c and d) EELS elemental maps of Cu and Ag from the two acquisition areas marked in (a). The skew in the images is caused by specimen drift. The location of Cu atomic columns and areas rich in Ag are marked in (b). Cu columns in a C configuration are connected by lines.



# PUBLICATIONS 2014

PEOPLE IN TEM GEMINI CENTRE ARE HIGHLIGHTED





## Journal publications

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### Conference talks/posters/seminars/meetings (presenter underlined)

P. I. Dahl, M. S. Thomassen, L. C. Colmenares, A. O. Barnett, S. Lomas, P. E. **Vullum**, H. Meli and T. Mokkelbost, “Flame Spray Pyrolysis of Electrode Materials for Energy Applications”, *MRS 2014 Fall Meeting*, Boston, U.S.A., 30 November - 5 December 2014. [Talk]

Q. Du, C. D. **Marioara**, J. **Friis**, E. Osmundsen and B. Holmedal, “Modelling precipitation of non-spherical and metastable particles in multicomponent aluminium alloys”, *14<sup>th</sup> International Conference on Aluminium Alloys (ICAA14)*, Trondheim, Norway, 15-19 June 2014. [Poster]

M. **Erlbeck**, V. T. **Fauske** and A. T. J. **van Helvoort**, “Graphene and nanowires characterization challenges - probing the electronic characteristics of nanowires”, *Webinar Imina Technologies*, [Internet], 24 September 2014. [Webinar]

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 nanostructures in a FIB”, *Nanolab 10<sup>th</sup> Anniversary*, Trondheim, Norway, 10-11 November 2014. [Talk]

J. **Friis**, O. M. Løvvik, J. C. **Walmsley** and O. Lunder, “Ab initio modelling of tin and lead segregation at the oxide-metal interface in aluminium”, *14<sup>th</sup> International Conference on Aluminium Alloys (ICAA14)*, Trondheim, Norway, 15-19 June 2014. [Poster]

Y. **Gan**, J. C. **Walmsley**, R. **Holmestad**, T. Peters and M. S. Stange, “TEM characterization of H<sub>2</sub>-selective Pd-based membranes”, *18<sup>th</sup> International Microscopy Congress (IMC)*, Prague, Czech Republic, 7-12 September 2014. [Talk]

I. Hallsteinsen, E. Folven, J. Grepstad, R. **Holmestad**, M. K. **Nord**, T. Tybell, P. E. **Vullum**, F. K. Olsen, Y. Takamura and E. Christiansen, “Magnetic properties and exchange mechanisms in epitaxial (111)-oriented magnetic heterostructures”, *Material Research Society - Spring meeting 2014*, Tønsberg, Norway, 21-25 April 2014. [Talk]

A. T. J. **van Helvoort**, “III-V semiconductor nanowires: from synthesis to novel devices”, *Disputas og foredrag ved Høgskolen i Buskerud Vestfold i Vestfold Innovation Park*, Horten, Norway, 30 April 2014. [Talk]

A. T. J. **van Helvoort**, “Probing electronic properties of nanowires”, *Webinar Imina Technologies* [Internet], 24 September 2014. [Webinar]

R. **Holmestad**, “HAADF-STEM studies of Ag-, Cu- and Zn-containing precipitates in Al-Mg-Si alloys”, *18<sup>th</sup> International Microscopy Congress (IMC)*, Prague, Czech Republic, 7-12 September 2014. [Talk]

J. Huh, H. Yun, D. C. Kim, A. M. Munshi, D. L. Dheeraj, H. **Kauko**, A. T. J. **van Helvoort**, S. W. Lee, B. O. Fimland and H. Weman, “GaAsSb nanowire diodes based on self-induced compositional variation”, *8<sup>th</sup> Nanowire Growth Workshop (Nanowire2014)*, Eindhoven, Netherlands, 23-27 August 2014. [Talk]

L. Jones, V. T. **Fauske**, K. E. MacArthur, A. T. J. **van Helvoort** and P. D. Nellist, “Visualising the Three-dimensional Morphology and Surface Structure of Metallic Nanoparticles at Atomic Resolution by Automated HAADF STEM Atom Counting”, *Microscopy & Microanalysis 2014*, Hartford, USA, 2-8 August 2014. [Talk]

