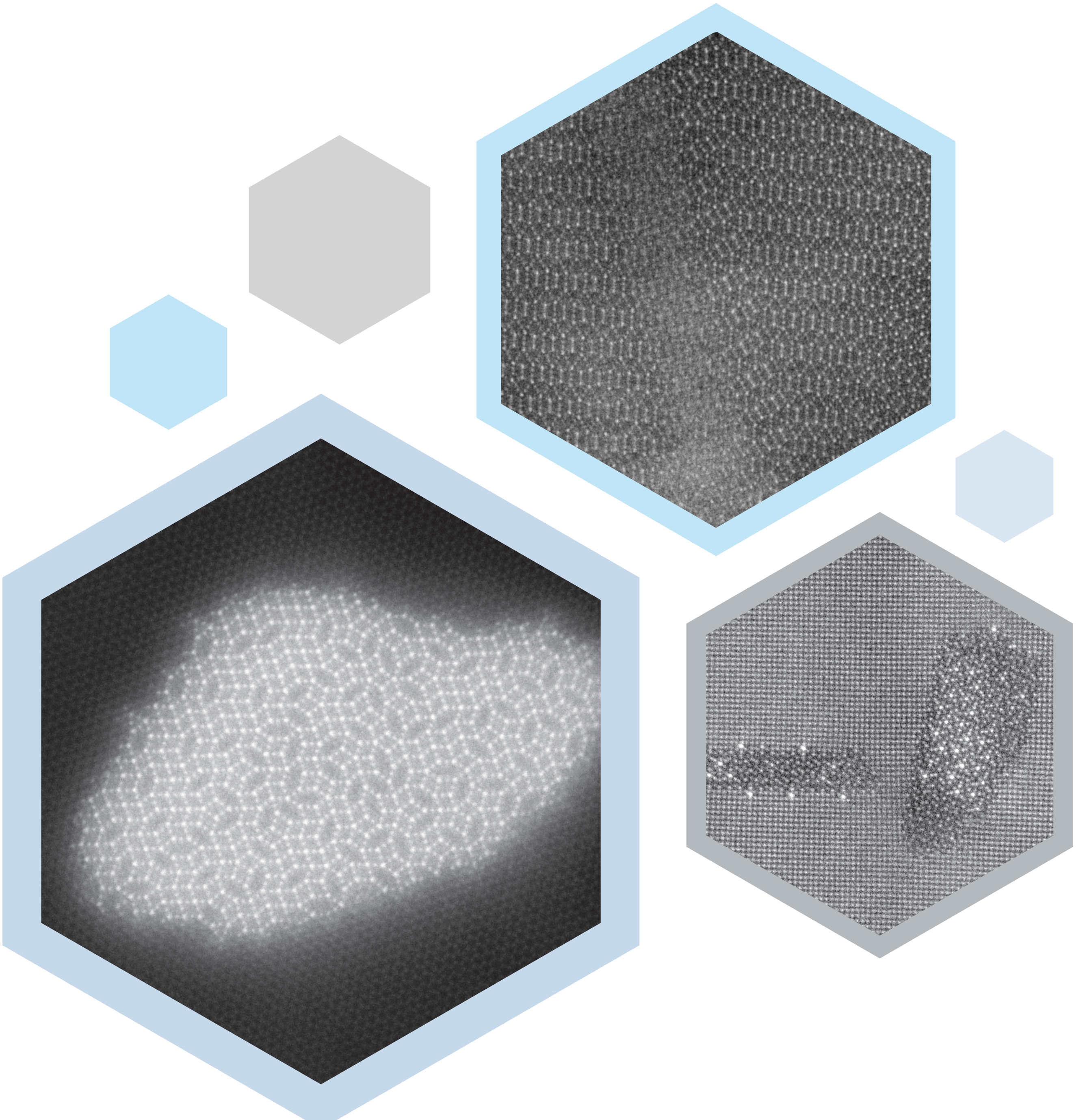
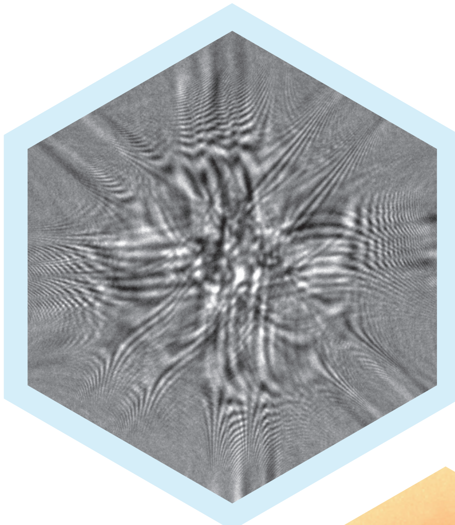


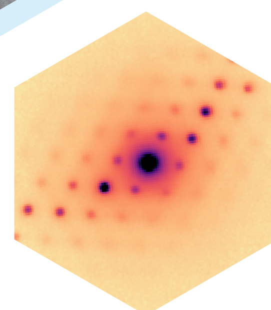
ANNUAL REPORT 2018

TEM GEMINI CENTRE

Department of Physics, NTNU;
Department of Materials Science and Engineering, NTNU;
Materials Physics, SINTEF Industry

CONTENT





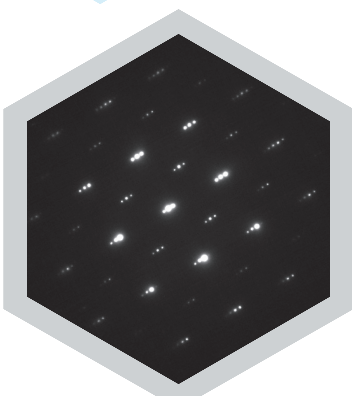
Graphic design: Tina Bergh.

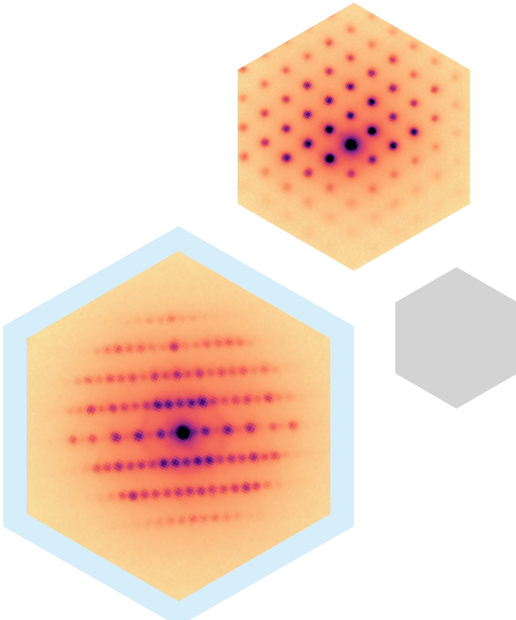
All images in this report, including all TEM images, are taken by members at the TEM Gemini Centre using local equipment.

HAADF-STEM image of a T-($Al_{20}Cu_2Mn_3$) phase dispersoid exhibiting internal faulting by Jonas Kristoffer Sunde.

HAADF-STEM image aquired using SmartAlign of an ordered $MgZn_2$ quasi-crystal in a Mg-Zn alloy by Adrian Lervik.

HAADF-STEM image of two nanoscale precipitates in a 6060 aluminium alloy by Adrian Lervik. These contribute to the hardness of aluminium alloys.





