ANNUAL REPORT 2013

TEM GEMINI CENTRE

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

CONTENT

Graphic Design: Ingebjørg Fyrileiv Guldvik
Photo: Ole Morten Melgård
TEM images: scientists at TEM GEMINI Centre

THE GEMINI CENTRE CONCEPT

The Gemini Centres form a model for strategic cooperation between SINTEF and the universities 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.

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

THE TEM GEMINI CENTRE

The TEM (Transmission Electron Microscopy) Gemini Centre was established in March 2006 and consisted of professors, postdocs and students 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 on 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 quality as a sound basis for growth, not only for the Centre itself, but also for other parts of NTNU and SINTEF.

INTRODUCTION

2013 has been a very special year for the TEM Gemini Centre. The TEM facilities have been significantly upgraded, with 3 new instruments being installed. These cover a wide range of activities, from basic analysis and teaching to leading-edge interdisciplinary research. The new laboratory has been established, adjacent to the NTNU Nanolab. The room have been refurbished and coordinated by JEOL, the supplier for three instruments. It has been a great achievement that all scientific, contract research and teaching activity has been maintained during this period and this has required a great deal of hard work from everyone involved within the group and the realisation of the new facility.

The investment has been through the national infrastructure project, NORTEM, a partnership between NTNU, UiO and 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 is gratefully acknowledged. The infrastructure is described in detail in this annual report.

2013 marks the transition from the planning to installation and the beginning of use of the new facilities. The need for development of the national role, and strategic objectives, such as increased activity at the European level, are also being addressed.

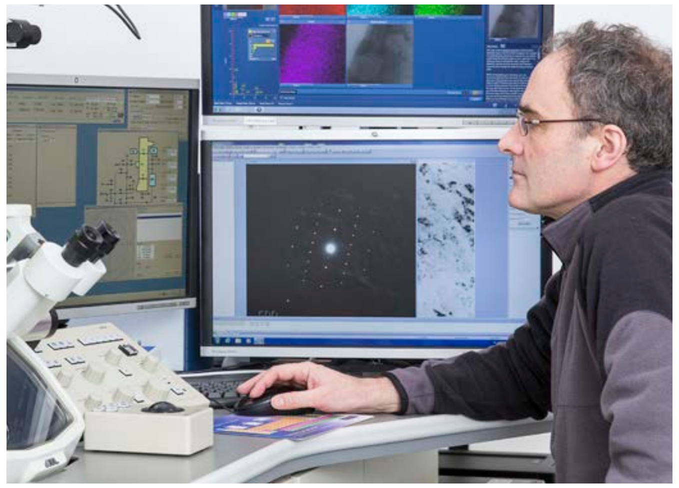
An inauguration seminar was held in September, 2013. The seminar was well attended. Unni Steinsmo, CEO of SINTEF, Kari Melby, Pro-Rector for Research at NTNU, Director for Research Infrastructure Asbjørn Mo, from the Research Council of Norway and Gon'emon Kurihara, President of JEOL Ltd, attended and contributed to mark the formal opening. Seven invited international experts made presentations during a scientific session.

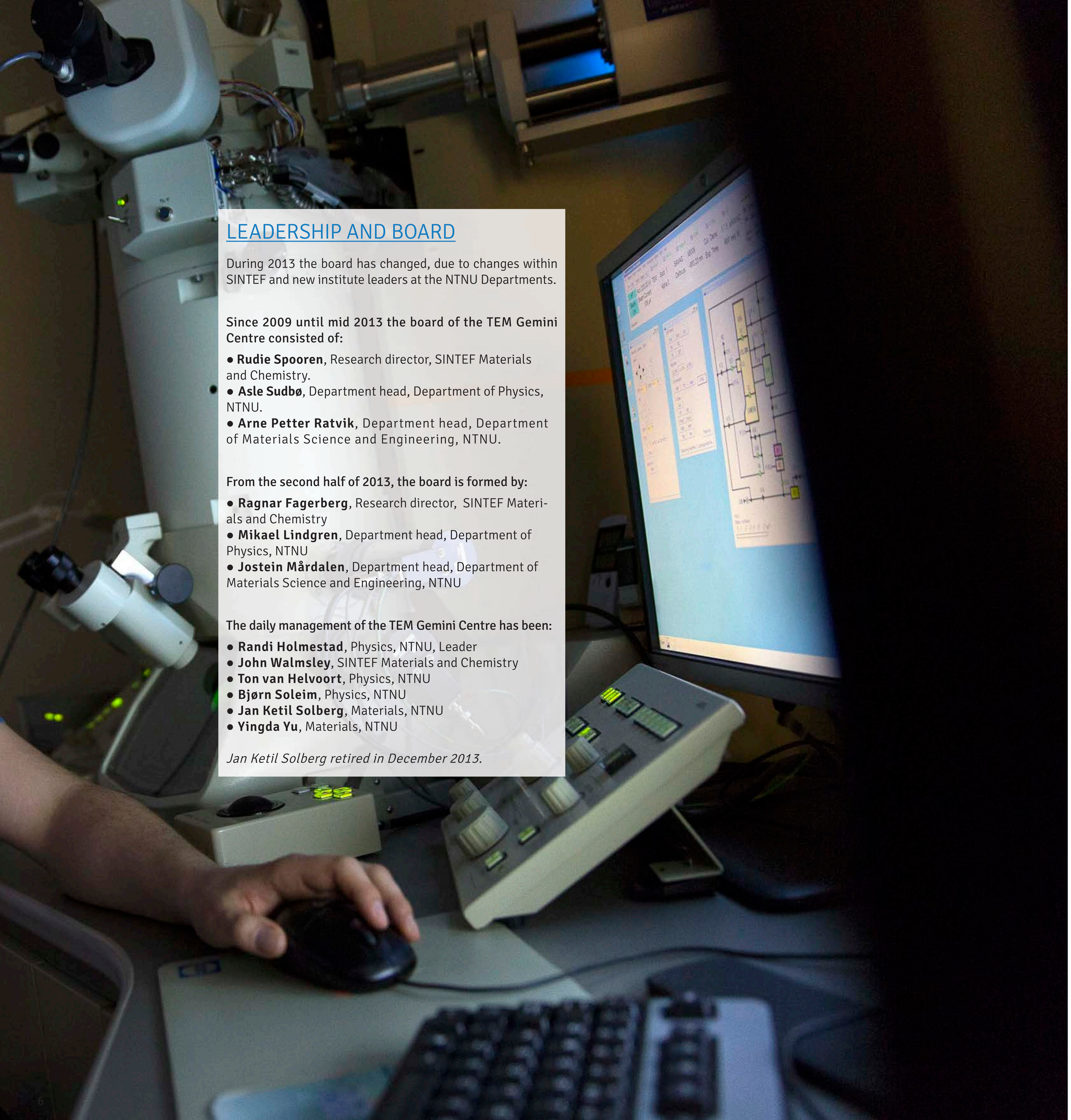
In November 2012 we were re-nominated for 3 new years as a Gemini Centre. We got a positive evaluation and feedback on how the Centre has functioned in the last years. Looking forward we will plan how to fully integrate the NORTEM project with the TEM Gemini Centre – We have begun to discuss integrating the TEM activity at UiO and SINTEF Oslo into the existing Gemini Centre.

The Gemini Centre participates in a range of internal and external projects, including national public and industrial and EU funding. The Centre had about 40 users of TEM and produced more than 40 journal publications in 2013. 16 of these publications have international co-authors. In addition, 6 Masters and 1 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 21 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 publication list at the end, most members of the TEM group participated in international conferences and meetings in 2013.

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 2013. 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
May 2014.





LEADERSHIP AND BOARD

During 2013 the board has changed, due to changes within SINTEF and new institute leaders at the NTNU Departments.

Since 2009 until mid 2013 the board of the TEM Gemini Centre consisted of:

- **Rudie Spooren**, Research director, SINTEF Materials and Chemistry.
- **Asle Sudbø**, Department head, Department of Physics, NTNU.
- **Arne Petter Ratvik**, Department head, Department of Materials Science and Engineering, NTNU.

From the second half of 2013, the board is formed by:

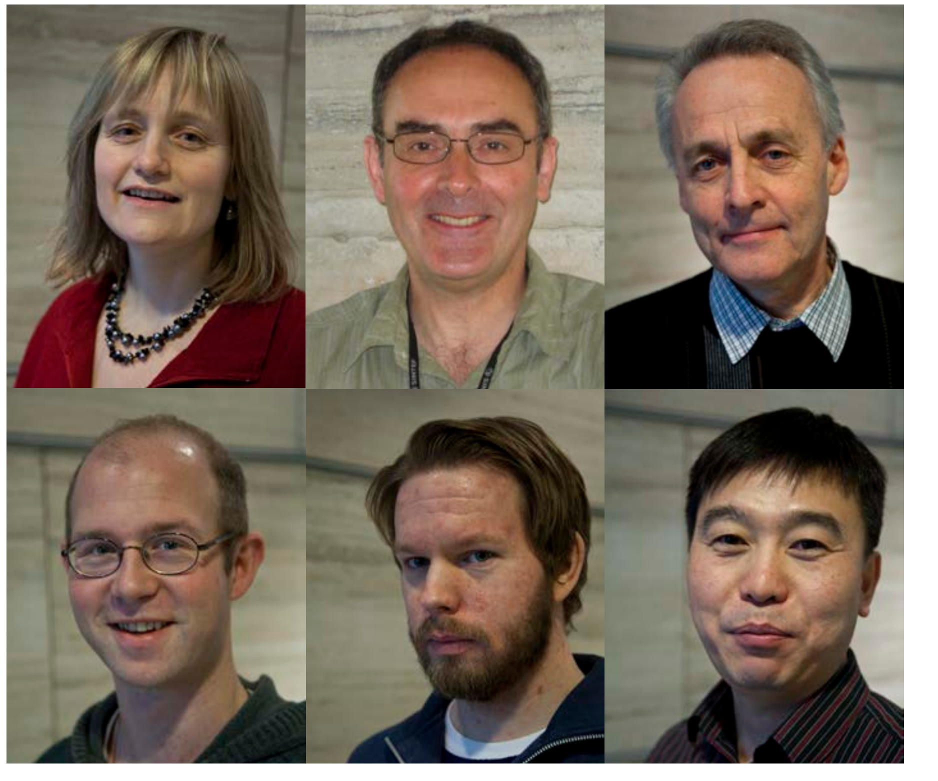
- **Ragnar Fagerberg**, Research director, SINTEF Materials and Chemistry
- **Mikael Lindgren**, Department head, Department of Physics, NTNU
- **Jostein Mårdalen**, Department head, Department of Materials Science and Engineering, NTNU

The daily management of the TEM Gemini Centre has been:

- **Randi Holmestad**, Physics, NTNU, Leader
- **John Walmsley**, SINTEF Materials and Chemistry
- **Ton van Helvoort**, Physics, NTNU
- **Bjørn Soleim**, Physics, NTNU
- **Jan Ketil Solberg**, Materials, NTNU
- **Yingda Yu**, Materials, NTNU

Jan Ketil Solberg retired in December 2013.

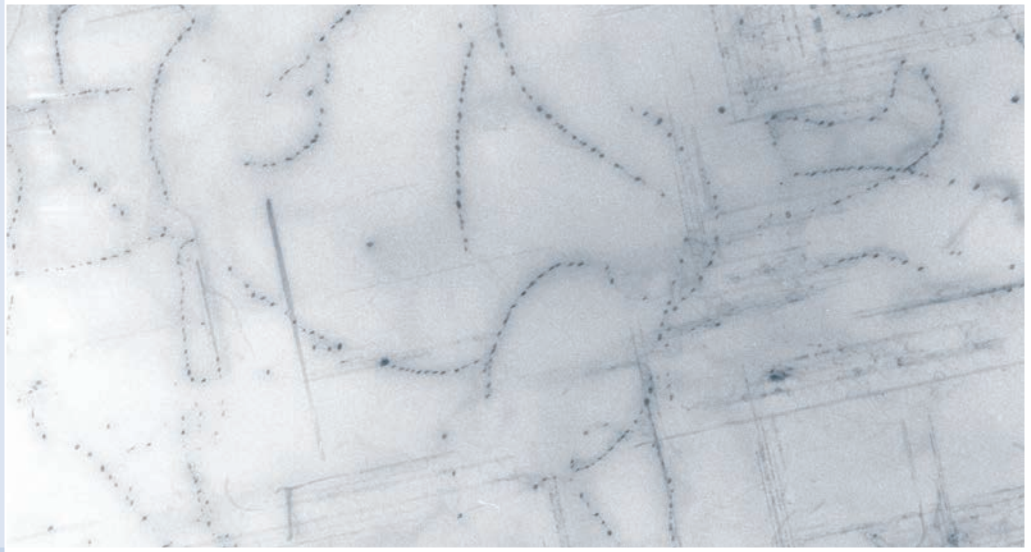
Management and key staff

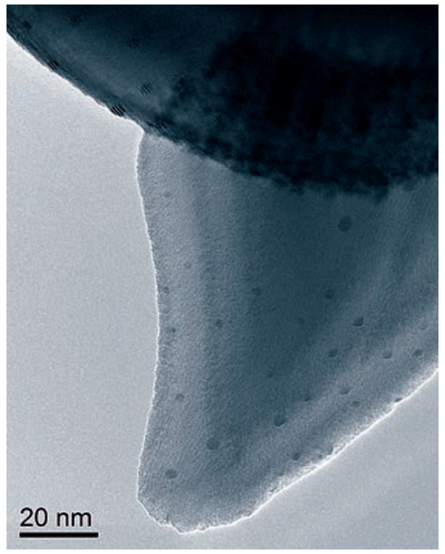


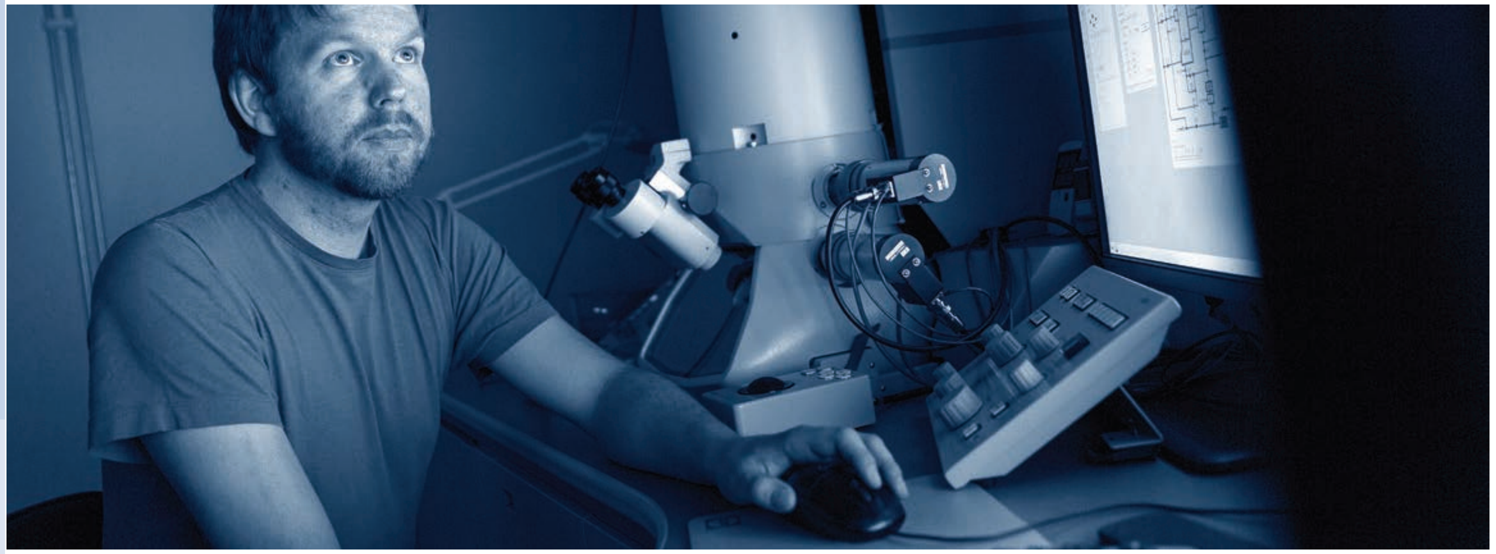
Randi Holmestad, Professor, Physics, NTNU, leader, John C Walmsley, Senior scientist/Professor II, SINTEF Materials and Chemistry, Jan Ketil Solberg, Professor, Materials Science and Engineering, NTNU, Ton van Helvoort, Associate professor, Physics, NTNU, Bjørn Gunnar Soleim, Senior engineer, Physics, NTNU, Yingda Yu, Senior engineer, Materials Science and Engineering, NTNU.