H. **Kauko**, T. Grieb, A. M. Munshi, K. Müller, A. Rosenauer, B. O. Fimland and A. T. J. **van Helvoort**, “The Outward Diffusion of Sb during Nanowire Growth Studied by Quantitative High-Angle Annular Dark Field Scanning Transmission Electron Microscopy”, *Microscopy & Microanalysis 2014*, Hartford, US, 4-7 August 2014. [Poster]

H. **Kauko**, T. Grieb, A. M. Munshi, K. Müller, H. Weman, A. Rosenauer, B.-O. Fimland and A. T. J. **van Helvoort**, “The outward diffusion of Sb during growth of antimonide III-V nanowires”, *8<sup>th</sup> Nanowire Growth Workshop (Nanowire2014)*, Eindhoven, Netherlands, 23-27 August 2014. [Poster]

E. Kuznetsova and J. C. **Walmsley**, “Opaline silica as a weathering product in the cold climate”, *4<sup>th</sup> European Conference on Permafrost*, 17-21 June 2014. [Talk]



E. A. **Mørtzell**, C. D. **Marioara**, J. Røyset and R. **Holmestad**, “The Effect of Cu, Ag and Ge on Strength and Precipitation in a Lean 6xxx Alloy”, *18<sup>th</sup> International Microscopy Congress (IMC)*, Prague, Czech Republic, 7-12 September 2014. [Talk]

A. M. **Munshi**, D. Dasa Lakshmi Narayana, V. T. **Fauske**, J. F. Reinertsen, L. Ahtapodov, K. D. Lee, B. Heidari, A. T. J. **van Helvoort**, B.-O. Fimland and H. Weman, “Position controlled GaAs nanowires on 2-inch Si wafers using nano-imprint lithography”, *MRS Spring Meeting*, San Francisco, U.S.A., 21-25 April 2014. [Talk]

A. M. **Munshi**, D. Dasa Lakshmi Narayana, V. T. **Fauske**, D. Ren, A. T. J. **van Helvoort**, B.-O. Fimland and H. Weman, “Limitations of self-catalytic growth in controlling the diameter of GaAs nanowires on Si substrates”, *8<sup>th</sup> Nanowire Growth Workshop (Nanowire2014)*, Eindhoven, Netherlands, 25-29 August 2014. [Talk]

A. M. **Munshi**, D. Dasa Lakshmi Narayana, V. T. **Fauske**, D. Ren, A. T. J. **van Helvoort**, B.-O. Fimland and H. Weman, “Limitations of self-catalytic growth in controlling the diameter of GaAs nanowires on Si substrates”, *18<sup>th</sup> International Conference on Molecular Beam Epitaxy (MBE2014)*, Flagstaff, U.S.A., 7-12 September 2014. [Talk]

K. **Nishimura**, K. Matsuda, R. Komaki, N. Nunomura, S. **Wenner**, R. **Holmestad** and T. Matsusaki, “Solute-Vacancy Clustering in Al-Mg-Si Alloys Studied by Muon Spin Relaxation Technique”, *9<sup>th</sup> International Conference on the Physical Properties and Application of Advanced Materials (ICPMAT2014)*, Krakow, Poland, 14-18 September 2014. [Talk]

M. K. **Nord**, J. E. Boschker, M. Moreau, P. E. **Vullum**, S. M. Selbach, T. Tybell and R. **Holmestad**, “Structural and electronic characterization of altered structure at the  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3/\text{SrTiO}_3$  heteroepitaxial interface”, *2014 MRS Spring Meeting & Exhibit*, Tønsberg, Norway, 21-25 April 2014. [Talk]

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D. S. G. **Panditha Vidana**, J. C. **Walmsley** and H. J. Venvik, “An experimental investigation of carbon formation phenomena and initial stage of metal dusting corrosion”, *The Nordic Surface Science and Catalysis meeting (NORD-FORSK)*, Tromsø, Norway, 3 June 2014. [Talk]

D. S. G. **Panditha Vidana**, J. C. **Walmsley** and H. J. Venvik, “The significance of catalysis to the initiation of metal dusting corrosion”, *16<sup>th</sup> Nordic Symposium on Catalysis*, Oslo, Norway, 15-17 June 2014. [Talk]

N. **Tsakoumis**, R. **Dehghan**, A. Voronov, J. C. **Walmsley**, Ø. Borg, E. Rytter, D. Chen, M. Rønning and A. Holmen, “A combined in situ XAS-XRPF-Raman study of Fischer-Tropsch synthesis over a carbon supported cobalt catalyst”, *6<sup>th</sup> International Symposium on Carbon for Catalysis (CARBOCAT-VI)*, Trondheim, Norway, 22-25 June 2014. [Talk]