THE TEM GEMINI CENTRE

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

In 2009 the Department of Materials Science and Engineering (DMSE) at NTNU was included in the Centre. The same constellation was last renominated in November 2018 for a new period of 4 years.

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

THE GEMINI CENTRE CONCEPT

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

"Global excellence together".

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

TEM Gemini Centre renomination in November 2018. From left Gunnar Bovim (rector NTNU), Randi Holmestad (professor NTNU), Ragnar Fagerberg (SINTEF) and Alexandra Bech Gjørv (president and CEO SINTEF).

INTRODUCTION

2018 has been a good year for the TEM Gemini Centre. We have continued to systematically build up routines, extend and further apply our competence around the NORTEM Trondheim node instruments that were installed in 2013. It is a big responsibility and long-term project to establish and have an effective role within Norway and in the NTNU/SINTEF landscape for such expensive and advanced equipment. It is therefore very satisfying to see the consistent trend of increasingly high levels of use and quality of scientific and educational output achieved during the last years. The Centre was renewed for 4 new years in October 2018. Furthermore, we organized the annual European ARM user meeting in Trondheim in June. Participation and funding of the EU Horizon 2020 network project ESTEEM3 have been a highlight in 2018 with huge impact on the years to come.

The total cost model for lab infrastructure introduced by NTNU has now been fully implemented for the TEM lab for two years. NTNU owns and runs the infrastructure, with SINTEF as an important user. We are working on defining NORTEMs national role and securing appropriate access for research groups who need TEM. The NORTEM project is a partnership between NTNU, UiO, and SINTEF, financed by the Research Council of Norway and the partners in 2012. In 2018 we submitted an application for a follow-up project NORTEM II, which should keep the infrastructure at the leading edge by investing in new columns and upgrading detectors. The decision if our application is granted will be announced in June 2019.

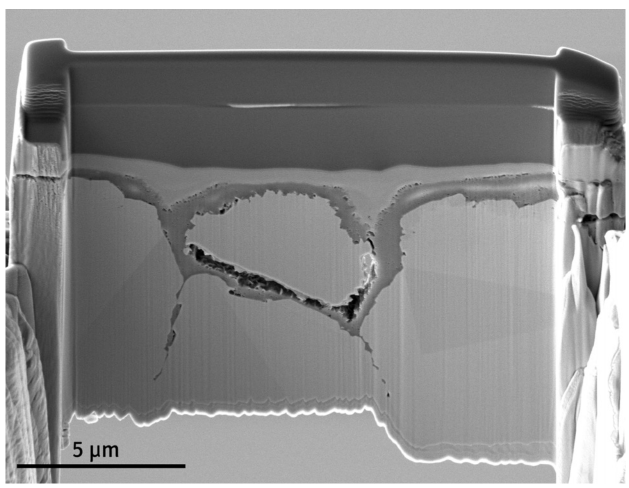
The Gemini Centre participates in a broad range of projects, including national, public, industrial and EU funding ones. The Centre is involved in three long-term SFI projects – Centre of advanced structural studies (CASA), Sustainable innovations for automated manufacturing of multi-material products (SFI-Manufacturing) and Industrial catalysis science and innovation for a competitive and sustainable process industry (iCSI). Furthermore, the TEM Gemini Centre is central in two ongoing KPN projects on aluminium with Norwegian aluminium industry – FICAL and AMPERE. In addition, we have extended the range of research areas and collaborators, which led to an increase in the number of various projects that utilize the infrastructure. The INTPART project with Japanese aluminium industry and academia was renewed for 4 new years in 2018.

As documented in this report, the Centre had 38 active hands-on users/operators, and served around 85 different projects, whose results have contributed to 48 journal publications (plus 8 in press) in 2018. Of these, 29 have co-authors from both NTNU and SINTEF. 18 of the publications have international co-authors. TEM Gemini Centre publications are found in a broad range of journals and cover a spectrum of topics, showing how generic TEM is. In addition, 7 Master candidates were educated with TEM as a substantial part of their theses in 2018. A positive trend is that the infrastructure gets more NTNU users from outside the Physics department. Three NTNU courses, with a total of approximately 140 students, used the facility. The yearly TEM introduction course was organized in September and had 30 participants. We had group meetings with presentations almost every week. Many guided tours for high school students and visitors to the microscopes took place. As seen from the publication list at the end, most members of the TEM Gemini Centre participated in international conferences and meetings in 2018.

Looking ahead into 2019 participating in the new EU Horizon 2020 INFRAIA initiative ESTEEM3 will be a central focus point. ESTEEM3 is a European Network for Electron Microscopy among the leading European TEM groups integrating activity for electron microscopy, and providing access, facilitating and extending transnational access services. We are also very satisfied that the Department of Physics will announce a new permanent position and an additional affiliation position within TEM in 2019. In addition, the Faculty of Natural Sciences (NV) and the Department of Physics will finance a dedicated direct detection detector for electron diffraction.

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 2018. For more details, see our home page: ntnu.edu/geminicentre/tem

- TEM Gemini Centre management, February 2019.



SEM image of a TEM lamella prepared by FIB lift-out by S. Wenner.