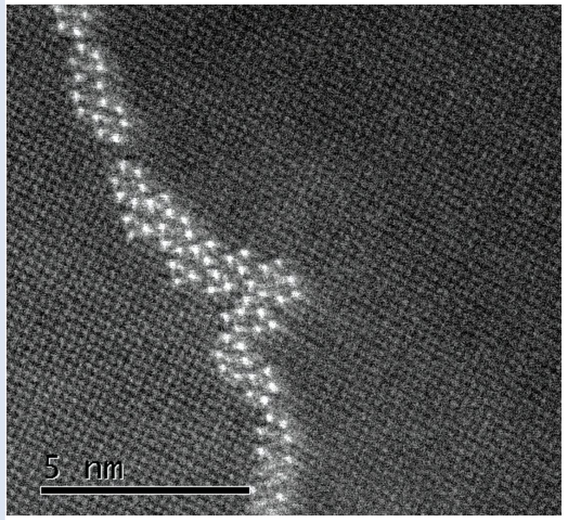
INSTRUMENTATION

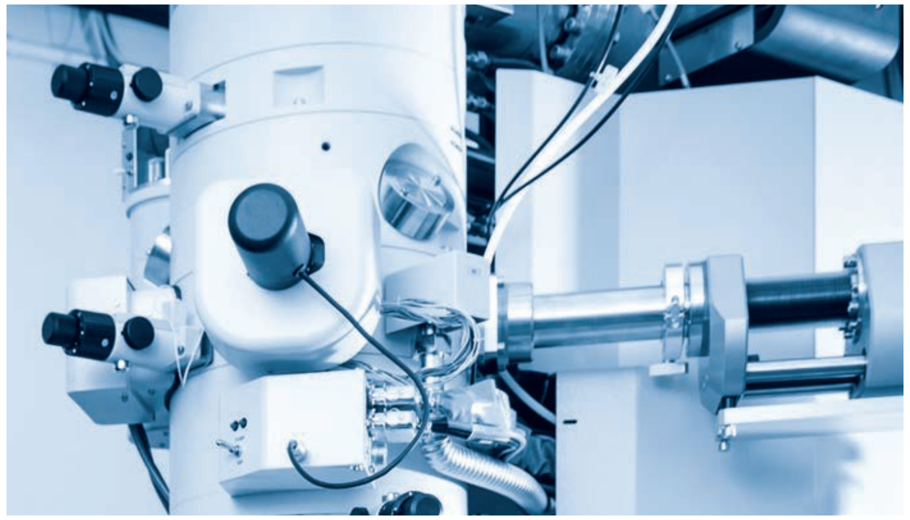
AND THE NORTEM PROJECT











TEM INSTRUMENTS AND SPECIMEN PREPARATION FACILITIES

2013 has been a transition year for the Centre. In the beginning of the year we had 3 working and well-used TEMs: a JEOL 2010F (2002) and a Philips CM30 (1989) at DP, and a JEOL 2010 (1993) at DMSE. Three new microscopes were installed as part of the NORTEM project during 2013. We installed a JEOL 2100 LaB6 instrument during the summer, the double corrected JEOL ARM200F was accepted in October, and the 2010F was replaced with a JEOL 2100F in December. The table to the right shows lists of all instrument used in 2013. The new instruments will be fully described in the next few pages.

The 2010F was used for research and teaching in 2013. The most important techniques were lattice imaging TEM (HRTEM), scanning transmission electron microscopy (STEM, including quantitative HAADF) and electron energy loss spectroscopy/energy filtered imaging (EELS/EFTEM). The instrument was gradually stripped down in the fall of 2013, and most of the additional equipment was transferred to the new 2100 and 2100F, including the GIF (2100), DigiScan, EDS, CCD camera and ASTAR (2100F). Sample holders including a reaction holder and two tomography holders, single and dual/axis rotation, can still be used on all the three new machines. The 2010F has been a very productive and important TEM for making the TEM Gemini Centre successful. It has now been taken apart and sold to another customer of JEOL.

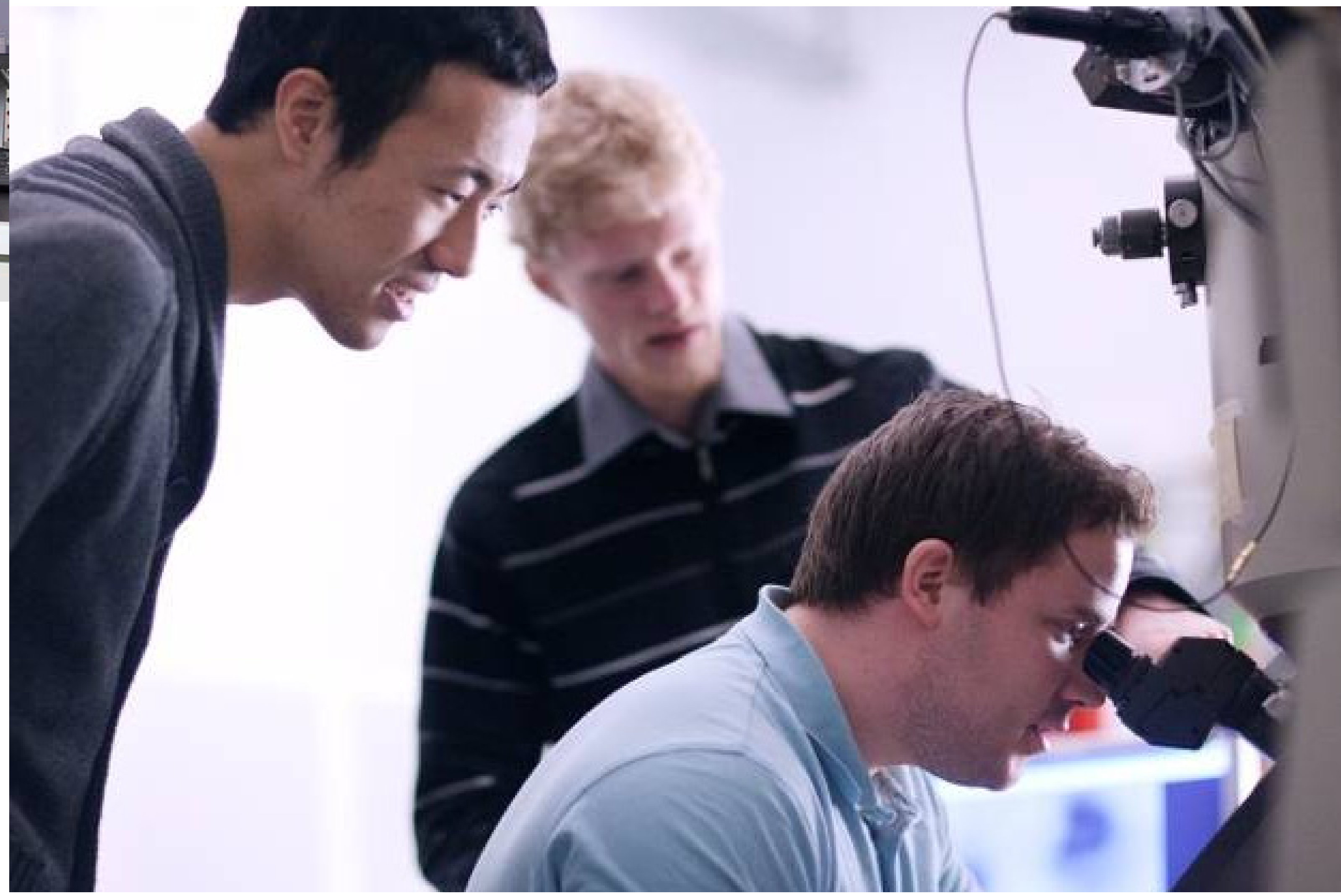
The CM30 is now 25 years old, and is still central to both teaching and research. In the recent years it has been used quite a lot and remains, even after installing the new TEMs, in significant use! This microscope is mainly used for conventional TEM, especially for electron diffraction and bright field/dark field studies. The microscope has a PEELS for thickness measurements and has been extensively used in aluminium research. Beside the CCD recording system, negatives still prove to be a good recording medium for the type of work done at the CM30. We expect to continue to use the CM30 for some more years, although maintenance is increasingly an issue.

The JEOL 2010 at DMSE, which is 21 years old and provides similar analysis to the CM30 has its main application areas in metallurgy and nanotechnology. It is used in undergraduate and post-graduate research and teaching. Maintenance and provision of spare parts are increasingly important for the latter two instruments. This illustrates the importance of ongoing evaluation and investment in large-scale facilities.

Microscopes in the TEM Gemini Centre today. All TEMs have a range of holders [1]

Instrument	Location	Configuration	Installed
JEOL 2010F	*DP, Natural Science building*	*FEG, 200 kV, GIF, EDS, gas reaction and tomography holders, 2k cameras*	*2002, decommissioned autumn 2013*
Philips CM30	*DP, Natural Science building*	*LaB6, 300 kV, PEELS, EDS, 1k camera*	*1989*
JEOL 2010	*DMSE, Alfred Getz vei 2*	*LaB6, 200 kV, GIF, EDS, 2k camera*	*1993*
JEOL 2100	*DP, Chemistry block I*	*LaB6, 200 kV, STEM, GIF, EDS, Orius camera*	*Aug. 2013*
JEOL ARM 200F	*DP, Chemistry block I*	*Cold FEG, image and probe corrected, Quantum GIF, Centurio EDS, CCD cameras*	*Oct. 2013*
JEOL 2100F	*DP, Chemistry block I*	*FEG, 200 kV, EDS, US camera, ASTAR, gas reaction and tomography holders*	*Dec. 2013*

[1] ***single, double, heating, cooling.***



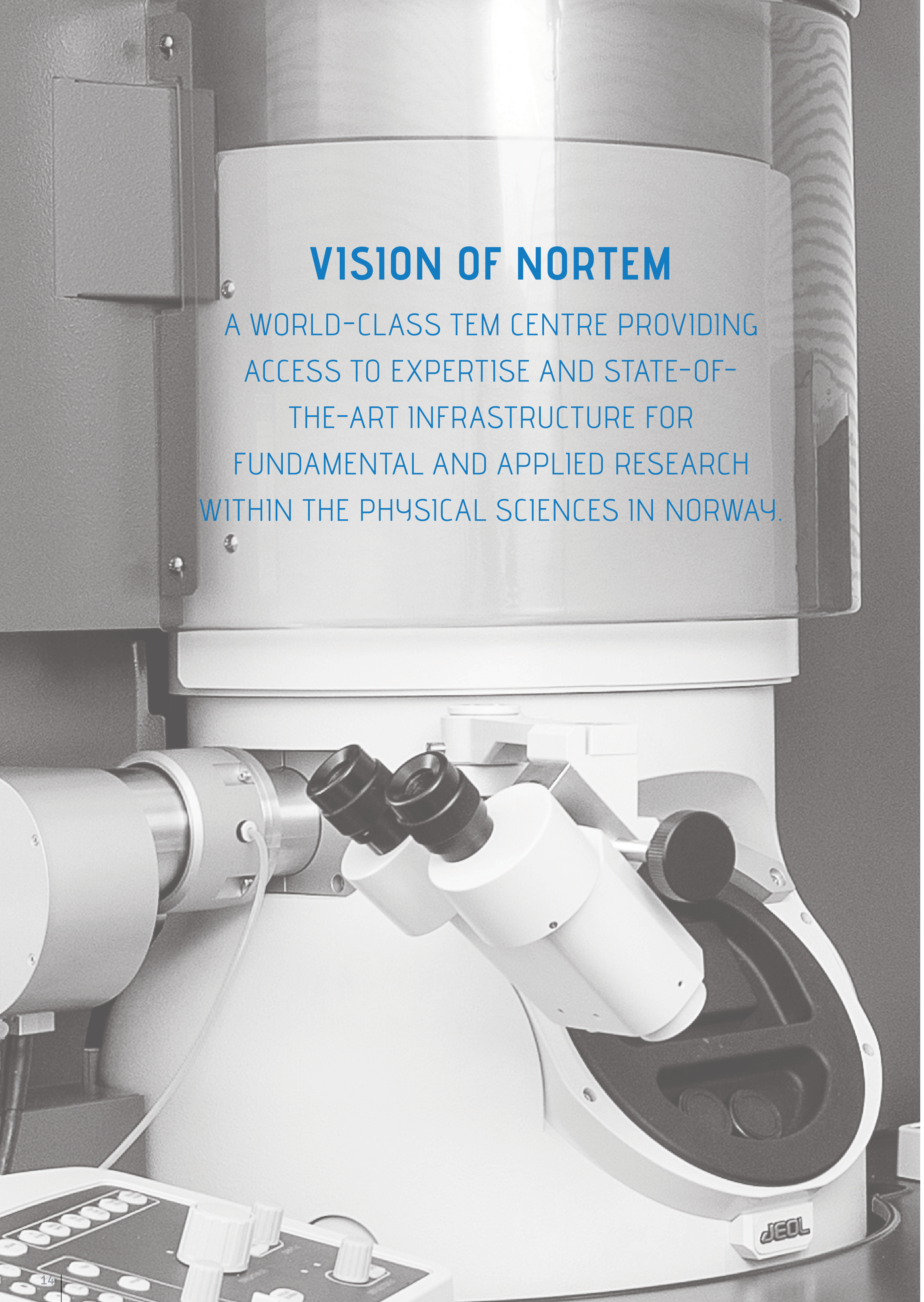
Takeshi Saito, Sigurd Wenner and Jason Granholt working on the CM30. Photo: Terje Trobe .

Ragnhild Sæterli preparing TEM specimens using the tripod polisher. Photo: Terje Trobe.

The limiting factor in a (S)TEM study is often the specimen quality rather than the microscope. This is particularly true for imaging and analysis in aberration corrected instruments. 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 installed in 2011 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, often avoiding artefacts introduced by other methods. For soft materials, as polymers, ultramicrotomy is an attractive preparation technique, but it is also used for preparation of catalysts, surface structures and nanoparticles. Several TEM projects could utilize the FIB at NTNU NanoLab with lift-out option for site-specific TEM specimen preparation. Effective access to this technique has become essential to the activities of the Gemini Centre.

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 different 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, as microscope vendor, 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 nanotechnology and solar energy in Norway. 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 have been the prime focus and have required a lot of the Centre's resources in 2013. We have so far had a smooth transition period from the old to the new instruments.

A LOT HAS CHANGED IN THE LABS DURING 2013.

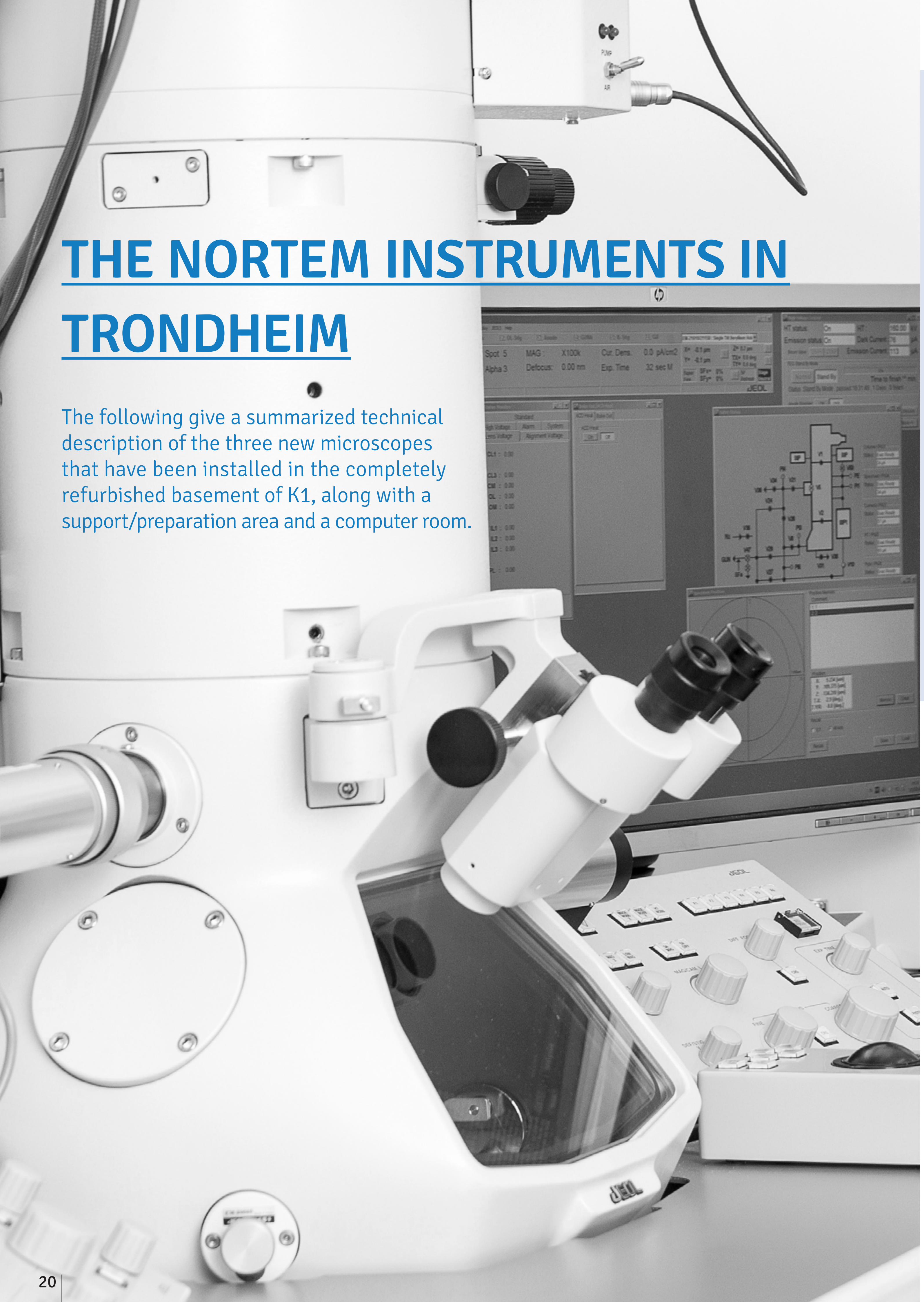
The next pages show collages of pictures taken over the year to show the progress of the different microscopes and the room facilities – from the rebuilding of the rooms, the equipment arriving Trondheim, the assembling of the microscopes and to the finished setups for the different instruments.