E. **Undheim**, M. **Vatanparast**, P. E. **Vullum**, T. W. Reenaas and R. **Holmestad**, “TEM studies of quantum dots for intermediate band solar cells”, *Annual Conference of the Nordic Microscopy Society (SCANDEM)*, Linköping, Sweden, 10-14 June 2014. [Poster]

J. C. **Walmsley**, P. E. **Vullum** and R. **Holmestad**, “Using (S) TEM Techniques to Study Energy Related Materials at the Nanoscale”, *Microscopy and Microanalysis 2014*, Hartford, U.S.A., 2-8 August 2014. [Invited talk]

S. **Wenner**, C. D. **Marioara** and R. **Holmestad**, “Precipitation in Al-Cu-Mg(-Zn) Alloys”, *9<sup>th</sup> International Conference on the Physical Properties and Application of Advanced Materials (ICPMAT2014)*, Krakow, Poland, 14-18 September 2014. [Poster]

Y. **Yu**, M. Liu, J. Hjelen and H. J. Roven, “TEM microstructure characterizations of Al-Mg-Si aluminum alloy processed by severe plastic deformation”, *International Forum on Systems and Mechanics (IFSM2014)*, Tainan, Taiwan, 2014. [Poster]

J. **Zhu**, A. H. Lillebø, Y. Zhu, Y. **Yu**, A. Holmen and D. Chen, “Compact reactor for Fischer-Tropsch synthesis based on hierarchically structured Co catalysts: towards better stability”, *6<sup>th</sup> International Symposium on Carbon for Catalysis (CARBOCAT-VI)*, Trondheim, Norway, 22-25 June 2014. [Talk]

## Proceedings from the 14<sup>th</sup> International Conference on Aluminium Alloys 2014: ICAA14, 15-18 June 2014, Trondheim, Norway

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M. Liu, C. D. **Marioara**, R. **Holmestad** and J. Banhart, "Ageing characteristics of Al-Mg-(Ge,Si)-Cu alloys", *Materials Science Forum*, **794-796**, 971-976, 2014.

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A. M. F. **Muggerud**, Y. J. **Li** and R. **Holmestad**, "Orientation studies of alpha-Al(Fe,Mn)Si dispersoids in 6xxx Al alloys", *Materials Science Forum*, **794-796**, 39-44, 2014.

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## PhD theses

A. M. F. **Muggerud**, "Transmission electron microscopy studies of dispersoids and constituent phases in Al-Mn-Fe-Si alloys"

T. **Saito**, "The effect of trace elements on precipitation in Al-Mg-Si alloys - A transmission electron microscopy study"

S. **Wenner**, "Transmission Electron Microscopy and Muon Spin Relaxation Studies of Precipitation in Al-Mg-Si Alloys"

## Master projects/theses

Ø. U. **Berntsen**, "Investigation of Co<sub>2</sub>AlO<sub>4</sub>/CeO<sub>2</sub> Catalyst for N<sub>2</sub>O Abatement using Electron Microscopy Techniques" (Master thesis).

M. **Erlbeck**, "Probing the electronic properties of p-doped gallium arsenide nanowires" (Master thesis).

H. **Grydeland**, "Characterization of Bioaerosols using Electron Microscopy with Special Emphasis on Airborne Bacteria" (Master thesis).

J. **Larsen**, "TEM of Cr doped ZnS for intermediate band solar cells" (Project).

A. B. **Mosberg**, "Examining the effect of shell growth temperature on structural and optical properties in self-catalyzed GaAs/AlGaAs core-shell nanowires using correlated PL-TEM" (Project).

J. S. **Nilsen**, "Position controlled growth of GaAs/AlGaAs core-shell nanowires - more uniform structural and optical properties?" (Master thesis).

E. **Seim**, "TEM characterization of Cr-doped ZnS Thin Films for Solar Cell applications" (Master thesis).

E. **Undheim**, "Transmission electron microscopy characterization of quantum dot based intermediate band solar cells" (Master thesis).

## Popular science contributions/Media

"Down to the atom, alloys by design", *International Innovations*, **160**, 74-76, November 2014.

"Dette skrapet er gull verdt for industrien", *Teknisk Ukeblad*, 15 August 2014.

"Se et elektronmikroskop i aksjon", guided tour at *Researchers' Night NTNU 2014*, 26 September 2014.

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