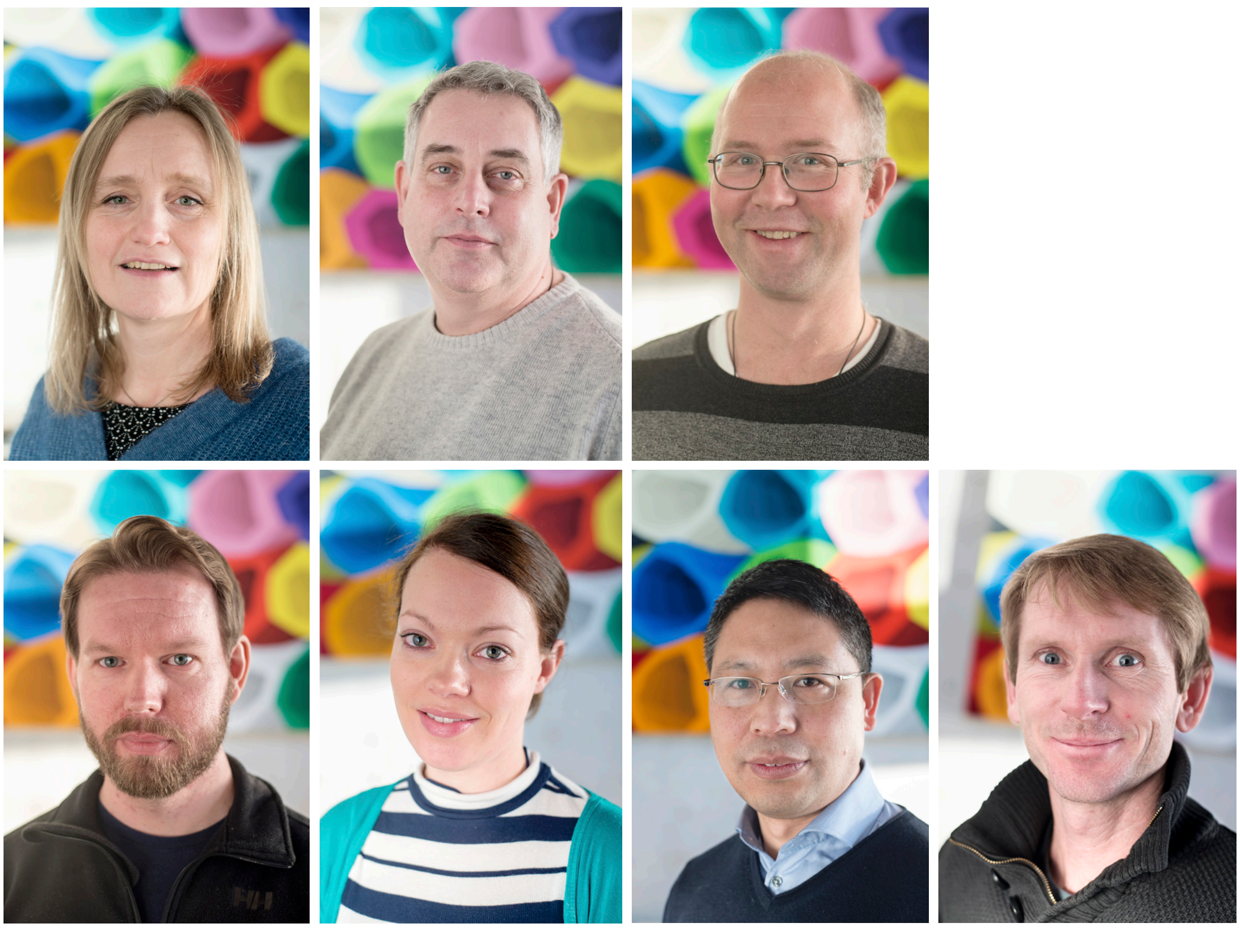
BOARD AND MANAGEMENT

TEM Gemini Centre board:

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

Centre management:

- **Randi Holmestad**, Physics, NTNU, Leader
- **Ragnar Fagerberg**, Materials Physics, SINTEF Industry
- **Ton van Helvoort**, Physics, NTNU
- **Bjørn Soleim**, Physics, NTNU
- **Ragnhild Sæterli**, Physics, NTNU
- **Yanjun Li**, Materials Science and Engineering, NTNU
- **Per Erik Vullum,** Materials Physics, SINTEF Industry



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

THE NORTEM PROJECT

NORTEM (Norwegian Centre for Transmission Electron Microscopy) is a nationally coordinated large-scale infrastructure project with three partners - SINTEF, NTNU and UiO, funded by the Research Council of Norway and the three partners. The budget for new equipment and the re-building in the project was about 75 MNOK for the two geographical nodes, Trondheim and Oslo. We have now been running the facility for close to five years. The support to NORTEM from the Research Council ended in 2016, but the project continues. In October 2018 we submitted a proposal for the follow-up project NORTEM II. This includes upgrading and new investments in both nodes. In the Trondheim node, we apply for detector upgrades on the existing instruments, in addition to a new TEM instrument specialised for spectroscopy.

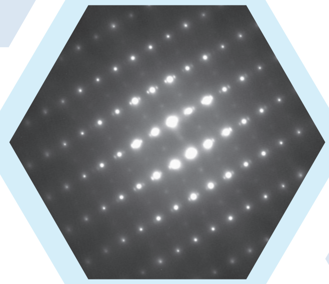
The vision of NORTEM is to be "A world-class TEM facility providing access to expertise and state-of-the-art infrastructure for fundamental and applied research within the physical sciences in Norway". Besides being a top research TEM lab, the infrastructure provides access to TEM for a broader user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

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

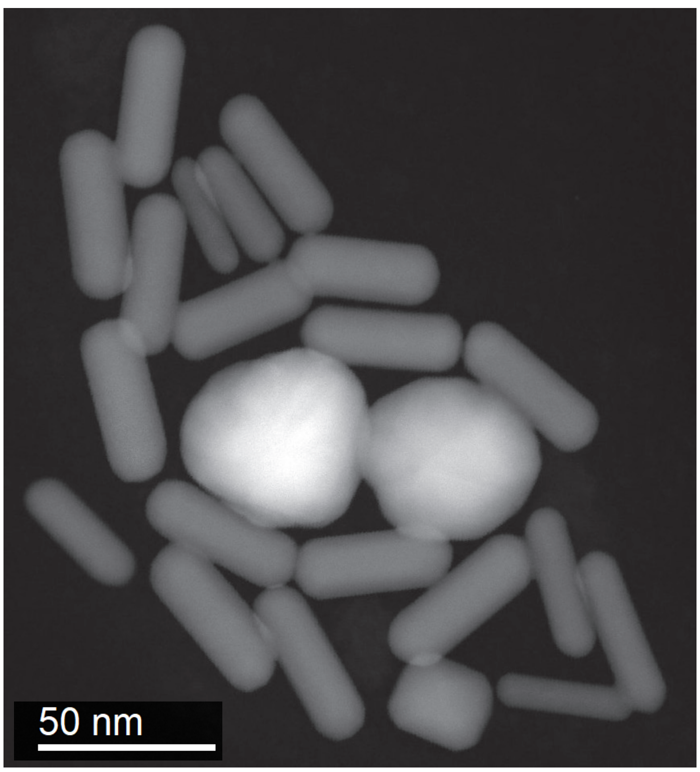
Attention is given to establishing and getting the best out of the huge and complex investment. The Trondheim node NORTEM facility has two senior engineers, Bjørn Soleim and Ragnhild Sæterli supporting maintenance, training, competence and techniques. We have a high uptime and ca. 15% of the users are based outside the host institutions. Per Erik Vullum has been working as adjunct (affiliated) professor, a position created due to NORTEM, which particularly contributes to developing interaction between NTNU and SINTEF.

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

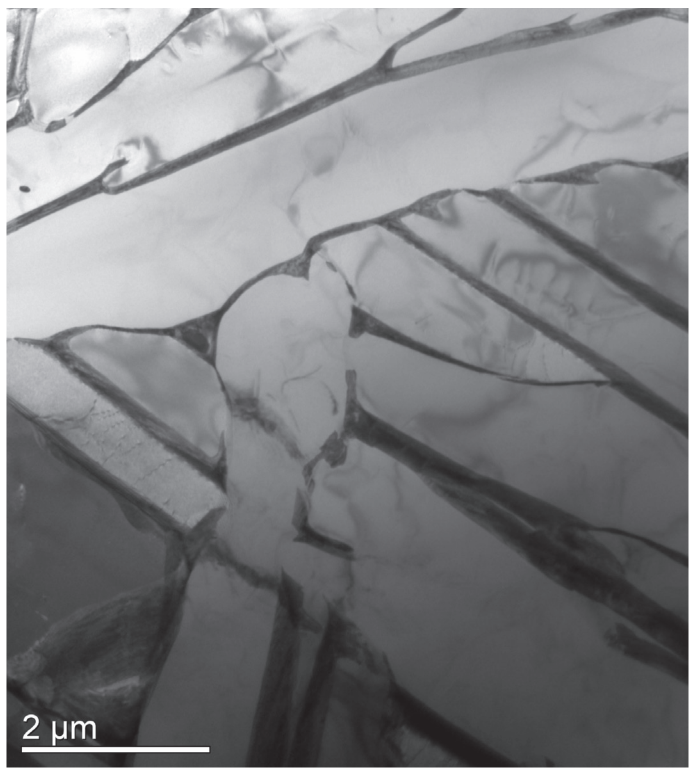
SINTEF researchers in the TEM Gemini Centre. Foto: Lena Knutli.