2100

2100F

ROOM REBUILDING

ARM



THE NORTEM INSTRUMENTS IN TRONDHEIM

The following give a summarized 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 (in use from Aug. 2013)

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 will be 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 (in use from Dec. 2013)

This FEG TEM will be optimized for all-round advanced materials studies with a special focus on precession diffraction 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 EDS (solid angle 0.23 sr)*
- *ASTAR orientation mapping system*
- *Gatan TEM/STEM Electron Tomography*

1) JEOL JEM-ARM200F (in use from Sept. 2013)

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 EDS and EELS systems allow unique studies at the atomic scale.

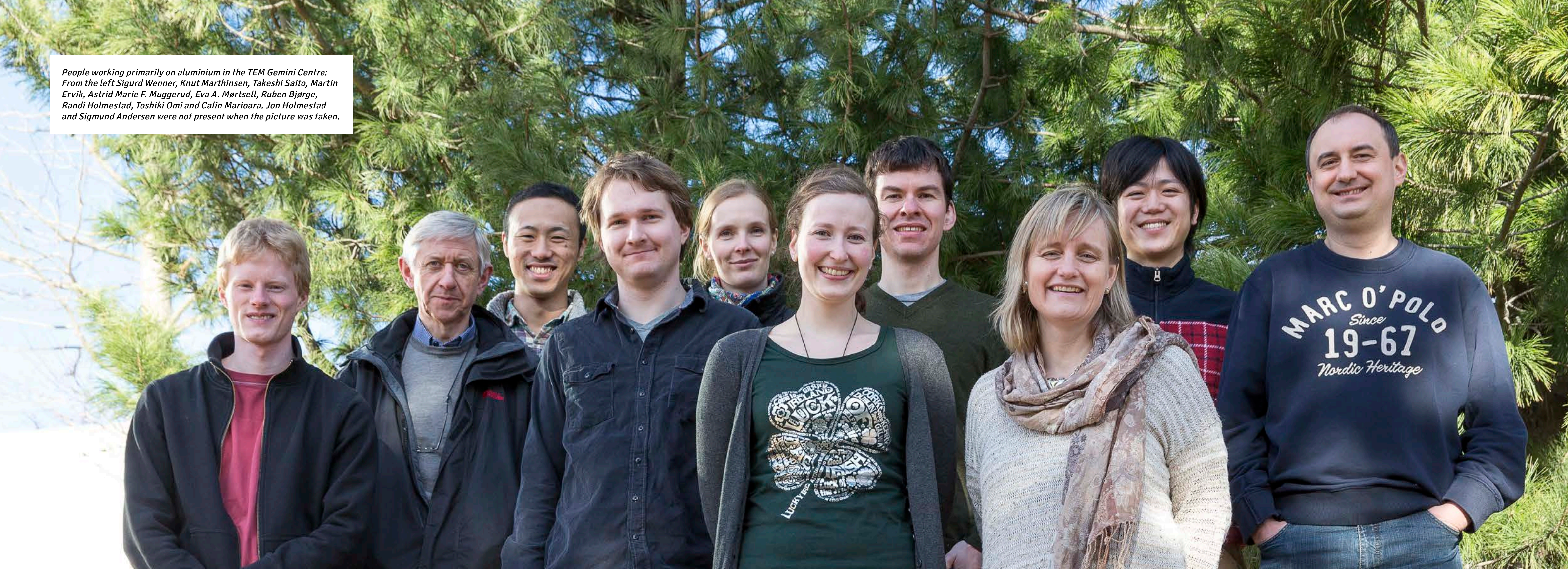
The ARM200F features:
- *Cold field emission gun with energy spread of 0.3 eV*
- *Cs-probe corrector*
- *Cs-image corrector*
- *Centurio SDD EDS (solid angle 0.98 sr)*
- *Quantum GIF with DualEELS*
- *2k Orius CCD (side-mounted) 2k UltraS can CCD (bottom mounted)*
- *Stable 5-axis gonio with piezo control in x, y and z-directions*
- *Detectors for BF, ABF, ADF and HAADF STEM*
- *Fully aligned at 80 and 200 kV*

SPECIMEN HOLDERS

For all three machines a broad range of holders is available. This includes single tilt holders, double tilt holders (±30 degrees), cold stage holder, heating holder, environmental cell/transfer holder, tomography holders and a rotation holder.

FOCUS AREAS

TEM is a powerful technique for fundamental and applied research in the physical sciences, used in different fields from geology, metallurgy and semiconductor industry to fundamental chemistry and physics research. NORTEM has identified four focus areas, which are already important for the TEM Gemini Centre activities today. 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 is playing 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 2013. The next sections describe these activities. Particularly strong were the activities in aluminium alloys and light metals.



People working primarily on aluminium in the TEM Gemini Centre: From the left Sigurd Wenner, Knut Marthinsen, Takeshi Saito, Martin Ervik, Astrid Marie F. Muggerud, Eva A. Mørtsell, Ruben Bjørge, Randi Holmestad, Toshiki Omi and Calin Marioara. Jon Holmestad and Sigmund Andersen were not present when the picture was taken.

ALUMINIUM ACTIVITIES (LIGHT METALS)

The study of aluminium alloy systems using TEM has been a pillar in the Trondheim TEM environment for many years. The KK (Nucleation Control) project ended in 2012, but one PhD student, Jon Holmestad is still working on this project as he started 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 2013. In this project TEM has been one of many characterisation tools which have been used in interdisciplinary studies to relate nano-scale understanding of microstructure and chemistry to macroscopic 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 already been published in this project and more will follow. The competence project Moreal, which is focusing mainly on 3xxx alloys, is in its final year. In this project SAPA is involved in addition to Hydro. Several PhD students have worked on this project, using SEM and TEM. PhD student Astrid Marie F. Muggerud is focusing on different TEM techniques used on dispersoids in her study.

In the bilateral competence building project 'The Japanese-Norwegian Al-Mg-Si precipitation project' Sigurd Wenner and Takeshi Saito are working as PhD students together with SINTEF scientists. Both will finish their PhD studies in 2014, and the scientific output measured in good publications has been high in 2013. The main topic in this project is 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 new aluminium projects started up in 2013. 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 Mørtsell 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 in order to test out the effect of different addition in very lean 6xxx alloys. In addition to SINTEF activity, also the Master student Toshiki Omi from Tokyo Tech has been working on this project in 2013. 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. A postdoc dedicated to HAADF STEM will be hired in the Spring of 2014. In collaboration with SINTEF scientists, these two will be 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 ordered and are now examined for different types of precipitates. STEM images from the new ARM microscope will be a main contribution to this project. These two projects will keep our Aluminium-TEM activities high in the coming 5 years period.

TEM Gemini group members actively participate in international aluminium scientific meetings. For example, 6 people from the Gemini Centre presented their work at the Thermec conference in Las Vegas in December. In 2014 the large ICAA conference is going to be held in Trondheim, with many contributions from the TEM Gemini Centre.

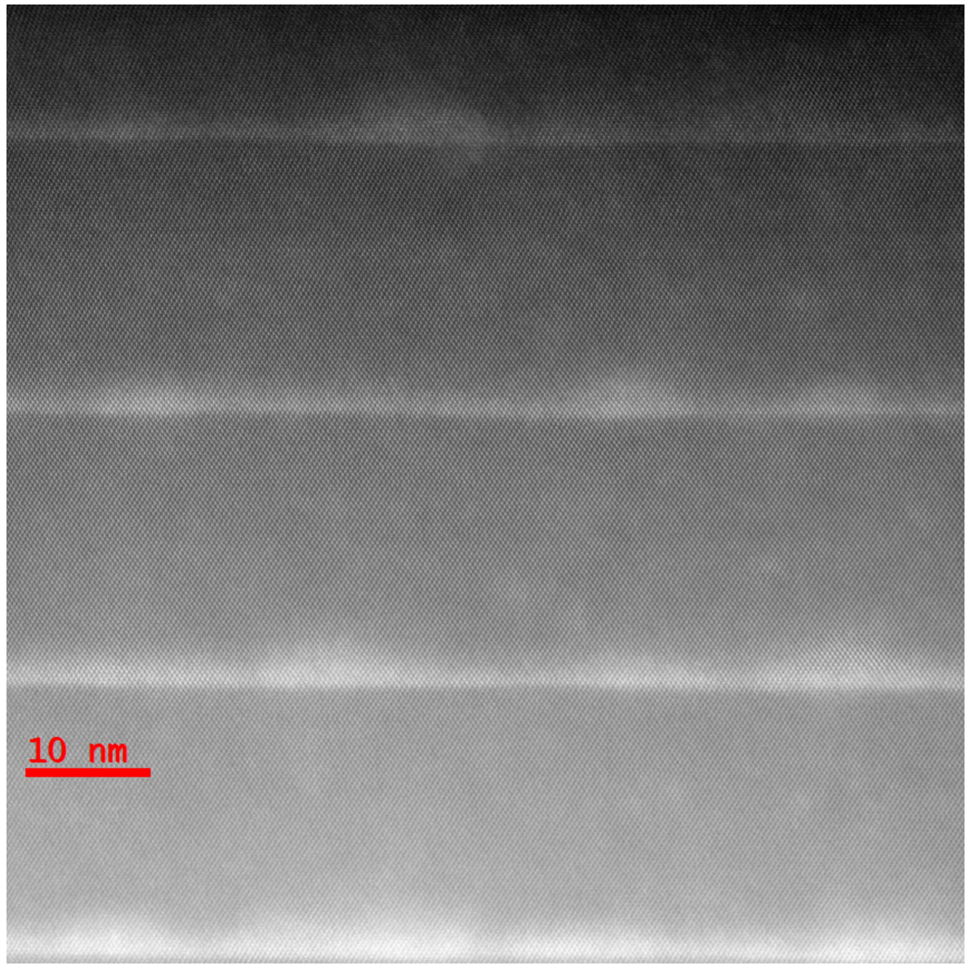
SOLAR CELLS

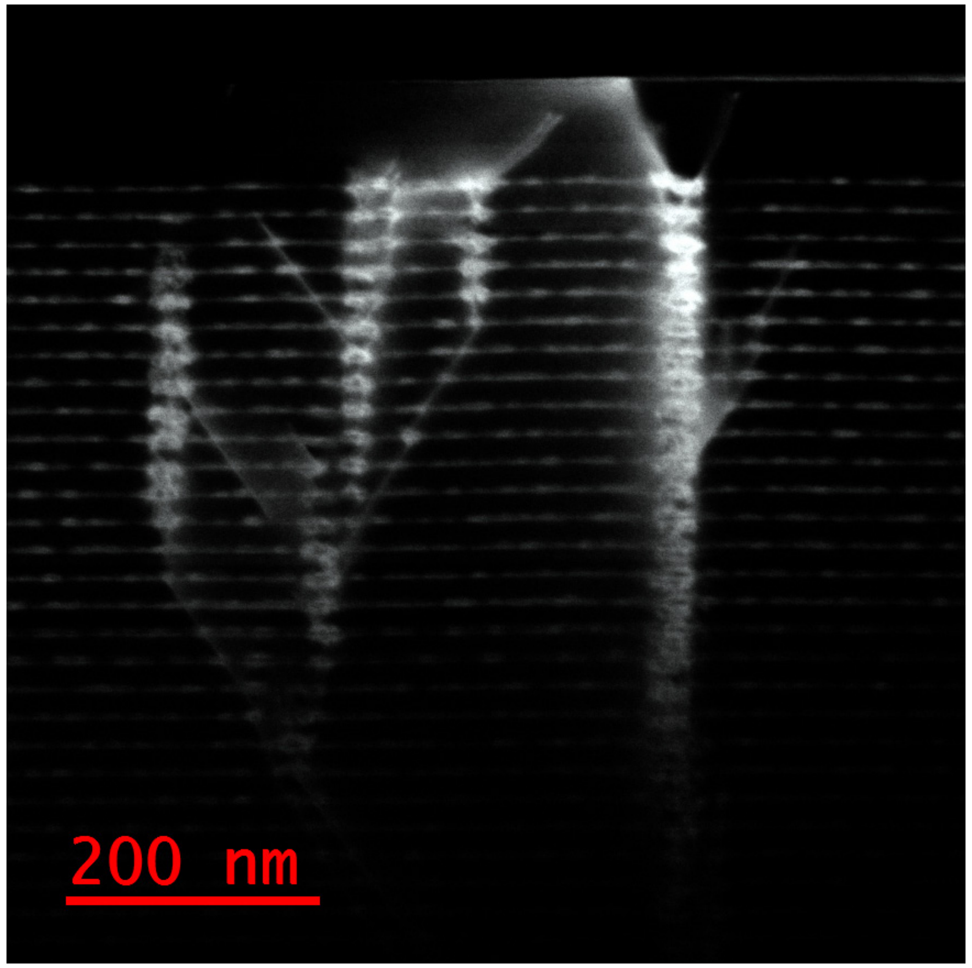
To improve the efficiency of conventional and novel types of solar cells, materials and devices are optimized at the length scale where TEM is one of the main characterization tools. TEM Gemini Centre activities within solar cells include both studies of conventional silicon solar cell materials and new materials, including InAs quantum dots in GaAs and GaAs nanowires for solar cell applications. Two 2-year Master students have been working within the field of solar cells in 2013. Eivind Seim and Espen Undheim will hand in their thesis Spring 2014. The RENERGI project Nanosolar – Core-shell ended in 2013, in collaboration with Profs. Weman and Fimland at IET-NTNU, and we are looking for a follow-up project in which we can do TEM on high-performance nanowire-based solar cells.

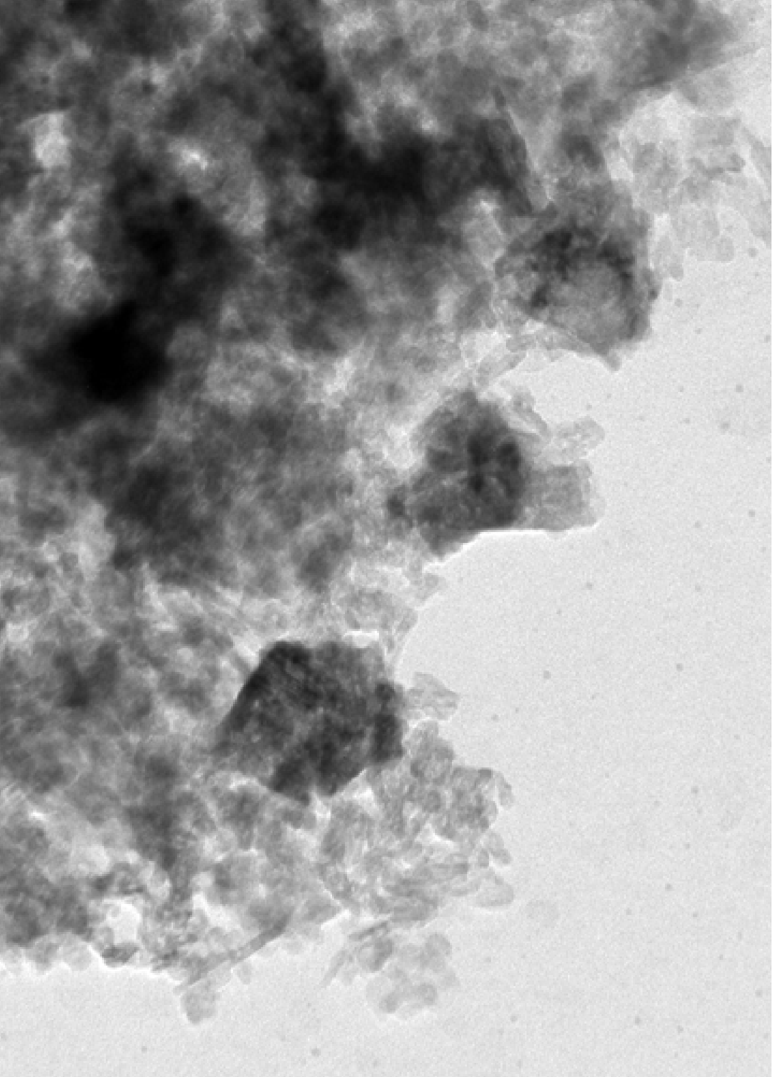
In 2013 we started up a new research council project within the Energix programme, called High Efficiency Quantum Dot Intermediate Band Solar Cells (HighQ-IB) where associate Prof. Turid W. Reenaas is the project leader. Maryam Vatanparast is since November 2013 hired as a PhD student on this project. The new ARM microscope will be highly used in this project 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 FME postdoc postdoc Ragnhild Sæterli worked on TEM studies of solar cell materials, including conventional Si solar cells, thin films and nanostructured materials for photovoltaic applications during 2013. The SINTEF TEM activities span also this broad spectrum.

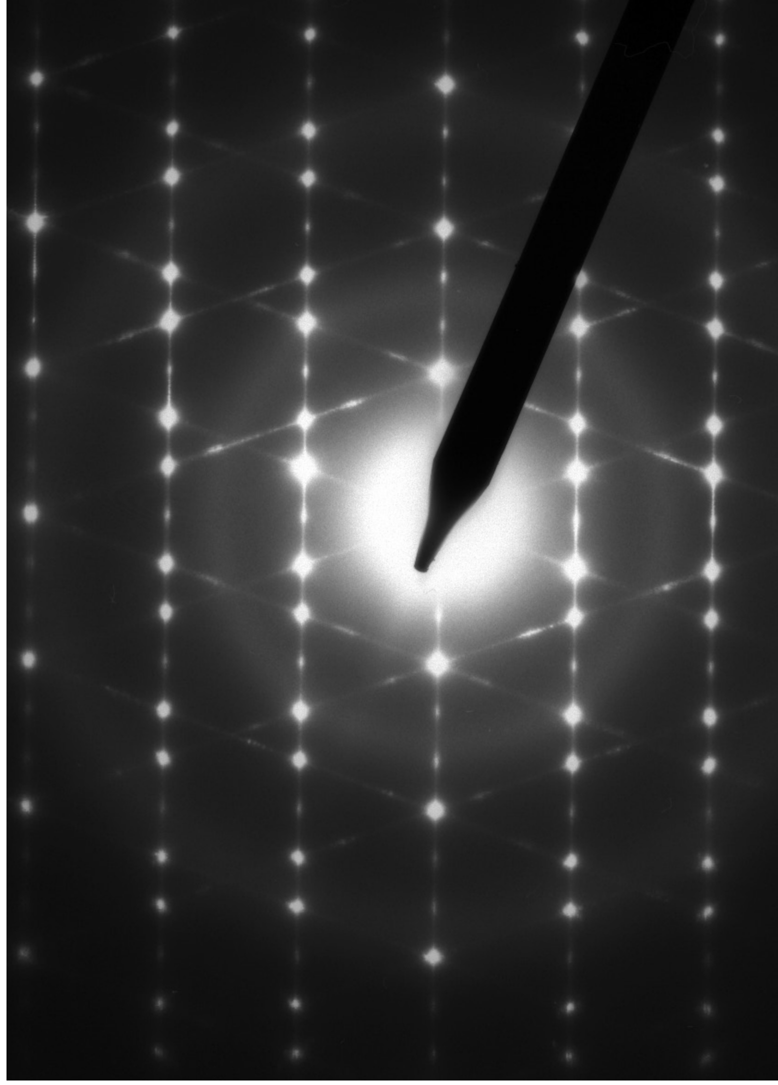
The Gemini Centre had in 2012 two visiting PhD students from Karunya University in India. Arivazhagan V and Manonmani Parvathi were supported by a Yggdrasil collaboration project from the Research Council. Both are working on new types of thin film multiple quantum well structures for solar cell applications. The structures were grown by vacuum evaporation techniques at their home institution. The materials they studied are PbSe/ZnSe and PbTe/InSe multiple quantum walls, respectively. During their stay in Trondheim they prepared cross-section specimens, learned and used TEM in order to understand their materials better. Ragnhild Sæterli was their supervisor during their stay. Part of their work done in the TEM Gemini Centre has now been published in 2013.







Hanne Kauko submitted and defended her PhD on quantitative HAADF STEM in December 2013. Her examiners where Prof. D. Gerthsen, Karlsruhe Institute of Technology (Germany) and Dr. M. Verheijen, Technical University Eindhoven/ Philips Research (Netherlands). Photo: Terje Trobe.



CATALYSIS AND ENERGY STORAGE 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), Statoil and the inGAP SFI, Innovative natural Gas Processes and Products. Trung Tran has been working asa postdoc studying catalyst nanoparticles in a project funded by the KOSK II programme in cooperation with Magnus Rønning and De Chen at NTNU IKP, and finished his contract in 2013. This work has included using electron tomography and atomic-scale imaging and EELS in a bottom-up approach to understanding catalyst nanoparticle systems. We expect to extend this work, particularly making use of the new ARM TEM. We have worked towards integrating TEM analysis with other characterisation techniques such as X-ray Absorption Spectroscopy High Resolution X-ray Powder Diffraction and Raman Spectroscopy at the same time as developing approaches for understanding catalyst system deactivation processes.

During 2013 we participated in an application to the Gassmaks programme lead by De Chen and Hilde Venvik at NTNU IKP to develop catalysts and materials for a compact steam reformer which has now received funding. Here we will address both catalyst and materials issues, such as degradation of steels 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.

In April 2013 Yanjie Gan was recruited into the Gemini Centre as a postdoc, to work on a project 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. Within the area of hydrogen technology we have continued to publish work from earlier projects with the Institute for Energy Technology and participate in new project initiatives.

We have collaborated with the Energy Research Institute (IFE) which has spanned several projects in the area of energy storage materials. TEM analysis is rather challenging, due to the sensitivity of the alanate to air and electron beam irradiation. However, it has been possible to contribute to a multidisciplinary programme involving synthesis, testing and characterisation, by other techniques such as synchrotron x-ray diffraction and neutron diffraction that has brought forward the fundamental understanding of these materials. Further work has been published during 2013.

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. 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 other PhD students working within the TEM Gemini Centre during 2013. The Network is also funding PhD Magnus Nord, who is working in the field of oxide electronics. Magnus is investigating epitaxial complex oxide thin films of LaSrMnO3 with advanced TEM. 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. Related to that activity Hanne Kauko defending her PhD, "Quantitative scanning transmission electron microscopy studies on heterostructured GaAs nanowires", in 2013. She used HAADF STEM imaging and simulations to determine the composition of $GaAs_{1-y}Sb_y$ segments and $Al_xGa_{1-x}As$ shells with a resolution beyond what standard analytical technique could demonstrate. The international collaborators in this project were Prof. A Rosenauer in University of Bremen (Germany) and Prof. J. Etheridge in Monash University (Australia). We expect that the TEM work dedicated to Nanotechnology will be growing and one of the new TEMs is dedicated to nanotechnology.

PEOPLE WORKING IN THE TEM GEMINI CENTRE DURING 2013

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)
Jan Ketil Solberg (Professor, DMSE, NTNU)
Ton van Helvoort (Associate professor, DP, NTNU)
Knut Marthinsen (Professor, DMSE, NTNU)
Bjørn Gunnar Soleim (Senior engineer, DP, NTNU)
Yingda Yu (Senior engineer, DMSE, NTNU)
Sigmund J. Andersen (Senior scientist, SINTEF)
Jesper Friis (Research scientist, SINTEF)
Yanjun Li (Research scientist, SINTEF, since October Professor DMSE)
Calin Marioara (Research scientist, SINTEF)
Per Erik Vullum (Research scientist, SINTEF and Associate Professor II)
Flemming Ehlers (Postdoc, DP, NTNU)
Ragnhild Sæterli (Postdoc, DP, NTNU)
Dung Trung Tran (Postdoc, DP, NTNU)
Ruben Bjørge (Postdoc, DP, NTNU)
Jon Holmestad (PhD student, DP, NTNU)
Hanne Kauko (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 Mørtsell (PhD student, DP, NTNU)
Martin Ervik (PhD student, DP, NTNU)
Maryam Vatanparast (PhD student, DP, NTNU)
Sindre Bunkholt (PhD student, DMSE, NTNU)
Qinglong Zhao (PhD student, DMSE, NTNU)
Sethulakshmy Jayakumari (Master student, DP, NTNU)
Andrea Klubicka (Master student, DP, NTNU)
Ingrid Snustad (Master student, DP, NTNU)
Espen Undheim (Master student, DP, NTNU)
Eivind Seim (Master student, DP, NTNU)
Line Rude Jacobsen (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)

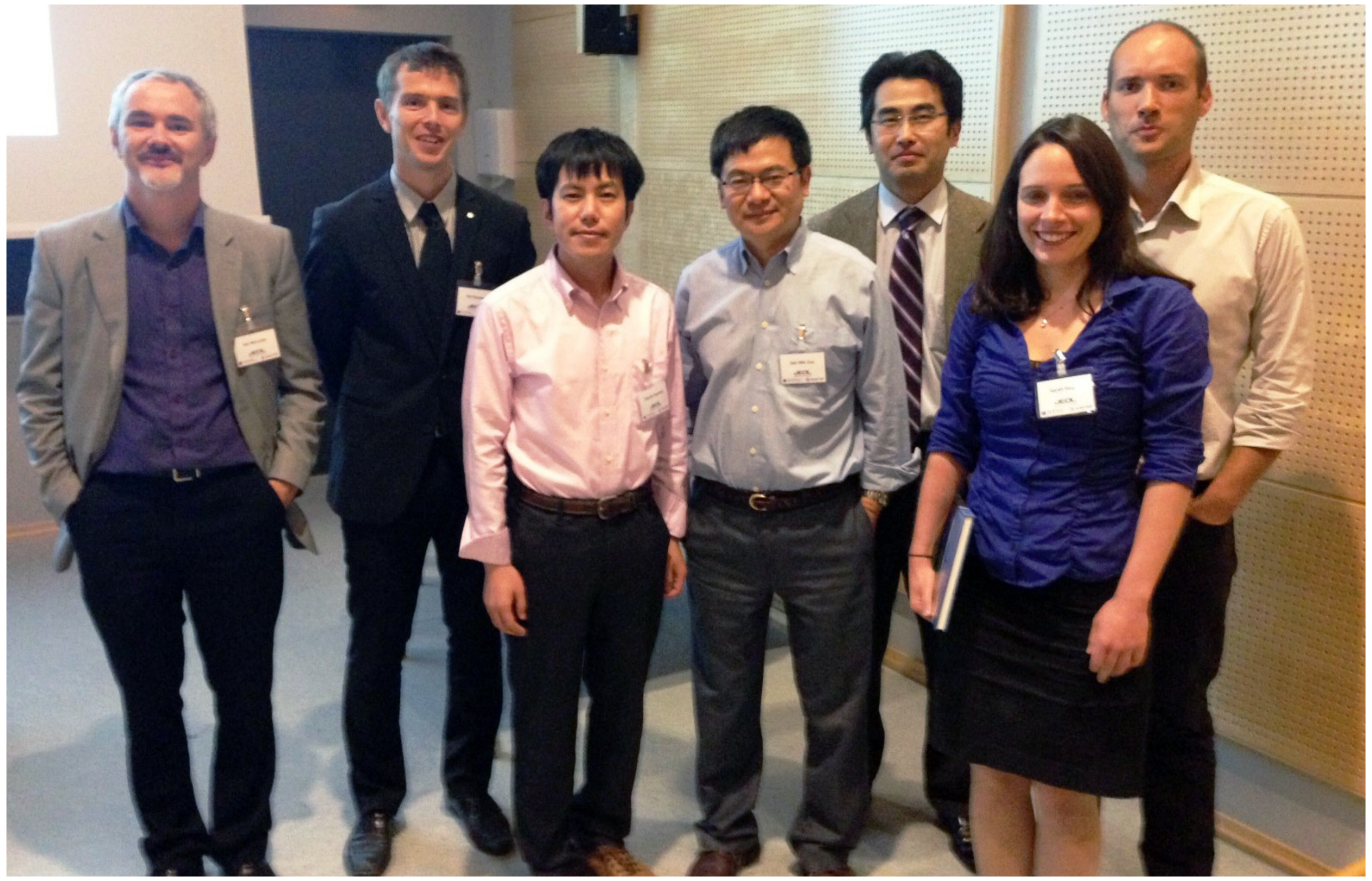
INAUGURATION OF JEOL MICROSCOPES IN TRONDHEIM, NORWAY

10th September 2013 the new transmission electron microscopes (TEMs) in the TEM Gemini Centre in Trondheim were inaugurated. The three new JEOL microscopes are presented on page 21 in this report. The President of JEOL Ltd. Gon'emon Kurihara participated in the formal ceremony before lunch, together with SINTEF's president Unni Steinsmo and NTNUs pro-rector for research Kari Melby. A contract for a JEOL Competence Centre (JCC) in Trondheim was signed by the three leaders. In this contract JEOL, SINTEF and NTNU agrees to have a common competence building around the new instruments, including application specialist visits and technical support. JEOL will use Trondheim as a demo site, and also the plan is to organize common workshops and meetings to exchange experiences.

85 people participated in the inauguration and were given a guided tour of the laboratories and the new microscopes. After the official opening there was a scientific seminar with seven invited speakers from around the world, giving presentations about what is possible to achieve with the new generation of TEMs. The scientific seminar had more than 50 participants. Pictures from the inauguration day, including the full program, are shown on the next pages.

John Walmsley shows the flagship instrument, a JEOL JEM-ARM200F ColdFEG electron source with probe and image correction. Photo: Irene Aspli.

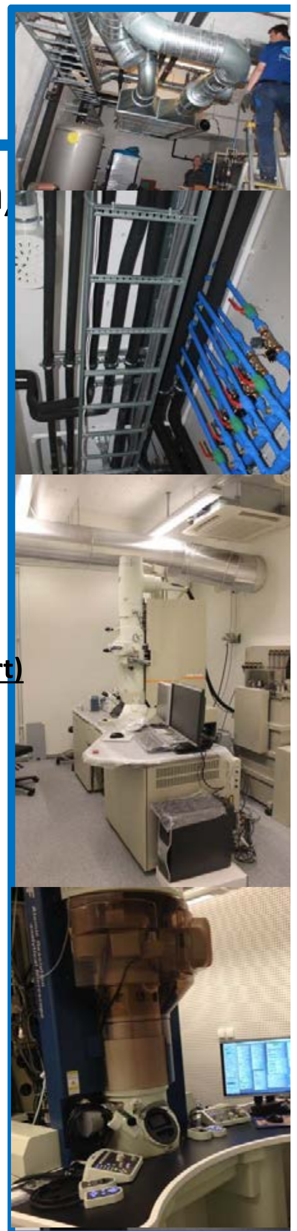
Official opening with the president of JEOL: From the left Unni Steinsmo, and Rudie Spooren, from SINTEF, Kari Melby, NTNU, Spyros Diplas, SINTEF, Gon'emon Kurihara, JEOL and Randi Holmestad, NTNU. Photo: Irene Aspli



Speakers at the seminar in advanced TEM. From the left Ian MacLaren (Glasgow University), Per Persson (Linköping University), Takeshi Kasama (CEN, DTU), Jian Min Zuo (University of Illinois), Masahiro Kawasaki (JEOL USA), Sarah Haigh (Manchester University) and Williams Lefebvre (University of Rouen). Photo: Randi Holmestad

TEM Gemini Centre · Forskningsrådet · JEOL Solutions for Innovation

Inauguration of NORTEM JEOL TEMs in Trondheim, Tuesday 10. Sept. 2013 in R8, Realfagbygget



<u>09-13 Inauguration</u> (chair J. Walmsley)

09.00 Welcome and introduction (Spyros Diplas, project leader NORTEM)
09.05 NORTEM Trondheim node (R.Holmestad, J. Walmsley and T. van Helvoort)
09.40 Key Principles for Norwegian Research Infrastructure Funding (Asbjørn Mo)
10.00 Speeches by Kari Melby, Pro-Rector for Research of NTNU
Unni Steinsmo, CEO of SINTEF
Gon'emon Kurihara, President of JEOL Ltd.
10.50 Signing of JCC (JEOL Competence Centre) contract by JEOL, SINTEF and NTNU
11.00 Guided tours of the facility in KJ1 and lunch in Realfagkantina

<u>13- 17 Scientific seminar on advanced TEM (chairs R. Holmestad and A. van Helvoort)</u>

13.00 Jian-Min Zuo, University of Illinois, US, "The pursuit of higher resolution: past, present and scientific opportunities"
13.30 Per Persson, Linköpings University, Sweden, "The long road towards scientific results in a new high resolution tem facility"
14.00 Takeshi Kasama, DTU, Denmark, "Nanoscale magnetic imaging of iron oxides"
14.30 Sarah Haigh, University of Manchester, UK, "High resolution imaging and EDX elemental mapping of low dimensional materials"
15.00 Coffee break
15.30 Masahiro Kawasaki, JEOL USA Inc, "Introduction of an AC STEM ARM-200F and its application for a thin film"
16.00 Williams Lefebvre, University of Rouen, France, "Recent results obtained on the "probe-corrected" ARM 200F at the University of Rouen"
16.30 Ian MacLaren, University of Glasgow, UK, "Quantitative Studies of Nanoscale Chemistry in a next generation STEM"