INSTRUMENTATION



HAADF-STEM image of Au nanoparticles by J.S. Nilsen.
Material provided by Dr S. Bandyopadhyay.



BF-TEM image of a Ti-6Al-4V alloy by G. Nordahl.

THE TEM INSTRUMENTS IN TRONDHEIM

The TEM Gemini Centre has three TEMs installed as part of the NORTEM project in 2013 - a JEM-2100 LaB_6, a JEM-2100F and a double corrected JEM-ARM200F.

JEOL double corrected JEM-ARM200F (cold FEG)

This is currently one of the most powerful operative advanced TEMs in Europe. The stable cold FEG 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 kV and 200 kV

JEOL JEM-2100F

This FEG TEM is optimized for all-round advanced materials studies with a special focus on scanning precession electron diffraction (SPED) and tomography.

The 2100F features:

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

JEOL JEM-2100

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

The 2100 features:

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

Foto: Ole Morten Melgård.

SPECIMEN PREPARATION

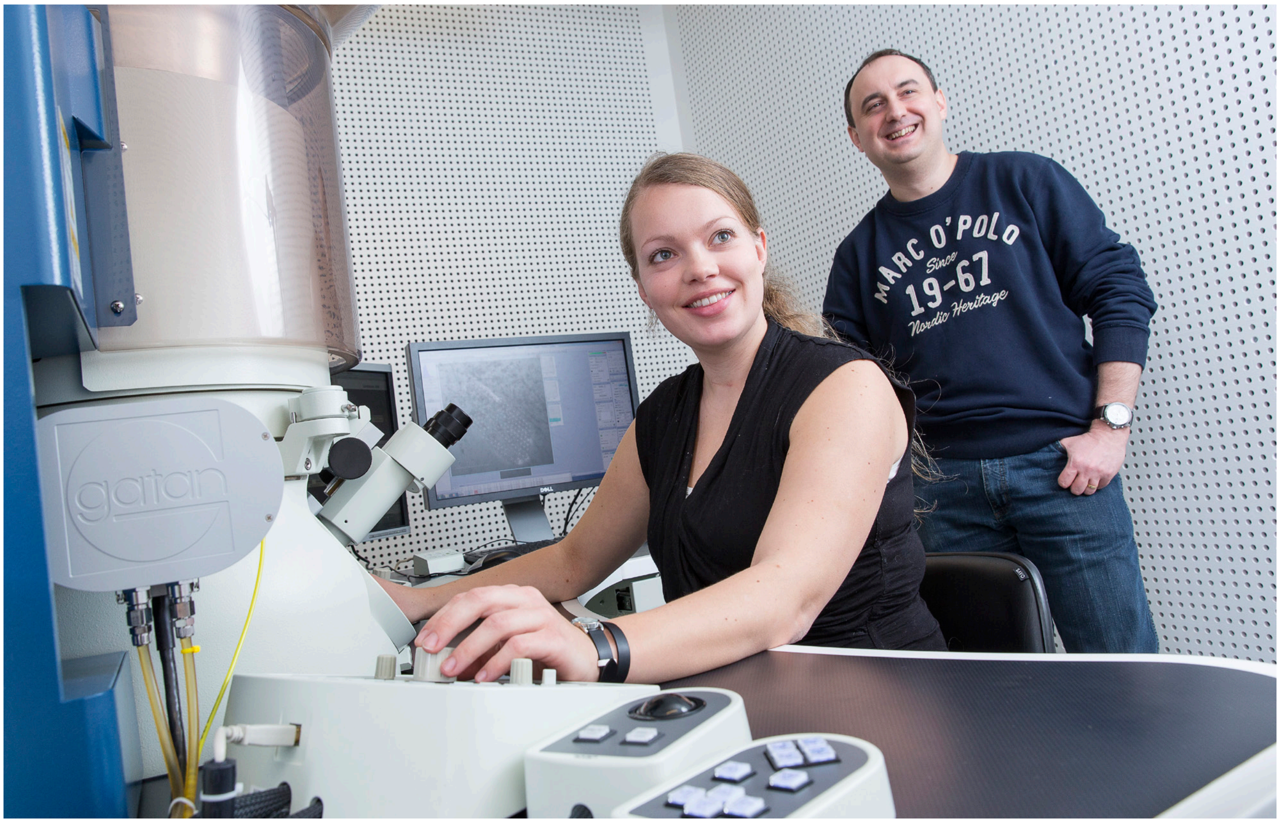
Given the high resolution of the TEM instruments, specimen quality is often the limiting factor. The Gemini Centre has well equipped specimen preparation facilities at both DP and DMSE, reflecting the broad range of materials studied. The Centre has different types of dimplers, saws, an ultrasonic cutter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

The Centre has three Gatan PIPS instruments, including a PIPS II, to make more high-quality and reproducible specimens. A routine has been developed to polish focused ion beam (FIB)-made TEM lamellas to obtain the highest specimen quality and the best possible TEM results. Many TEM projects utilize the FIB at NTNU NanoLab with lift-out option for site-specific TEM specimen preparation.

The electropolisher at DP is essential in producing high quality Al TEM specimens. A semi-automatic tripod polishing set-up is available for large area preparation of hard materials. For soft materials, such as polymers, ultramicrotomy is an essential technique that is also used for preparation of catalysts, surface structures and nanoparticles.

SUPPORTING FACILITIES

With the aberration corrected microscope, the cleanliness requirements of the specimen and the holders are high. We have a dedicated room close to the microscopes with general equipment, such as a plasma cleaner, ozone cleaner, a stereomicroscope, user specimen storage and special holders that are used on all three TEMs. In addition, there is a data transfer room with additional facilities as a printer and a support PC with the most crucial software packages. The room has a sofa and tea/coffee machine for socializing and efficient breaks during long running sessions. The dedicated TEM computer room has five machines, for post-processing and simulating TEM results, some of which can be remotely accessed. At the end of 2018 an order is placed for a more powerful unit, dedicated to more demanding data processing. All acquisition software is accessible via offline licenses in the computer room.



SPECIMEN HOLDERS

Each TEM has its own set of single and double tilt holders. A broad range of additional holders is available for use on all three microscopes. This includes a cold stage holder, a conventional heating holder, an environmental cell holder, a transfer holder, several tomography holders, two tilt-rotation holders and back-up double tilt holders. A special holder is a MEMS based heating holder, which can also be used for biasing. In addition, we are developing routines for preparing TEM lamella of metal and ceramic materials on the MEMS chip for in-situ TEM studies.

USER STATISTICS IN 2018

The total registered used time for the three instruments in 2018 was 3489 hours, including 124 non-paid hours used for testing, competence development, demonstrations and guided tours. Of the 3365 paid hours, the use by NTNU corresponds to 85 % and by SINTEF 15 %. NTNUs use is divided over seven departments, where the main use is from Department of Physics (76 % of NTNUs paid hours). About 85 different projects used TEM in 2018.