<u>19.00 Dinner</u> (Tapas buffet at Kvilhaugen gård)

SINTEF · UiO : University of Oslo · NTNU

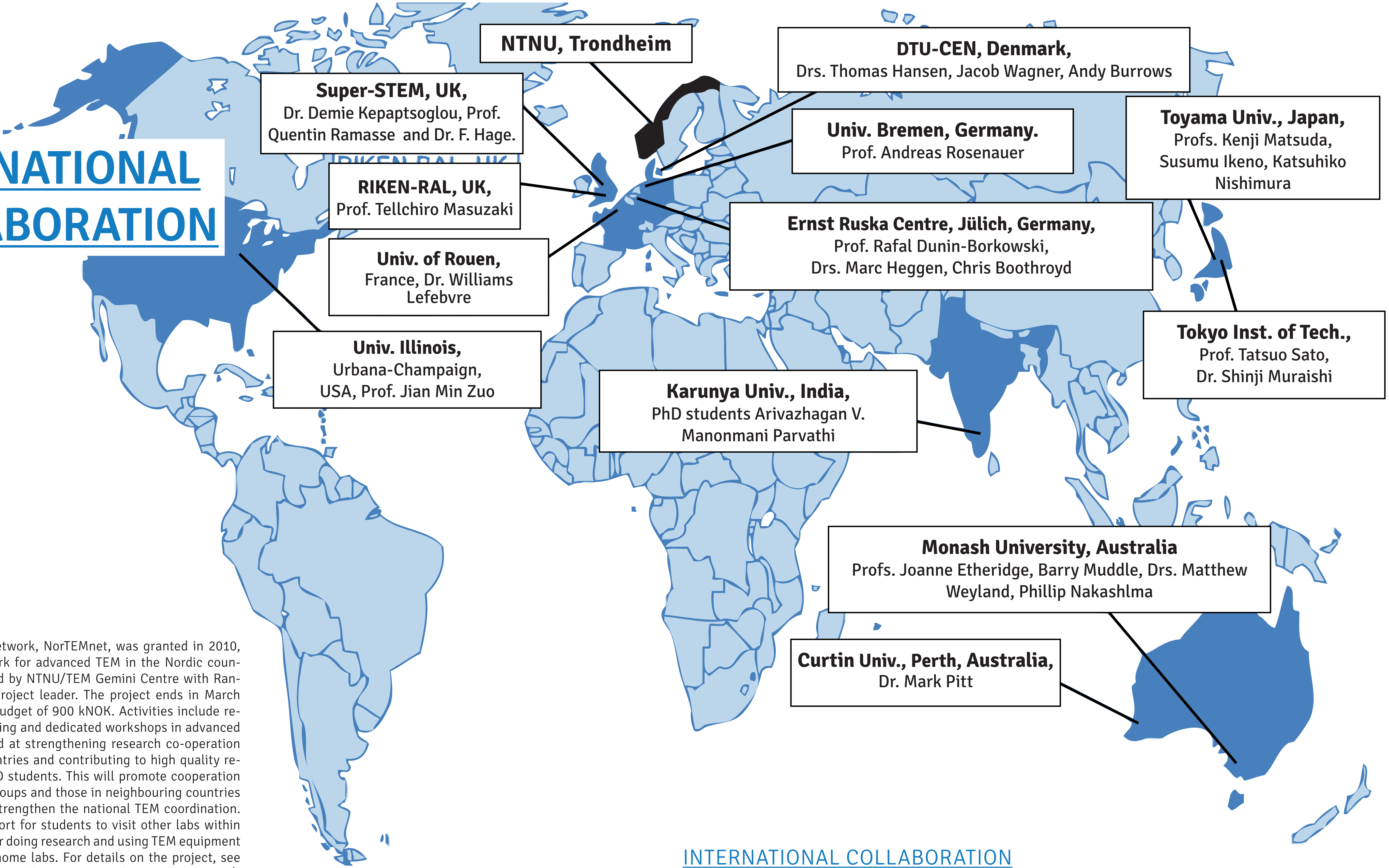
The Norwegian Centre for Transmission Electron Microscopy

NORTEM (the Norwegian Centre for Transmission Electron Microscopy) is a large scale infrastructure project supported by the Norwegian Research council and the three partners, NTNU, SINTEF and UiO, for investing in and running TEM infrastructure in Norway. The project has two nodes. The Trondheim node will have three TEMs from JEOL and the Oslo node two TEMs from FEI and JEOL. All instruments will be operative by the end of 2013. The official opening of NORTEM will take place in Oslo early 2014.

Gon'emon Kurihara, JEOL, signing the JEOL Competence Centre (JCC) contract.

President of SINTEF Unni Steinsmo, NTNUs pro-rector Kari Melby, and JEOLs president Gon'emon Kurihara.

INTERNATIONAL COLLABORATION



NORTEMNET

The NordForsk TEM network, NorTEMnet, was granted in 2010, and 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 ends in March 2014 and has 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, with lectures from several high profile researchers in the STEM community - Peter Nellist, and Lewys Jones from Oxford, Sara Bals from EMAT Antwerp, Miran Cêh from Jozef Stefan Institute Ljubljana and Knut Müller from University of Bremen. The workshop had 21 participants, mostly from the Nordic countries.

INTERNATIONAL COLLABORATION

As can be seen in the publication list, the TEM Gemini Centre has several international collaborators. During 2013 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). We visited many of the labs to use aberration corrected microscopes. 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!

USER STATISTICS IN 2013

There were in total 41 users of the microscopes in 2013. Of these are 4 SINTEF operators, who can give access to industry/third parties. There were 22 users from DP, 12 users from DMSE and 3 users from other departments.

2013 is a transition year. During 2013 the three NORTEM instruments have been taken into use. They are included in the statistics from the day they were in use, as follows; 2100 in use from 12. Aug, 2100F in use from 16. Dec and ARM200F in use from 11. Sept. The 2010F was taken out of use during fall 2013.

The first table below shows hours which are written down in the log book ("real hours spent"), for the three microscopes. The second table below shows an overview *of days the microscopes were used during the year.* Despite all work done to get the new TEMs online and build up a user base, the overall use was high throughout the year.

Use in hours

User	ARM200F	2100F	2100	2010F	CM30	2010
SINTEF	***57***	***28***	***6***	***253***	***41***	***0***
NTNU	***9***	***0***	***253***	***765***	***643***	***99***
Physics	*9*	*0*	*253*	*761*	*607*	*0*
IMT	*0*	*0*	*0*	*0*	*0*	*98*
Other departments	*0*	*0*	*0*	*4*	*36*	*1*
External	*0*	*0*	*0*	*0*	*0*	*0*
NTNU Teaching lab	*9*	*0*	*19*	*47*	*43*	*45*
NTNU, Setup/testing/demonstrations	*323*	*19*	*166*	*51*	*4*	*53*
Total use in hours	***398***	***47***	***444***	***1116***	***731***	***197***

Use in days

	ARM200F	2100F	2100	2010F	CM30	2010
Use in work days	*42*	*5*	*64*	*158*	*112*	*66*
Use in weekends	*0*	*0*	*9*	*19*	*23*	*0*
Total days in use	***42***	***5***	***73***	***177***	***135***	***66***
Downtime	*2*	*5*	*16*	*38*	*18*	*1*
Work days unused	*19*	*0*	*20*	*57*	*123*	*169*

Type	Project title	SINTEF	Postdoc	PhD	Master	Duration
SFI	*inGAP – Metal dusting, Fischer Tropsch*	*X*				*2006-2014*
BIP	*Nucleation control for optimised properties (KK)*	*X*		*1*		*2007-2012*
KMB	*SALSA –Sustainable aluminium surface applications*	*X*			*1*	*2007-2013*
KMB	*Norwegian-Japanese Al-Mg-Si Alloy Precipitation project*	*X*	*1*	*2*	*2*	*2009-2014*
FME	*Solar United*	*X*	*1*			*2009-2017*
KMB	*MoreAl*	*X*		*1*		*2009-2013*
RENERGI	*Nanosolar – Core-shell nanowires*			*1*	*4*	*2009-2013*
SO-project	*Quantitative STEM*			*1*		*2009-2013*
SUP	*Improvement (Innovation in light metals proc. ..)*	*X*	*1*			*2009-2014*
SO-project	*Nanowire-substrate interfacial characterization*			*1*	*1*	*2011-2015*
Renergi	*SolBiOpta*	*X*				*2010-2013*
eVITA	*MultiModAl- Multiscale modelling in Al*	*X*	*1*			*2011-2014*
SO-project	*Advanced TEM studies of oxide interfaces*	*X*		*1*	*1*	*2011-2015*
KOSK II	*Fundamental understanding of catalyst nanoparticles*	*X*	*X*			*2011-2013*
BIP	*RolEx (Al Alloy Development for Rolling and Extrusion)*	*X*		*1*	*1*	*2012-2016*
Climit	*pre-memCO2*	*X*	*1*			*2012-2014*
FRINATEK	*Fundamental investigations of precipitation in the solid state*	*X*	*1*	*1*		*2013-2017*
Energix	*High Efficiency Quantum Dot Intermediate Band Solar Cell*	*X*		*1*	*1*	*2013-2017*

ACTIVE PROJECTS IN 2013

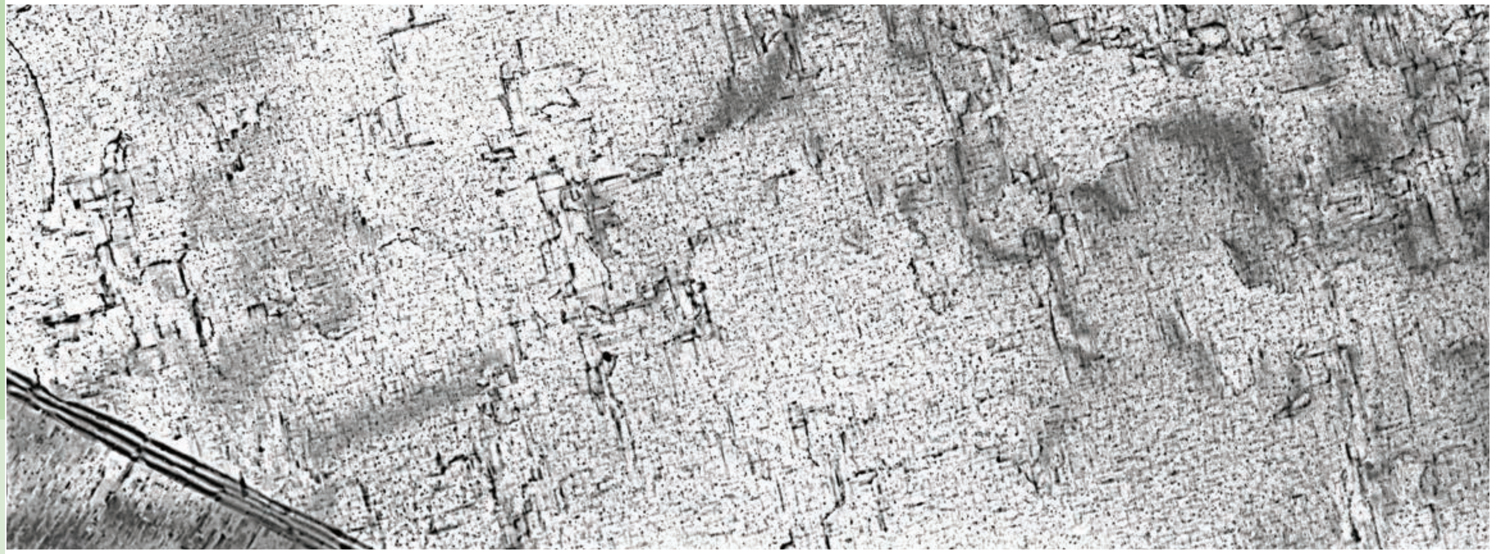
The table shows the larger projects connected to TEM within the Gemini Centre. They are listed by funding type, title and duration. Most of these projects involve both NTNU and SINTEF activity.

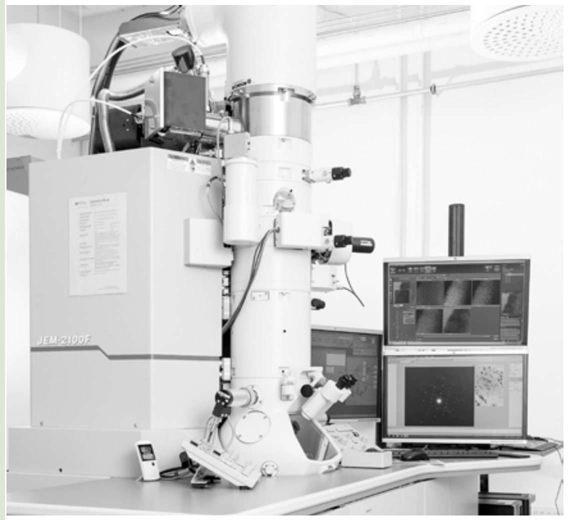
The listed numbers for Postdoc, PhDs and Master students are only those that are involved in TEM activities within those projects. A number of smaller projects, with direct industrial support, are not listed here.

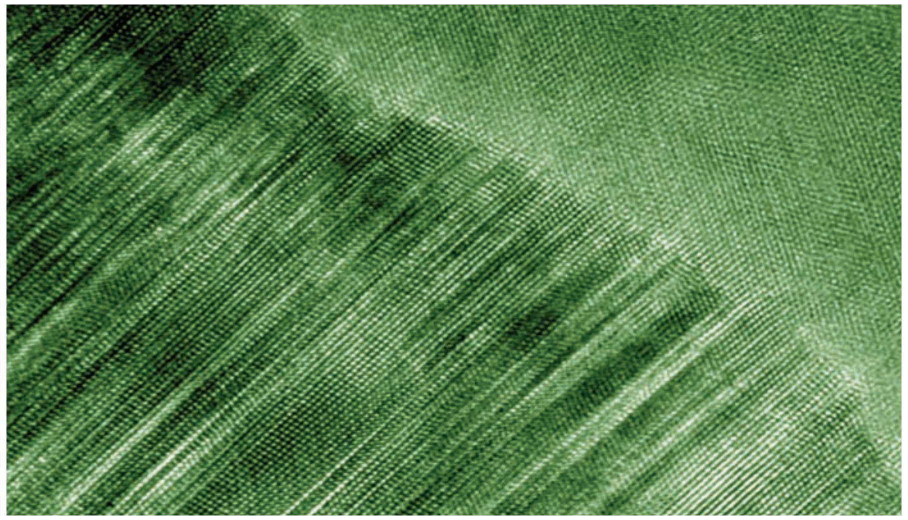
Overview of larger projects within TEM Gemini Centre which have been running in 2013 with positions in TEM. Numerous smaller projects, both academic and industrial have been carried out in addition.

SELECTED SCIENTIFIC PAPERS

IN 2013







APPLIED PHYSICS LETTERS **103**, 232111 (2013)

Compositional analysis of GaAs/AlGaAs heterostructures using quantitative scanning transmission electron microscopy

H. Kauko,[1,a)] C. L. Zheng,[2,a)] Y. Zhu,[2,3] S. Glanvill,[2] C. Dwyer,[2,4] A. M. Munshi,[5] B. O. Fimland,[5] A. T. J. van Helvoort,[1] and J. Etheridge[2,3,b)]
[1]*Department of Physics, Norwegian University of Science and Technology (NTNU), Trondheim, Norway*
[2]*Monash Centre for Electron Microscopy, Monash University, VIC 3800, Australia*
[3]*Department of Materials Engineering, Monash University, VIC 3800, Australia*
[4]*Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons, and Peter Grünberg Institute, Forschungszentrum Jülich, D-52425 Jülich, Germany*
[5]*Department of Electronics and Telecommunications, Norwegian University of Science and Technology (NTNU), Trondheim, Norway*

(Received 15 October 2013; accepted 14 November 2013; published online 5 December 2013)

We demonstrate a method for compositional mapping of $Al_xGa_{1-x}As$ heterostructures with high accuracy and unit cell spatial resolution using quantitative high angle annular dark field scanning transmission electron microscopy. The method is low dose relative to spectroscopic methods and insensitive to the effective source size and higher order lens aberrations. We apply the method to study the spatial variation in Al concentration in cross-sectioned GaAs/AlGaAs core-shell nanowires and quantify the concentration in the Al-rich radial band and the AlGaAs shell segments. © *2013 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4838556]

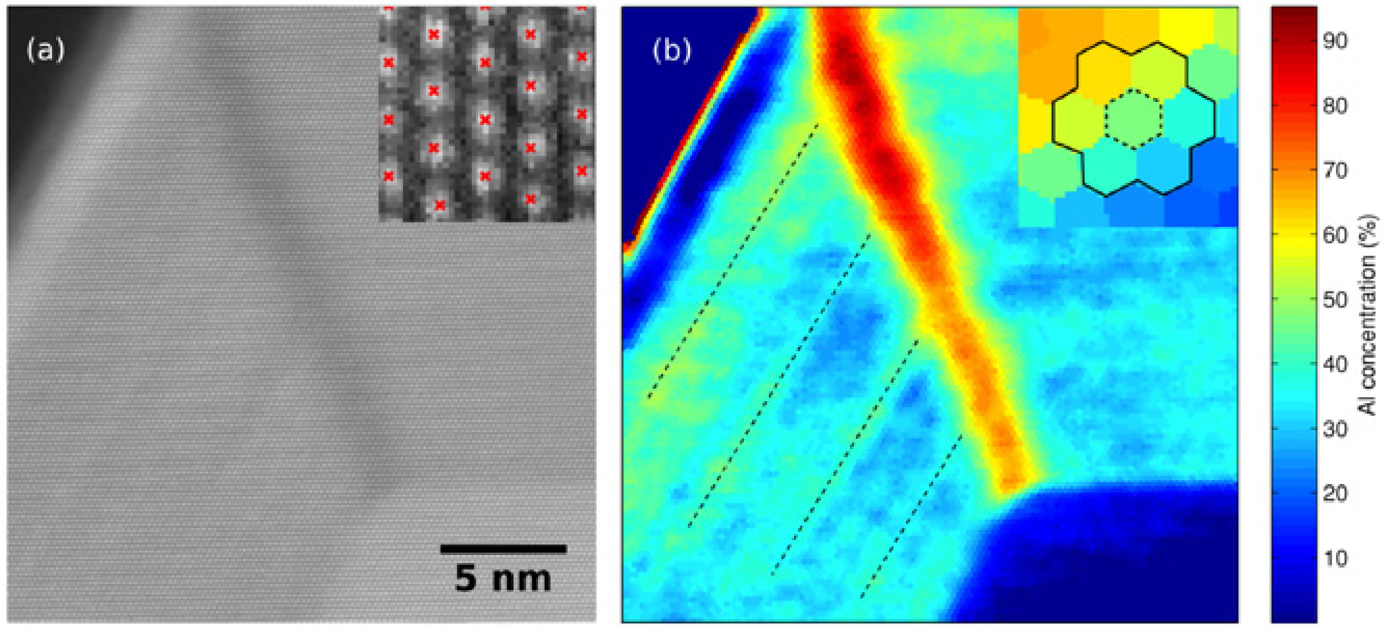


FIG. 3. (a) High-resolution HAADF STEM image of the upper left corner of the cross-section in Fig. 1(b). The inset shows a small magnified region, with the atomic column positions located. (b) The corresponding Al composition map. The black dashed lines in the AlGaAs shell mark the Al-rich lines parallel to the {110} planes. In the inset, taken from the start of the Al-rich band near the core, the unit cell region employed for averaging the intensity is depicted.

DOI 10.1007/s10853-013-7158-3

Atomic structure of hardening precipitates in an Al–Mg–Zn–Cu alloy determined by HAADF-STEM and first-principles calculations: relation to η-$MgZn_2$

Calin D. Marioara · Williams Lefebvre · Sigmund J. Andersen · Jesper Friis

Received: 14 November 2012 / Accepted: 11 January 2013 / Published online: 24 January 2013

Abstract The structures of two nanoscale plate precipitates prevalent at maximum strength and over-aged conditions in a 7449 Al–Mg–Zn–Cu alloy were investigated. Models derived from images of high angle annular dark field scanning transmission electron microscopy were supported by first-principles calculations. Both structures are closely linked to the η-$MgZn_2$ Laves phase through similar layers of a rhombohedral atomic subunit. The finest plate contains one such layer together with a layer of an orthorhombic unit. The second plate contains rhombohedral layers only, normally four, but rotated relatively to form different stacking variants, one of which may be likened to η. For both structures, the same atomic planes describe the main interface with Al. Both plates could be described in space group P3. The unit cells comprise interface and arbitrary numbers of {111}Al (habit) planes. Eight Al-planes were included in the first-principles calculations. The enthalpy indicates high layer/unit stability. The plate thickness can be understood by a simple mismatch formulation.

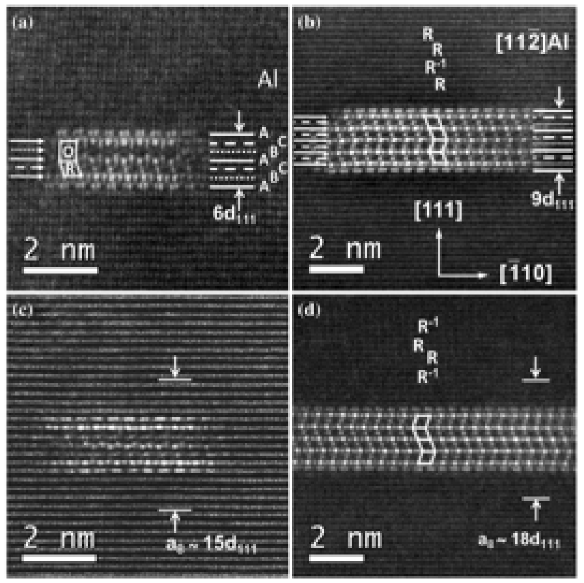


Fig. 1 ⟨112⟩ Al zone HAADF-STEM images of the two most abundant, coherent precipitates in conditions T6 (a, b) and T7 (c, d). Coordination system choice (b) applies to all images. *Arrows (left)*: three (a) and two (b) different planes define *inner parts*. *Lines (right)* indicate Al (ABC) stacking. Type 1 (a, c) and Type 2 (b, d) contain layers of units with rhombic (R) projections (a–b, d). Type 1 has one layer of orthorhombic units (O) and one R-layer (a). Layers connect to Al through identical solute-enriched {111}Al *interface* planes (*outer solid* 'A' *lines*) (a, b). Type 2 stacking variants (b, d) are overlaid with rhombs 'R' and 'R^{-1}' (R^{-1} rotated 180° about [111]Al). Unit cell parameters a_l with $l = 8$ integrated Al-planes used for first-principles refinements are indicated for both precipitates in (c, d)

IOP Publishing — Semiconductor Science and Technology

Semicond. Sci. Technol. **28** (2013) 115004 (13pp) — doi:10.1088/0268-1242/28/11/115004

The effects of Sb concentration variation on the optical properties of GaAsSb/GaAs heterostructured nanowires

J Todorovic[1], H Kauko[1], L Ahtapodov[2], A F Moses[2], P Olk[2], D L Dheeraj[2], B O Fimland[2], H Weman[2] and A T J van Helvoort[1,3]

[1] Department of Physics, Norwegian University of Science and Technology, NO-7491, Trondheim, Norway

[2] Department of Electronics and Telecommunications, Norwegian University of Science and Technology, NO-7491, Trondheim, Norway

E-mail: a.helvoort@ntnu.no

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Abstract

In this work we have investigated the variation of Sb concentration among and within zinc blende (ZB) GaAsSb inserts in wurtzite (WZ) GaAs bare-core and WZ GaAs/AlGaAs core–shell nanowires (NWs) grown by Au-assisted molecular beam epitaxy. The Sb concentration variation was related to the optical properties as determined by photoluminescence (PL). The NW structure and the Sb concentration were studied by transmission electron microscopy (TEM), energy dispersive x-ray spectroscopy (EDX) and quantitative high angle annular dark field scanning TEM (HAADF STEM). A clear trend relating the maximum Sb concentration with the insert length was observed: the longer the insert, the higher the Sb concentration. In addition, there are graded Sb concentration gradients both along and across the GaAsSb inserts. The influence of the Sb concentration variation on the PL emission from the GaAsSb inserts was investigated with correlated micro-PL and TEM-EDX on the same single NWs. Based on the PL results and the observed Sb concentration profiles, we propose a qualitative energy band diagram for a typical ZB GaAsSb insert in a WZ GaAs NW for the heterostructured NWs studied here. Type I transitions within the central region of the ZB GaAsSb inserts were found to dominate the insert-related PL emission. Weak type II transitions within the inserts due to the graded Sb concentration were observed as well. Using an existing empirical model, the Sb concentrations were additionally determined from the ground state PL energies (type I transition). For the average Sb concentration, the concentrations based on PL were in agreement with EDX and quantitative HAADF STEM results.

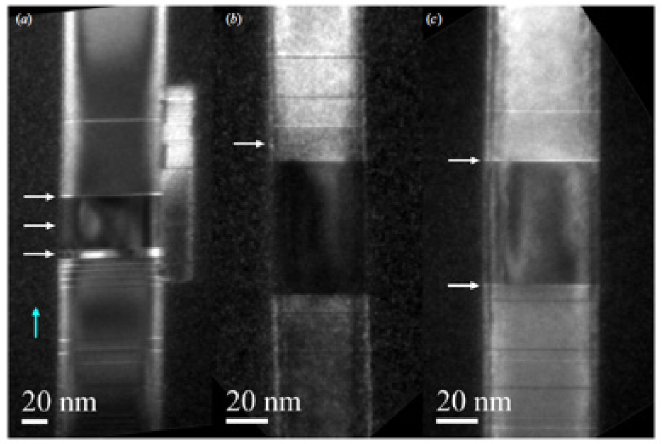

Figure 1. DF TEM images of bare-core NWs with a ZB GaAsSb insert: (*a*) 47 nm long with two twinned segments, 7 nm and 40 nm, and a 2 nm long twinned SF at the upper interface; (*b*) 64 nm long with a 4H polytype segment at the upper interface; (*c*) 59 nm long with twinned SFs at both insert interfaces. All three NWs have SFs both below and above the insert. The white arrows indicate special features such as: twinned segments in (*a*), 4H polytype in (*b*) and twinned SFs in (*c*). The blue (vertical) arrow in (*a*) indicates the $[0\,0\,0\,1]_{WZ}$ ($[1\,1\,1]_{ZB}$) growth direction for the NWs.

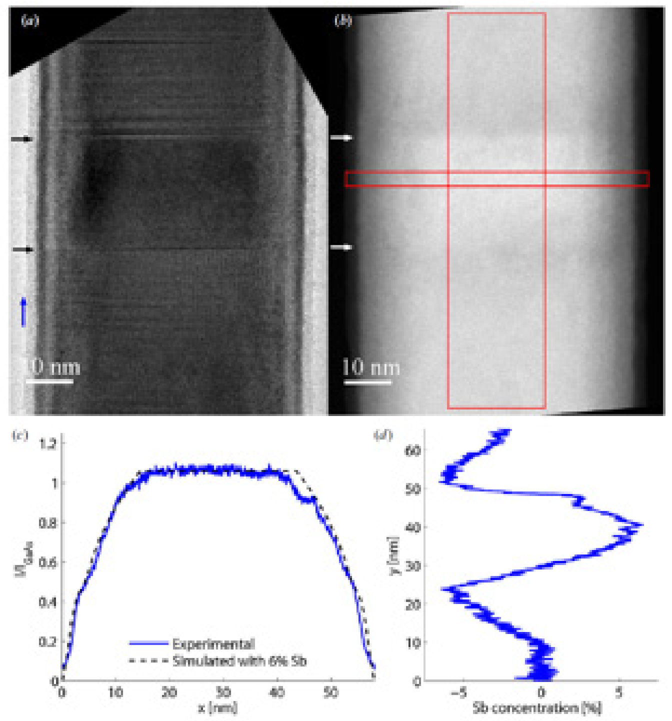

Figure 4. A bare-core NW with a 23 nm long ZB GaAsSb insert. (*a*) HRTEM image. The blue (vertical) arrow shows the growth direction. (*b*) HAADF STEM image. The black and white arrows in (*a*) and (*b*), respectively, mark the insert interfaces. (*c*) Relative HAADF STEM intensity profiles across the insert (*x*-direction, horizontal in (*a*, *b*)): the experimental profile (solid blue line) averaged over the 3 nm wide red box in (*b*), and the simulated profile (dashed black line) assuming a radially uniform Sb distribution with 6% Sb. (*d*) The Sb concentration profile along the insert (*y*-direction, vertical in (*a*, *b*)), obtained from HAADF STEM intensity profile averaged over the 20 nm wide red box in (*b*). Note that the negative values at the interfaces are not related to composition variations, but are due to dechanneling related to surface strain relaxation and/or the SFs [19, 33].

4124—VOLUME 44A, SEPTEMBER 2013 METALLURGICAL AND MATERIALS TRANSACTIONS A

The Effects of Low Cu Additions and Predeformation on the Precipitation in a 6060 Al-Mg-Si Alloy

TAKESHI SAITO, SHINJI MURAISHI, CALIN D. MARIOARA, SIGMUND J. ANDERSEN, JOSTEIN RØYSET, and RANDI HOLMESTAD

Effects of low Cu additions (≤0.10 wt pct) and 10 pct predeformation before aging on precipitates' microstructures and types in a 6060 Al-Mg-Si alloy have been investigated using transmission electron microscopy (TEM). It was found that predeformation enhances precipitation kinetics and leads to formation of heterogeneous precipitate distributions along dislocation lines. These precipitates were often disordered. Cu additions caused finer microstructures, which resulted in the highest hardness of materials, in both the undeformed and the predeformed conditions. The introduced predeformation led to microstructure coarsening. This effect was less pronounced in the presence of Cu. The precipitate structure was studied in detail by high-resolution TEM and high angle annular dark-field scanning TEM (HAADF-STEM). The Cu additions did not alter the respective precipitation sequence in either the undeformed or the predeformed conditions, but caused a large fraction of β'' precipitates to be partially disordered in the undeformed conditions. Cu atomic columns were found in all the investigated precipitates, except for perfect β''. Although no unit cell was observed in the disordered precipitates, the presence of a periodicity having hexagonal symmetry along the precipitate length was inferred from the fast Fourier transforms (FFT) of HRTEM images, and sometimes directly observed in filtered HAADF-STEM images.

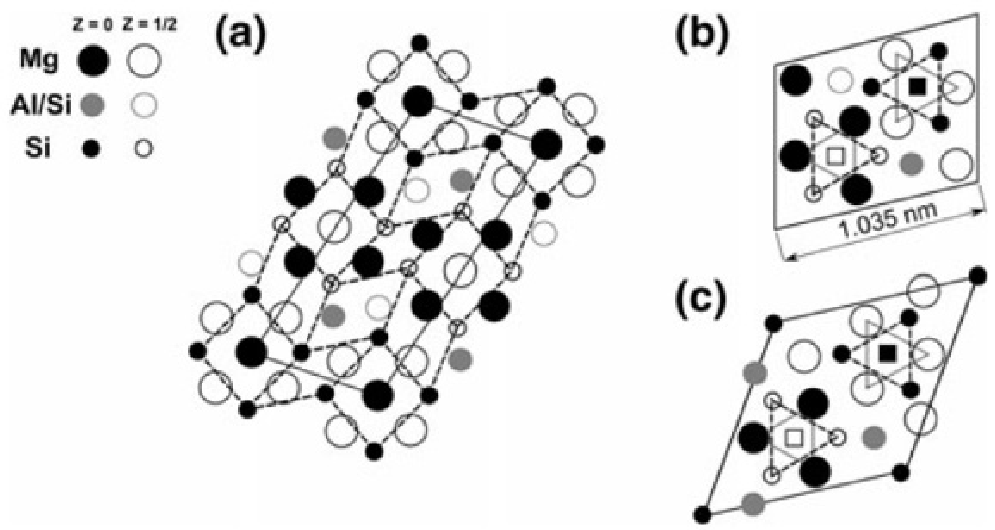

Fig. 10—Schematic images for unit cells for (*a*) β'',[2,3] (*b*) C phase,[27] and (*c*) Q phase precipitates,[13] drawn with the same scale. The common local arrangement around Cu atomic columns is indicated by dotted triangles on (b) and (c), which is identified for C, Q, Q′, and L phases.[14,27] This arrangement does not exist in the β'' structure, see (a). In (a), the Si atomic columns are connected by dashed lines.