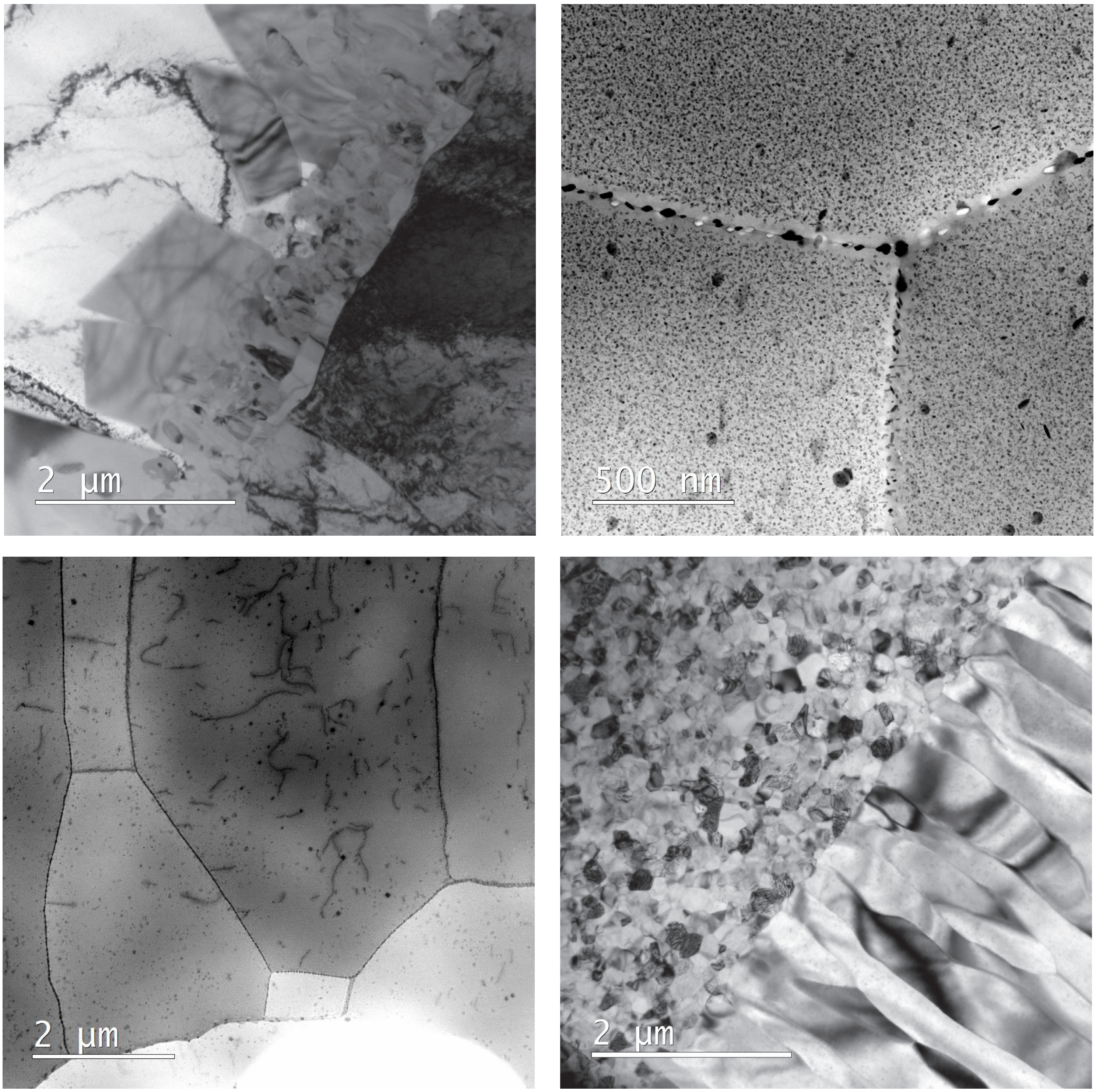
The infrastructure had 38 hands-on users in 2018, where 4 were based at SINTEF, 14 were PhD candidates and 11 Master students. The use from outside the Physics department has experienced an increase. 7 PhD candidates from other departments used the microscopes within the infrastructure in 2018.

Foto: Ole Morten Melgård.

Microscope use in hours	ARM-200F	2100F	2100	SUM
SINTEF	366	105	24	495
NTNU – Physics	620	824	550	1994
NTNU – Other departments	89	152	420	661
NTNU – Visitors from abroad	54	0	35	89
NTNU – Teaching lab	0	88	32	120
External	0	6	0	6
NTNU – Set-up/testing/training/demonstrations	31	60	33	124
Total use	**1160**	**1235**	**1094**	**3489**

ACTIVITIES

RESEARCH AND EVENTS



BF-TEM image of the intermetallic phases α-Al-Si-(Fe,Mn), Fe_4Al_{13} and Fe_2Al_5 at the interface between Al and steel by T. Bergh (top left). BF-STEM images of the microstructure in an Al alloy with $MgZn_2$ precipitates by A. Lervik (top right and left bottom). BF-TEM image of Al_3Ni_2 and Al_3Ni by T. Bergh (bottom right).

FOCUS AREAS

TEM is a powerful technique for fundamental and applied research in the physical sciences, in different fields from geology, metallurgy and semiconductor industry to fundamental chemistry and physics. NORTEM has identified four focus areas, which have been important for the TEM Gemini Centre activities since the Centre was formed. Within these areas we see potential for further growth and tackling unsolved issues. The focus areas are light metals, catalysis, energy materials and nanotechnology. TEM plays an important role in these research areas, which will be strategically important for Norway also in the future. The TEM Gemini Centre had activities in all these four areas in 2018. The next sections describe these activities. Activities in aluminium alloy research are the largest.

ALUMINIUM - LIGHT METALS

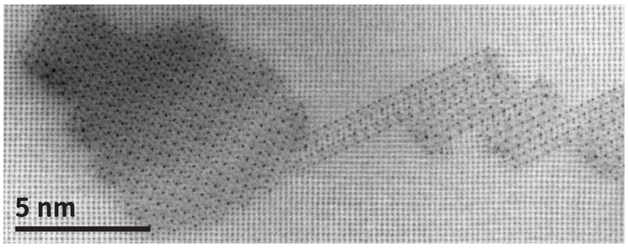
The study of aluminium alloys using TEM has been a pillar in the Trondheim TEM environment for many years, and there have been many successful projects. All these projects have been jointly between NTNU and SINTEF, and supported by the Research Council of Norway. In addition, many of them were supported by Norwegian light metal industry, in particular Hydro Aluminium. In 2018 we have been involved in 2 SFI Centers, 2 competence projects and one Digitalization project in aluminium research.

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

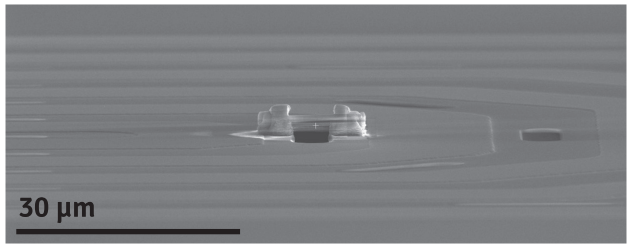
People working with aluminium and TEM at NTNU/SINTEF and visitor Katsuhiko Nishimura, February 2018.

In SFI Manufacturing, headed by Sverre Guldbrandsen-Dahl from SINTEF Manufacturing, joining of aluminium with other materials in multi-material products is a central topic. PhD student Tina Bergh characterises the microstructure of the interfacial region in aluminium-steel joints made by various joing techniques, including joints made by the start-up company Hybond. Tina uses conventional and advanced TEM techniques and also works on electron microscopy data analysis.

Two competence projects involving NTNU and SINTEF on aluminium research are ongoing. One is the project 'Fundamentals of intergranular corrosion in aluminium alloys' (FICAL) where Randi Holmestad is the project leader. FICAL is



ABF-STEM image of a chain of S-(Al_2CuMg) phase precipitates nucleated on a dislocation in aluminium by J.K. Sunde.