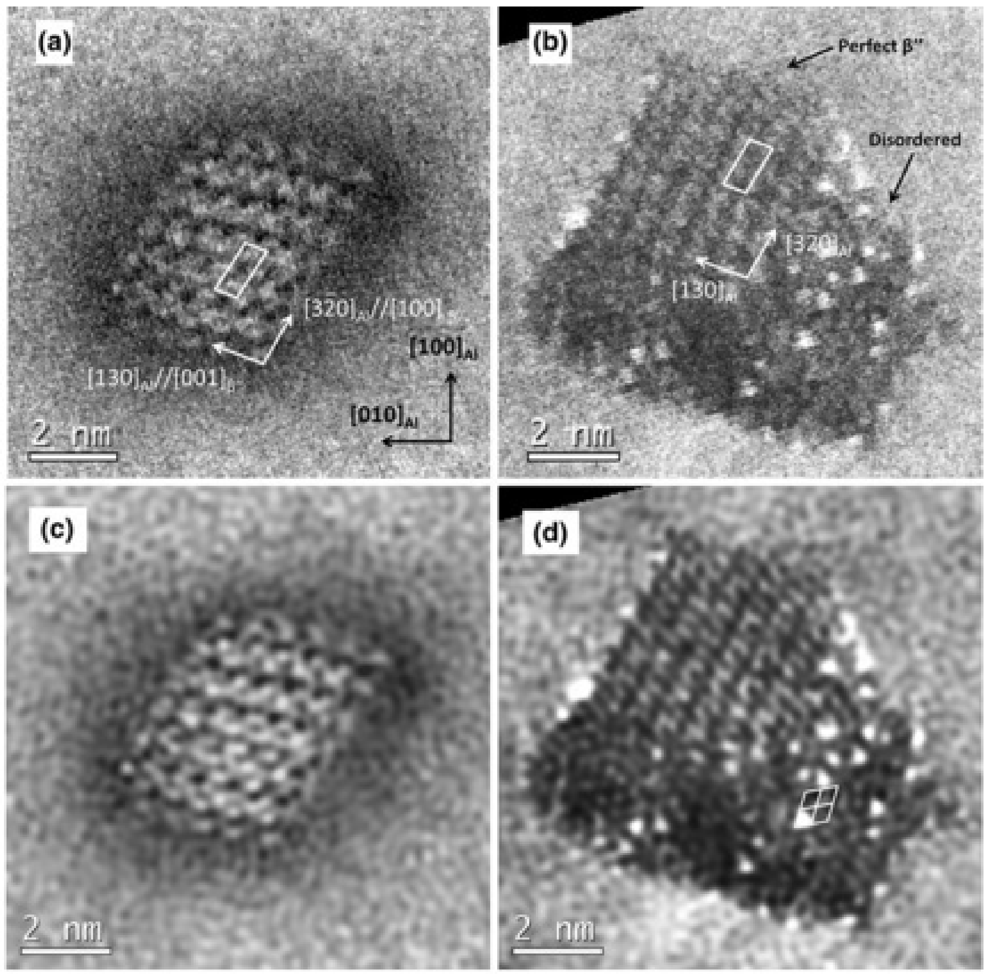

Fig. 8—HAADF-STEM images of precipitate cross sections taken along ⟨001⟩Al for the alloy containing 0.10 wt pct Cu in the undeformed condition. (*a*) Unprocessed image of a perfect β'', (*b*) unprocessed image of β''/disordered precipitate, (*c*) inverse FFT (IFFT)-filtered image of (a) and (*d*) IFFT-filtered image of (b). The IFFT filtering was applied to reduce noise using a circular band pass mask removing all periods shorter than 0.3 nm. A unit cell of the perfect β'' is shown by solid line in (a) and (b). Crystallographic orientation shown in (a) is common for all images. No specific atomic columns with high intensities are observed in the perfect β'' precipitate, or in the β'' part of the β''/disordered precipitate. However, strong contrasts corresponding to Cu-containing atomic columns are present in the disordered part of the β''/disordered precipitate, as well as at the precipitate–matrix interface. The periodicity of the hexagonal Si-network can be discerned in the disordered part, see the grid overlay in (d).

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Surface segregation of tin by heat treatment of dilute aluminium–tin alloys

Juan Tan [a], John Charles Walmsley [b], Børge Holme [c], Heidi Nordmark [b], Kemal Nisancioglu [a,*]

[a] Department of Materials Science and Engineering, Norwegian University of Science and Technology, NO-7491 Trondheim, Norway
[b] SINTEF Materials and Chemistry, NO-7465 Trondheim, Norway
[c] SINTEF Materials and Chemistry, NO-0314 Oslo, Norway

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ABSTRACT

The changes in the microstructure and surface morphology, caused by heat treatment, of binary AlSn model alloys, containing 30–1000 ppm tin were investigated with the purpose of understanding activation of aluminium by trace element tin in aqueous media. Specimens prepared by casting and cold-rolling in the laboratory were annealed for 1 h at 300 or 600 °C and quenched in water, and then characterised by various surface-analytical techniques. In the as-rolled condition, tin was in solid solution with aluminium. The microstructure and surface morphology in this condition were not different from those of pure aluminium. Significant segregation of liquid Sn occurred by annealing at 300 °C to the metal surface, caused by the combined effect of limited solubility and high mobility of Sn in Al at this temperature. Segregated Sn caused oxidation of aluminium during water quenching following heat treatment. The resulting oxide formation increased with increasing Sn content of the alloy from localised cases to extensive coverage by a 1.3 μm thick scale. Most of the Sn was homogenised in solid solution with Al by annealing at 600 °C for all Sn concentrations. The alloy containing 1000 ppm Sn still formed a thick oxide as a result of quenching in water after heat treatment, caused by enrichment of Sn at the metal–oxide interface by dealloying of Al.

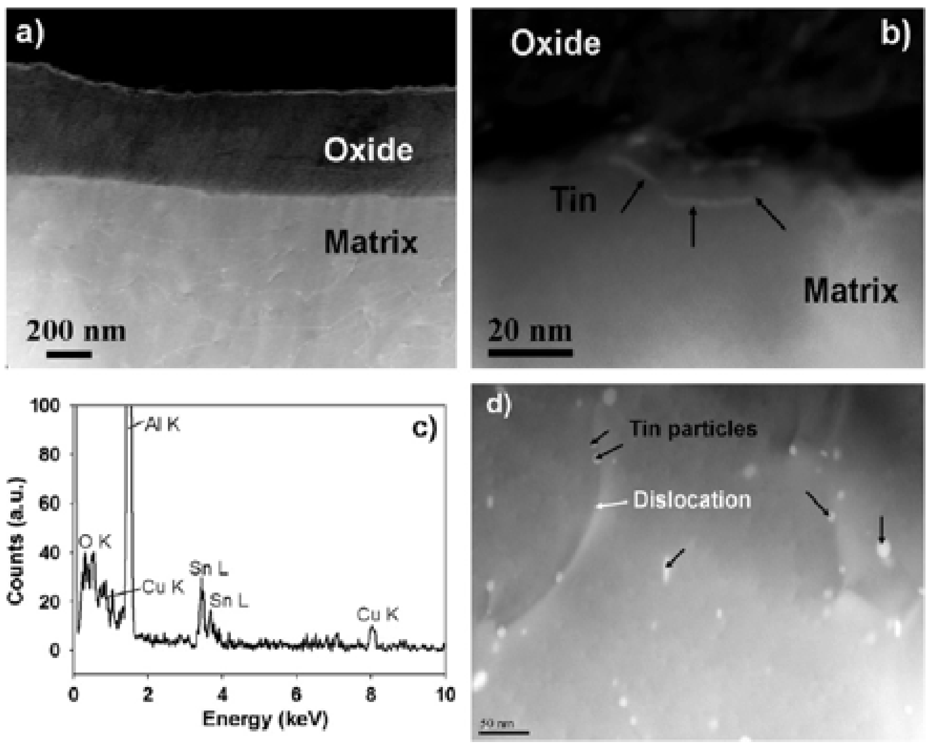

Fig. 10. (a) STEM dark field cross-sectional image of alloy AlSn1000, annealed for 1 h at 600 °C followed by water quenching. (b) Higher magnification image of the metal–oxide interface. (c) X-ray EDS spot analysis of the interfacial film in (b). (d) Image of the bulk metal close to the surface.

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Dispersoid strengthening in AA3xxx alloys with varying Mn and Si content during annealing at low temperatures

Astrid Marie F. Muggerud [a,*], Eva Anne Mørtsell [a], Yanjun Li [b], Randi Holmestad [a]

[a] Department of Physics, University of Science and Technology, NTNU, N-7491 Trondheim, Norway
[b] SINTEF Materials and Chemistry, N-7465 Trondheim, Norway

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ABSTRACT

The precipitation behaviour of dispersoids in Direct Chill (DC)- cast 3xxx alloys with varying compositions of manganese and silicon has been studied during isothermal annealing at low temperatures. The evolution of density, size and volume fraction of the dispersoids has been quantitatively studied by Transmission Electron Microscopy (TEM) and electrical conductivity measurements. Moreover the influence of precipitation of dispersoids on hardness and tensile strength of the alloys is systematically investigated. A clear dispersion hardening effect from dispersoids is revealed and the hardening effect increases with increasing Mn and Si contents in the alloys. The hardening effect from dispersoids is discussed in light of the Orowan mechanism.

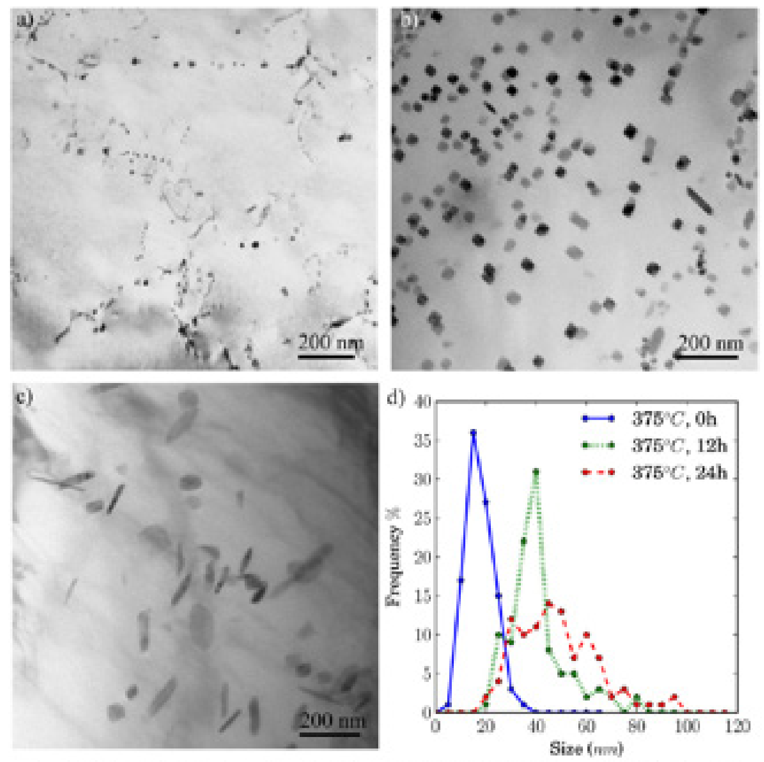

Fig. 2. Evolution of dispersoid morpholgy in alloy D2 during annealing at 375 °C. (a) As-heated to 375 °C, (b) 12 h annealing, (c) 24 h annealing, and (d) histogram showing the size distribution of dispersoids (equivalent diameter) after annealing at 375 °C for 0, 12 and 24 h.

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Ru@Pt core–shell nanoparticles for methanol fuel cell catalyst: Control and effects of shell composition

Navaneethan Muthuswamy [a], *Jose Luis Gomez de la Fuente* [b], *Dung T. Tran* [c], *John Walmsley* [c], *Mikhail Tsypkin* [b], *Steinar Raaen* [c], *Svein Sunde* [b], *Magnus Rønning* [a], *De Chen* [a,*]

[a] *Department of Chemical Engineering, Norwegian University of Science and Technology (NTNU), 7491 Trondheim, Norway*

[b] *Department of Materials Science and Engineering, Norwegian University of Science and Technology (NTNU), 7491 Trondheim, Norway*

[c] *Department of Physics, Norwegian University of Science and Technology (NTNU), 7491 Trondheim, Norway*

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ABSTRACT

The present work presents a method to encapsulate pre-synthesised Ru nanoparticles (NPs) by Pt using a polyol method without capping agents at various pH values (6, 7, 8 and 10). The structural and surface properties of the catalysts were characterised using X-ray diffraction, transmission electron microscopy, CO stripping, and energy-dispersive X-ray spectroscopy. The studies suggest that the pH during encapsulation of Ru by Pt plays an important role in controlling of shell composition. A core shell catalyst with an alloy shell was obtained at a pH of 6, whereas a monometallic Pt shell was obtained at a pH of 10. The core shell catalysts gave higher steady-state current for methanol oxidation: 10-fold higher for alloy shells and 5-fold higher for Pt-enriched shells compared to the pure Pt catalyst. It is suggested that the highest catalytic enhancement of the core shell catalysts is obtained through the bi-functional character that dominates the alloy shells rather than the ligand-effect-promoted Pt-enriched shells.

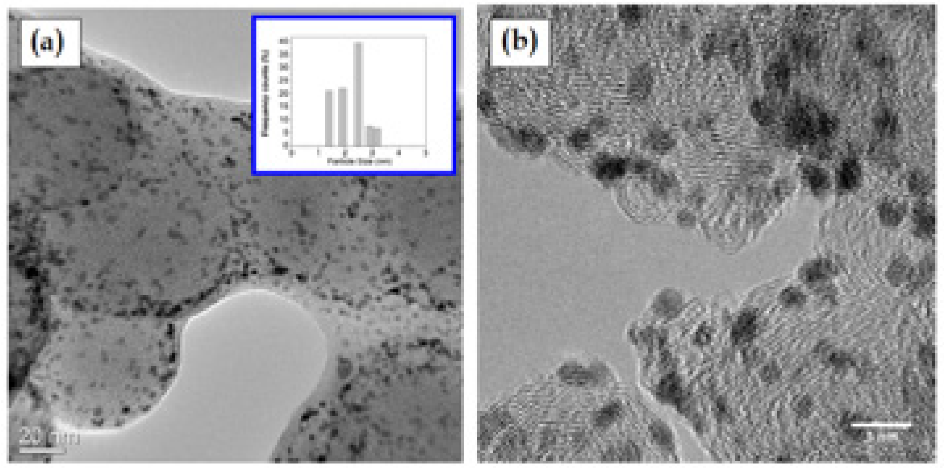

Fig. 2 – TEM images of the Pt/C catalyst. (a) Low resolution and (b) high resolution images. The insert in (a) is Pt distribution histograms.

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TEM study of β′ precipitate interaction mechanisms with dislocations and β′ interfaces with the aluminium matrix in Al–Mg–Si alloys

Katharina Teichmann [a], *Calin D. Marioara* [b], *Sigmund J. Andersen* [b], *Knut Marthinsen* [a,*]

[a] *Norwegian University of Science and Technology, Trondheim, Norway*
[b] *SINTEF Materials and Chemistry, Trondheim, Norway*

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ABSTRACT

The interaction mechanisms between dislocations and semi-coherent, needle-shaped β′ precipitates in Al–Mg–Si alloys have been studied by High Resolution Transmission Electron Microscopy (HRTEM). Dislocation loops appearing as broad contrast rings around the precipitate cross-sections were identified in the Al matrix. A size dependency of the interaction mechanism was observed; the precipitates were sheared when the longest dimension of their cross-section was shorter than approximately 15 nm, and looped otherwise. A more narrow ring located between the Al matrix and bulk β′ indicates the presence of a transition interface layer. Together with the bulk β′ structure, this was further investigated by High Angle Annular Dark Field Scanning TEM (HAADF-STEM). In the bulk β′ a higher intensity could be correlated with a third of the Si-columns, as predicted from the published structure. The transition layer incorporates Si columns in the same arrangement as in bulk β′, although it is structurally distinct from it. The Z-contrast information and arrangement of these Si-columns demonstrate that they are an extension of the Si-network known to structurally connect all the precipitate phases in the Al–Mg–Si(–Cu) system. The width of the interface layer was estimated to about 1 nm.

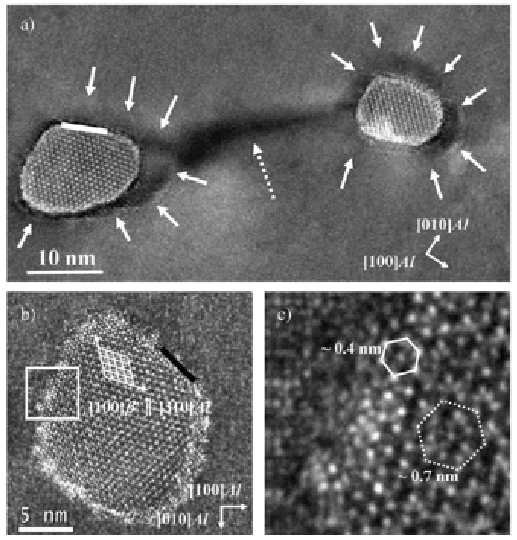

Fig. 1 – HRTEM images of cross-sections of β′-precipitates in <001> Al projection. In a) two β′-precipitates from the deformed condition of alloy C are lying along the same dislocation line — the dark contrast indicated by the dotted arrow. The solid arrows point to the surrounding contrast associated with dislocation loops. A bright ring can be observed on the rim of the precipitate cross sections. A small section of such a ring is indicated by the thick white line on the upper part of the left-side precipitate. b) Image of a β′-precipitate in the un-deformed condition of alloy A. The superposed black solid line indicates part of a bright contrast ring on the precipitate rim, similar to what has been observed in a). The white-line square delimits a part that is shown magnified in c). It can be observed that the hexagonal ≈0.7 nm β′ periodicity (indicated with white dotted lines) in the bulk changes to hexagonal ≈0.4 nm periodicity (indicated with white full lines) in the bright ring region. The overlay in b) also shows a grid with 4×4 unit cells of β′.