SEM image of a TEM lamella fastened to a heating chip by S. Wenner.

a 5 years project that has the objective of establishing new fundamental understanding of the mechanisms of intergranular corrosion (IGC) susceptibility. Industrial funding is provided by a consortium of four aluminium companies; Hydro Aluminium, Benteler, Gränges and Steertec. These companies represent the entire value chain, from alloy production to component manufacturing. The mechanisms of IGC are studied at the nm-scale utilizing advanced laboratory infrastructure, especially TEM, plus modeling, where NTNU and SINTEF researchers study the detailed connections between IGC, composition and thermal history. It is essential to optimize corrosion resistance with mechanical and other alloy properties for best alloy design and performance. Adrian Lervik is working as a PhD student in the FICAL project and focuses on quantitative understanding of nanoscale structure and chemistry around grain boundaries. One concrete case studied is stress corrosion cracking in 7xxx alloys from Benteler Automotive.

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

NAPIC (NTNU aluminium product innovation Centre) was established in 2017, and Håkon Wiik Ånes is hired as a PhD student in this centre to study nucleation of recrystallization. He partly uses TEM in these studies.

During the last years, we have had several aluminium alloy related collaborations abroad. The largest is the Japanese collaboration with academia and industry, where the INTPART project was renewed for 4 new years in 2018 and extended with new partners. This is further presented elsewhere in this report. In addition, we have in 2018 worked with the group of Dr. Benjamin Mikereit from Rostick, Germany, who is doing DSC (differential scanning calorimetry) experiments, complementary to TEM.

The newest aluminium project is the NTNU-financed Digitalization project AllDesign with Prof. Jaakko Akola as project leader. AllDesign will provide fundamental insight on solid-state precipitation in aluminium alloys based on synergistic multiscale modelling, and its impact on macroscopic properties and manufacturing processes. The concept is data-driven and utilizes new trends in materials research. Elisabeth Thronsen was hired as a PhD student on this project in August 2018 and will work on TEM of early stage clustering and precipitation.

Dr Marat Gazizov is a postoc in the TEM Gemini Centre, supported by the Natural Sciences. Two Master students (Elisabeth Thronsen and Sigurd Oftstad) plus three project students (Yngve M. Ender, Edwin N. Traore and Haakon Tvedt) did their thesis work on aluminum alloys in 2018.

Nine members of the TEM Gemini Centre participated in the ICAA16 conference in Montreal, Canada in June 2018, and most of them gave oral presentations. Dr. Eva Mørtsell, Postdoc at DMSE, NTNU, was given the ICAA16 early career researcher award.

The last two years we have started to study titanium alloys in the TEM . In 2018 Susanne Araya did her Master thesis on additively manufactured titanium grade 5 in collaboration with Norsk Titanium. Gregory Nordahl followed up this work with a project in the fall of 2018.

ENERGY MATERIALS – SOLAR CELLS

TEM has proven to be a crucial characterization tool to understand and improve the efficiency of both conventional and novel types of solar cells. The TEM Gemini Centre activities within solar cells include both types and a large range of materials.

The Gemini Centre is participating in the FME SUSOLTECH (The Norwegian Research Centre for Sustainable Solar Cell Technology) on solar cells and project students, PhD students and SINTEF researchers within TEM are actively taking part in subprojects related to both conventional as well as third generation solar cells.

Maryam Vatanparast is a PhD student working on TEM characterization of intermediate band solar cells, funded by the project 'High Efficiency Quantum Dot Intermediate Band Solar Cells' headed by Turid D. Reenaas at DP. The intermediate band is created by multiple layers of InAs quantum dots positioned inside a GaAs-based matrix semiconductor. Here we are studying the microstructure in detail, and also working on measuring band gaps with electron energy loss spectroscopy. Hogne Lysne is a PhD student in the FME on solar cells working on the deep level impurity approach doing Ag and W implantation into Si with Turid D. Reenaas as main supervisor. In 2018 Jørgen Sørhaug worked on this project as a project student.

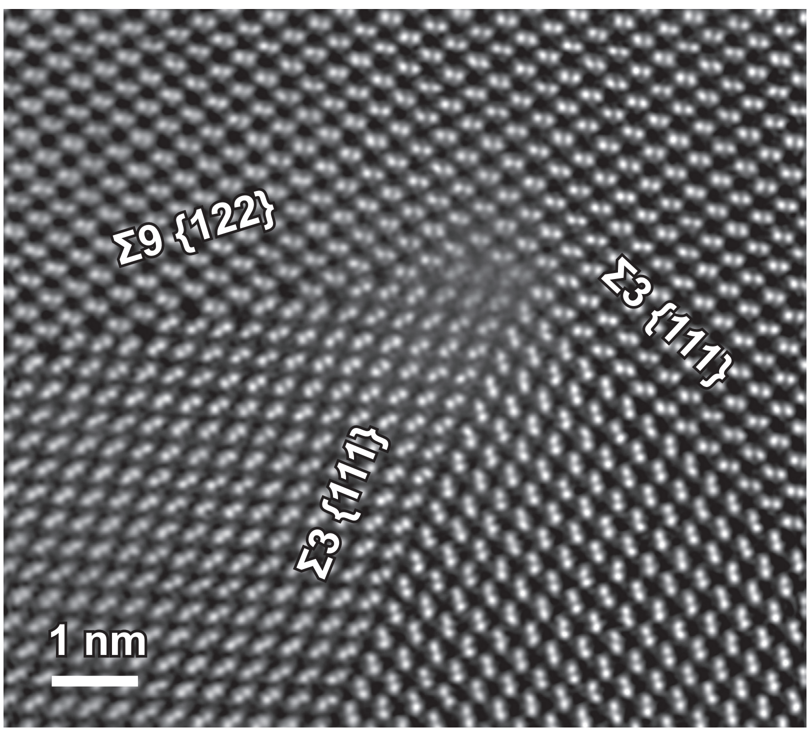
PhD student Julie Stene Nilsen is part of the NANO2021 GRANASOL project (Low Cost, Ultra-High Efficiency Graphene/Nanowire Solar Cells) and use TEM to understand metal contacts, compositional variations and defect in the nanowires.

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

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

SINTEF has worked together with ELKEM and IFE in two consecutive IPN projects within production of tailored Si powders for use in Li-ion batteries. The last one is called "SiCANODE" (2016–2018). The aim is to develop Si/graphite based composites as anodes in commercial Li-ion batteries. TEM has been one of the primary tools to characterize and understand the behavior of the anode composites as a function of structure, morphology and cycling conditions. This work will continue in a new IPN project, "SiBanode", that will start in 2019. An IPN project called "DOVRE" was started in 2018. The project owner CENATE, a spin-off company to Dyntec, aims to develop Si-based materials optimized for anodes in commercial Li-ion batteries. TEM is here a central characterization tool to study and understand the behavior of the initial and cycled Si-based electrodes.

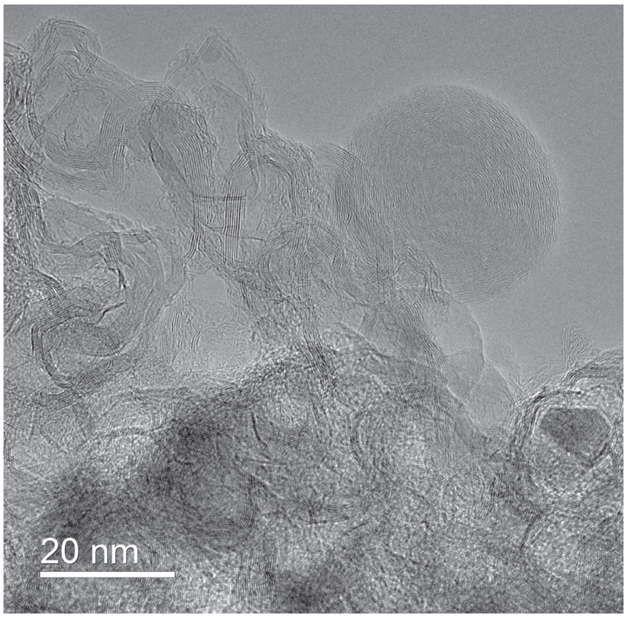
SINTEF is also partner in another project related to Li-ion battery R&D: "LiMBAT" is a researcher project where SINTEF, IFE,



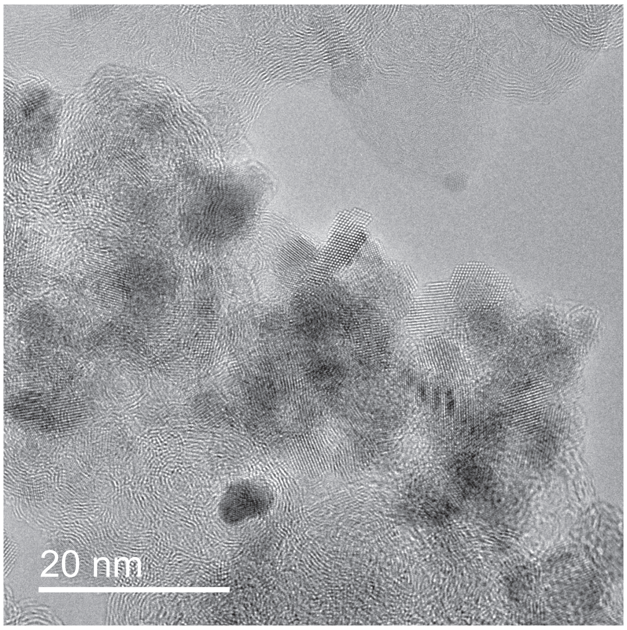
HAADF-STEM image of a grain boundary triple junction in HP mc-Si for photovoltaic applications by M. Tsoutsouva and P.E. Vullum.

NANOTECHNOLOGY

UiO, CNRS (France) and Hiroshima University (Japan) are cooperating to develop high capacity anodes based on metal hydrides for use in conversion electrodes in Li-ion batteries. TEM is in this project one of the most important tools to understand the correlations between morphologies and the electrochemical performances of the synthesized anode materials.



HR-TEM image of carbon structures in charcoal by S. Wenner.

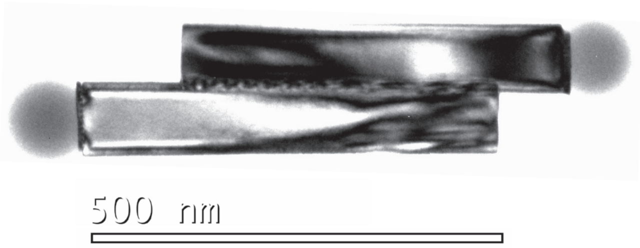


HR-TEM image of CeO_2- and Pd-particles on carbon by S. Wenner.

In nanotechnology and nano-sciences, TEM is a valuable tool to understand the relation between functional properties and morphology, structure and composition variations at the (sub)nm scale. TEM is not only important because of its resolving power, but also that structural characteristics can be simultaneously analyzed. The TEM Gemini Centre activities cover nanoparticles, 1D-nanostructures (i.e. nanowires, carbon nanotubes), thin films and 2D-materials such as graphene. These activities are part of NANO@NTNU. NTNU NanoLab is our direct neighbor and many of the TEM operators also use equipment in the cleanroom. Especially the FIBs are important for the TEM Gemini Centre.

NorFab-II is an important partner for the TEM Gemini Centre. PhD Aleksander Mosberg is funded by NTNU's "Enabling technologies: Nanotechnology", and is based in the TEM Gemini Centre. Mosberg's PhD is focused on using FIB for nanostructuring. TEM is used to understand how the ion beam alters the materials. He uses TEM to study the made structures in detail. Further many of our master students follow the Nanotechnology study program, a further demonstration that TEM is an essential tool for practical nanotechnology. These student projects include both practical as well as theoretical/computational focus.

Several core TEM Gemini people are actively involved in nanotechnology projects, for example as co-supervisors. In the TEM Gemini Centre, both NTNU and SINTEF have worked with the start-up company CrayoNano. The Centre has close ties with the Norwegian PhD Network on Nanotechnology for Microsystems. As can be seen from the publication list, many TEM studies on nanomaterials resulted in journal publications in 2018. Further, many of the example TEM images in this report can be classified under nanotechnology.



BF-TEM image of self-catalyzed GaAsSb nanowires by J. Neumann.

ADVANCED DATA PROCESSING

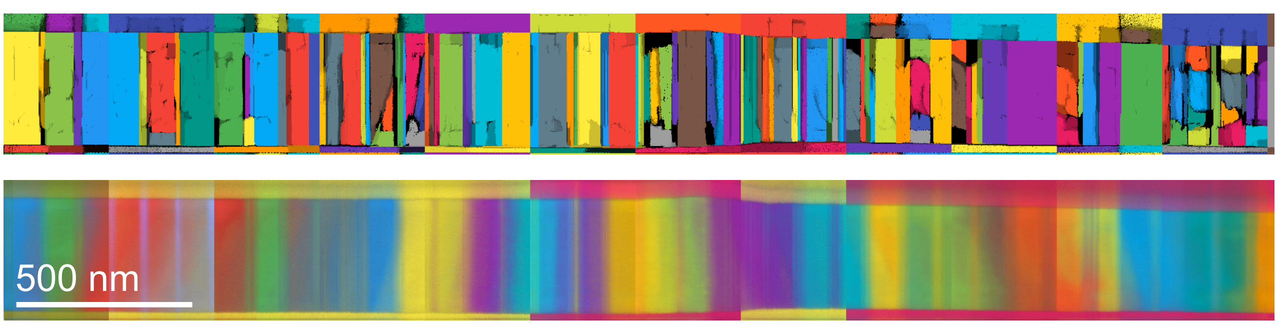
The ongoing revolution within TEM research is digital. Big/smart data, machine learning, open source, digital transformation, etc. are are some of the current "hot topics". In 2018, procedures were developed within the centre to handle larger data sets and automation for more robust as well as more transparent TEM data handling. There were MSc, PhD and SINTEF projects dedicated to improve the data handling in 2018. The 2017 investment in data storage and handling capacity worked well, and further investments will be made in 2019. In 2018 we secured funding for the first direct detection TEM detector in Norway and this will be installed in 2019.

In TEM, especially multidimensional data set acquisition and handling, data processing transparency and dynamic in-situ studies are focus points. Students within the group have contributed over many years to the open-source software, especially the Python library HyperSpy (hyperspy.org) and especially for electron diffraction pyXem (github.com/pyxem). Image, diffraction, EDX and EELS data from all our TEMs can now be handled in the same user interface. For large datasets or demanding calculations there is access to a large cluster. In 2019 a powerful workstation will be installed for users who have no access to the cluster. SINTEF had in 2018 an internal project to support these data handling developments and to incorporate them in their TEM activities. Furthermore, we organized an internal workshop on scanning precession electron diffraction (SPED) data handling using Python and clusters.