PUBLICATIONS 2013

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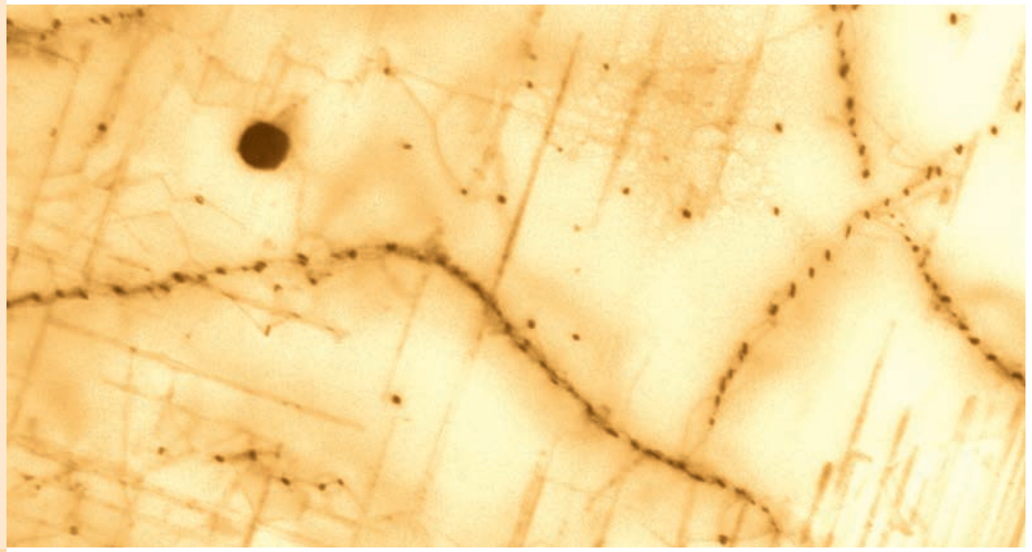

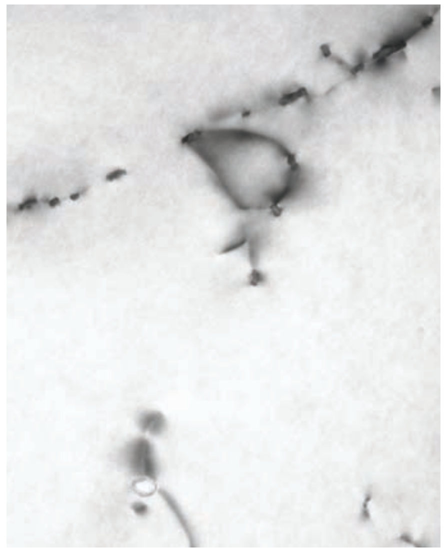

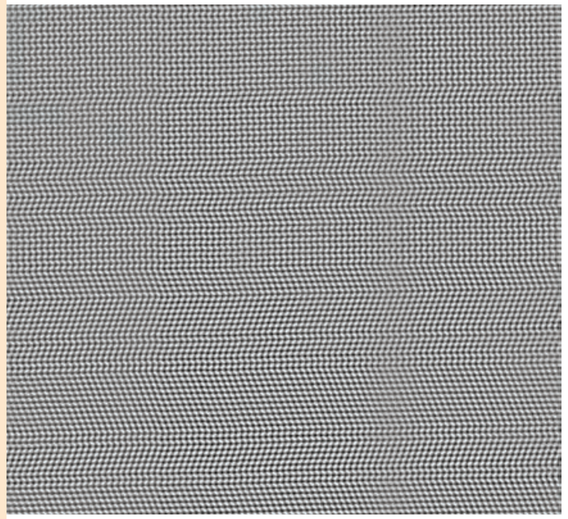

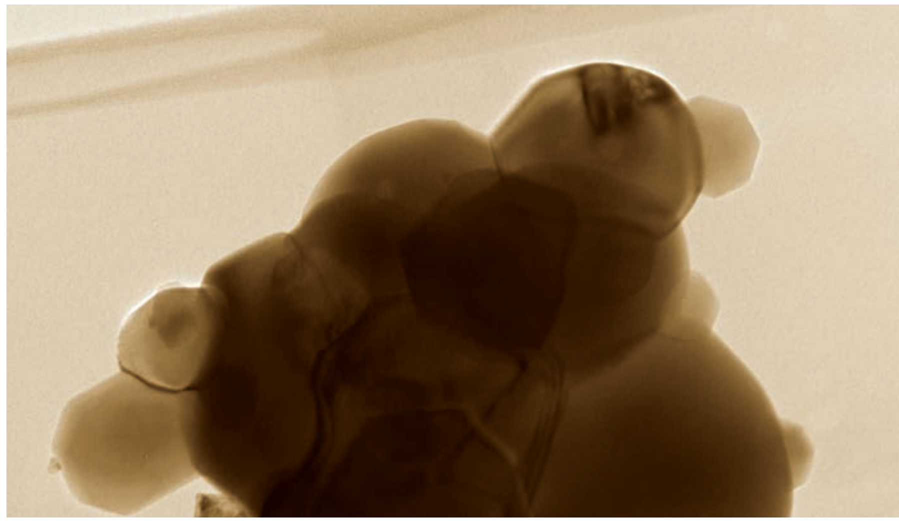

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Postdocs Flemming Ehlers, Ruben Bjørge and Dung Trung Tran working on phase simulations, image processing and data analysis in our computer room.

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L. Ahtapodov, H. **Kauko**, A. M. Munshi, B.-O. Fimland, A. T. J. **Van Helvoort** and H. Weman, "Optical properties of self-catalyzed GaAs Nanowires with axial GaAsSb inserts", Nanowires-2013, Rehovot, Israel, 12-15 November 2013. [Poster]

L. Ahtapodov, J. **Todorovic**, J. F. Reinertsen, D. Dasa Lakshmi Narayana, A. M. Munshi, **A**. **T**. **J**. **Van Helvoort**, B.-O. Fimland and H. Weman, "Optical Characterization of Single Semiconductor Nanowires", MYFAB&NORFAB Meetting, Uppsala, Sweden, 17-18 April 2013. [Talk]

K. M. Beckwith, M. K. **Nord**, V. T. **Fauske** and A. T. J. **Van Helvoort**, "Nanotools - A Lab Course Based on Modern Characterization Tools", Scandem 2013, Copenhagen, Denmark, 10-14 June 2013. [Talk]

D. L. N. Deeraj, A. M. Munshi, V. T. **Fauske**, D. C. Kim, A. T. J. **Van Helvoort**, B.-O. Fimland and H. Weman, "Epitaxial growth and characterization of vertical III-V semiconductor nanowires on graphene", 7th Nanowire Growth Workshop, Lausanne, Switzerland, 10-12 June 2013. [Poster]

D. L. N. Deeraj, A. M. Munshi, V. T. **Fauske**, D. C. Kim, A. T. J. **Van Helvoort**, B.-O. Fimland and H. Weman, "Epitaxial growth of vertical III-V semiconductor nanowires on graphene", Graphene2013, Bilbao, Spain, 23-26 April 2013. [Poster]

F. J. H. **Ehlers**, S. Dumoulin and R. **Holmestad**, "3D hybrid atomistic modeling of '' in Al–Mg–Si: putting the full coherency of a needle shaped precipitate to the test", 2nd World Congress on Integrated Computational Materials Engineering, Salt Lake City, Utah, USA, 7-11 July 2013. [Talk]

V. T. **Fauske**, D. C. Kim, A. M. Munshi, D. Dasa Lakshmi Narayana, B.-O. Fimland, H. Weman and A. T. J. **Van Helvoort**, "In-situ electrical and structural characterization of individual GaAs nanowire", EMAG2013, York, England, 2-6 September 2013.

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R. **Holmestad**, "Nanoscale precipitates in 6xxx aluminium alloys", Thermec 2013, Las Vegas, USA, 1-6 December 2013. [Talk]

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R. **Holmestad**, J. **Walmsley**, B. G. **Soleim**, P. E. **Vullum** and A. T. J. **Van Helvoort**, "NORTEM - new possibilities for atomic scale structure characterization", 8th Nanolab usermeeting, Trondheim, Norway, 12 November 2013. [Talk]

J. Huh, D. C. Kim, H. Yun, A. M. Munshi, D. Dasa Lakshmi Narayana, H. **Kauko**, A. T. J. **Van Helvoort**, S. W. Lee, B.-O. Fimland and H. Weman, "Electrical Characteristics of Individual GaAsSb Nanowires Grown by Molecular Beam Epitaxy", 8th Nanolab usermeeting, Trondheim, Norway, 12 November 2013. [Talk]

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A. M. Munshi, D. Dasa Lakshmi Narayana, V. T. **Fauske**, A. **Van Helvoort**, B.-O. Fimland and H. Weman, "Limitations of self-catalytic growth in controlling diameter of GaAs nanowires on Si substrates", 7th Nanowire Growth Workshop, Lausanne, Switzerland, 10-12 June 2013. [Poster]

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M. K. **Nord**, J. E. Boschker, P. E. **Vullum**, T. Tybell and R. **Holmestad**, "Study of electronic reconstruction in $La_{0.7}Sr_{0.3}MnO_3/SrTiO_3$ thin film interface using Transmission Electron Microscopy", 4th annual workshop arranged by the Norwegian PhD Network on Nanotechnology for Microsystems, Bergen, Norway, 17-19 June 2013. [Talk]

D. S. G. Panditha Vidana, H. J. Venvik and J. **Walmsley**, "Investigation of the Initial Stage of Metal Dusting Corrosion in the Conversion of Natural Gas to Synthesis Gas", NACE Corrosion 2013 - International Conference & Expo, Orlando, Florida, USA, 17-21 March 2013. [Talk]

T. **Saito**, "Effect of trace elements (Cu and Zn) on 6xxx series aluminum alloys", BILAT meeting in Toyama 2013, Toyama, Japan, 4-7 June 2013. [Talk]

T. **Saito**, W. Lefebvre, C. D. **Marioara**, S. J. Andersen and R. **Holmestad**, "Cu and Zn atomic columns of precipitates in Al-Mg-Si alloys investigated by aberration-corrected scanning transmission electron microscopy", MC2013, Regensburg, Germany, 25-30 August 2013. [Poster]

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G. Singh, A. T. J. **Van Helvoort**, S. Bandyopadhyay, S. Volden, J.-P. Andreassen and W. Glomm, "Shape and Size Controlled Synthesis of Metal Nanostructures", 8th Nanolab usermeeting, Trondheim, Norway, 12 November 2013. [Talk]

S. **Wenner**, C. D. **Marioara**, S. J. Andersen and R. **Holmestad**, "Precipitation in Al-Mg-Si alloys with Ca additions", THERMEC 2013, Las Vegas, USA, 2-6 December 2013. [Poster]

S. **Wenner**, C. D. **Marioara**, J. Røyset and R. **Holmestad**, "Transmission electron microscopy and muon spin relaxation studies of precipitation in Al–Mg–Si alloys", BILAT meeting 2013, Toyama, Japan, 4-7 June 2013. [Talk]

PhD theses in 2013

Hanne Kauko, "Quantitative scanning transmission electron microscopy studies on heterostructured GaAs nanowires".

Master/project theses within TEM Gemini Centre

Ørjan Berntsen, "Investigation of Co_2AlO_4/CeO_2 catalyst for N_2O abatement using electron microscopy", (Project thesis (DP)).

Maximilian Erlbeck, "Exploring FIB for NW circuit repair", (project (DP)).

Sethulakshmy Jayakumary, "The effects of Be doping on the structure of Ga and Au-assisted GaAs-based heterostructured semiconductor nanowires", (Master thesis (DP)).

Andrea Klubicka, "TEM study of GaAs/GaAsSb core-shell Nanowires", (Master thesis (DP)).

Julie Stene Nilsen, "The effect of the V/III ratio on the structural and optical of self-catalysed GaAs/AlGaAs core/shell nanowires", (project (DP)).

Ingrid Snustad, "Selective examination of optically and structurally separable parts within GaAs/AlGaAs core-shell nanowires by micro-photoluminescence and transmission electron microscopy", (Master thesis (DP))

Popular science contributions/Media

Flemming Ehlers and Randi Holmestad, "Prøver å forstå styrken til aluminium", Teknisk Ukeblad, 17. April 2013.

Randi Holmestad and Bjørn Gunnar Soleim, "Nytt supermikroskop", Teknisk Ukeblad, 5. September 2013.

Gemini.no; forskningsnytt fra NTNU og SINTEF: http://gemini.no/2013/09/supermikroskop/

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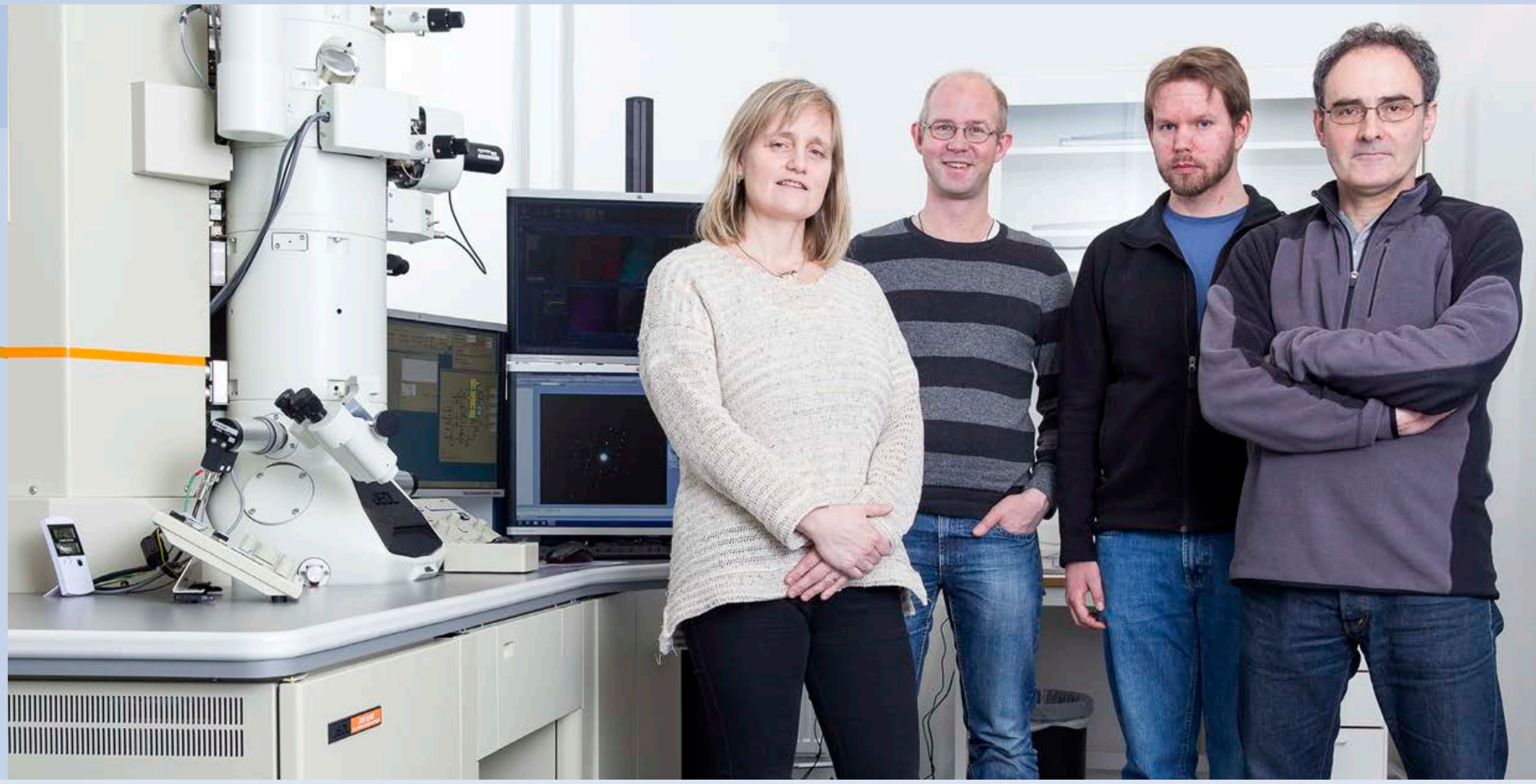
Contacts

Professor Randi Holmestad
Dept. of Physics, NTNU,
randi.holmestad@ntnu.no
Phone +4748170066

Senior scientist John C. Walmsley
SINTEF Materials and Chemistry
john.walmsley@sintef.no
Phone +4798283914

Senior engineer Bjørn Gunnar Soleim
Dept. of Physics, NTNU,
Bjorn.soleim@ntnu.no
Phone +4773590714

Associate professor Ton van Helvoort
Dept. of Physics, NTNU,
a.helvoort@ntnu.no
Phone +47 73593637



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