CHEMICAL ENGINEERING - CATALYSIS AND MEMBRANE MATERIALS

The Centre has continued a strong interaction with the national catalysis environment, including the NTNU Chemical Engineering department, SINTEF Industry, Chemistry Oil and Gas Process Technology Departments. The Gassmaks programme project to develop catalysts and materials for a compact steam reformer is addressing both catalyst and materials issues, such as degradation of steels by metal dusting corrosion during exposure to synthesis gas. We hope that the SFI Innovation for a Competitive and Sustainable process Industry (iCSI), headed by professor Hilde Johnsen Venvik, will provide a platform for further applications of TEM in both academic and industrial catalysis research. Membrane research has contributed to a study in the BIGCCS carbon capture FME project.

TEM work on catalyst in 2018 inlcuded catalysts for Fisher-Tropsch synthesis, carbon- ionic liquid super-capacitors and ceramic-ceramic hydrogen permeable membranes.



Visualization of machine learning results for a SPED dataset of a GaAsSb nanowire by S. Høgås. The nanowire was grown by Dr D. Ren (NTNU), while specimen preparation and SPED was done by J.S. Nilsen. Clusters identified with UMAP and HDBSCAN (top) and UMAP loadings color coded according to the clusters (middle). Color coded non-negative matrix factorization loadings (bottom).

ACTIVE PROJECTS

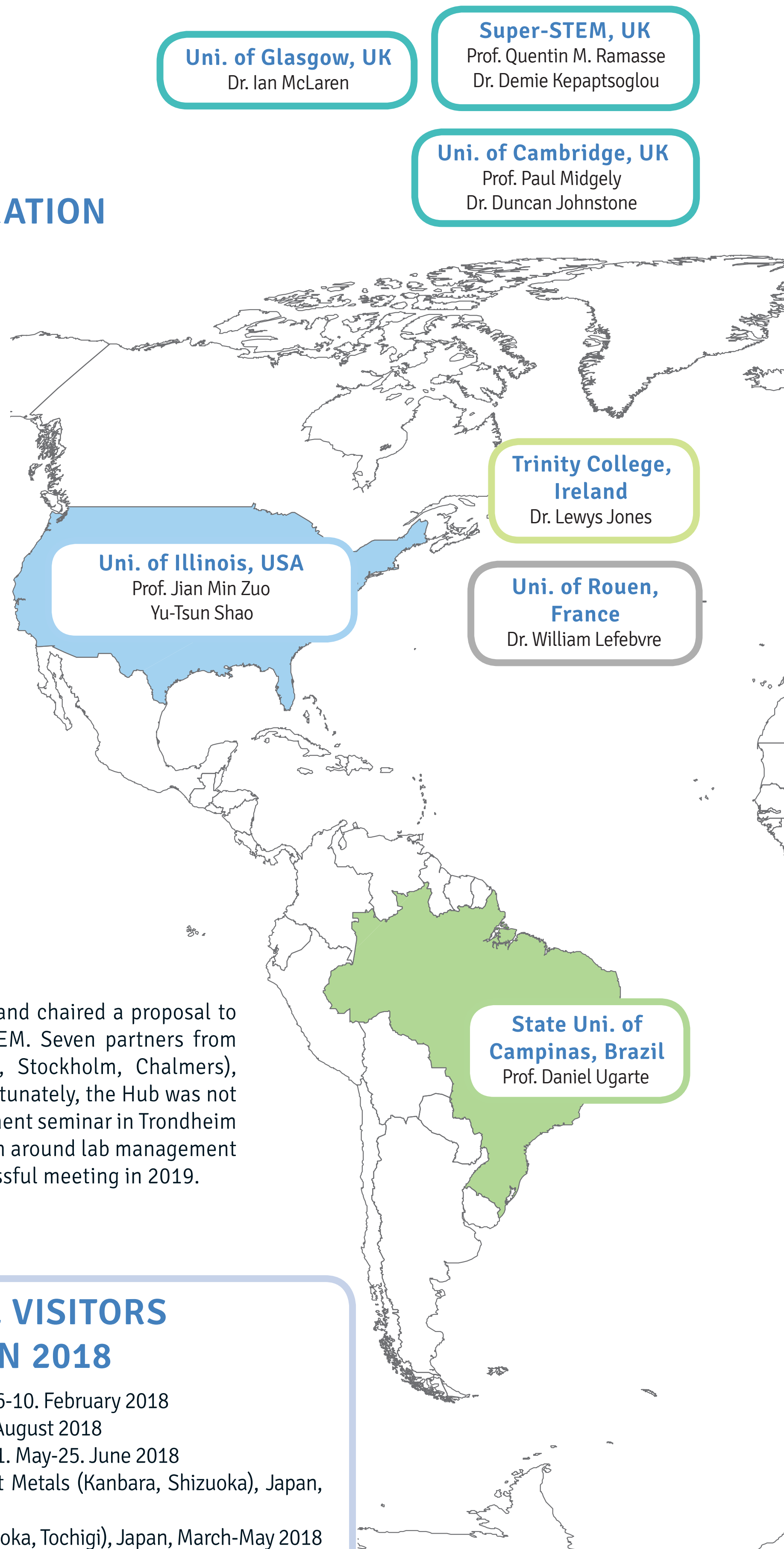
The table below shows the larger projects connected to TEM within the Gemini Centre. They are listed by funding type, title, duration and research partners. A number of smaller projects not listed, both academic and with direct industrial support, run in parallel. In total the Centre had over 85 different projects using the facilities.

Project type	Project title	Involved with TEM	Duration
SFI	**CASA - Centre for Advanced Structural Analysis**	1-2 PhDs, SINTEF	2015-2023
Partners: NTNU, SINTEF, Statens vegvesen, Forsvarsbygg, Norwegian ministry of local government and modernisation, NSM, Audi, Benteler, BMW, DNV GL, Gassco, Honda, Hydro, MultiConsult, Sapa, Statoil, Toyota, Renault			
SFI	**SFI Manufacturing**	1 PhD, SINTEF	2015-2023
Partners: SINTEF, NTNU, Benteler, Brødrene AA, Ekornes, GKN Aerospace, Hexagon composites, Kongsberg Automotive, Nammo, Raufoss Neuman, Plastal, Plasto, Rolls Royce, Teeness, Hybond, Hydro			
SFI	**Industrial Catalysis Science and Innovation for a Competitive and Sustainable process Industry (iCSI)**	SINTEF	2015-2023
Partners: Yara Norge, K.A. Rasmussen, Dynea INOVYN Norge, Haldor Topsøe AS			
FME	**SuSolTech – The Research Center for Sustainable Solar Cell Technology**	1 PhD, Postdoc, SINTEF, UiO	2009-2025
Partners: IFE, NTNU, SINTEF, the University of Oslo, CleanSi, Dynatec, Elkem Solar, Mosaic, Norsun, Norwegian Crystals, Quartz Corp, REC Silicon, REC Solar, Semilab			
FP/FRINATEK	**Fundamental investigations of precipitation in the solid state with focus on Al-based alloys**	1 PhD, 1 Postdoc, SINTEF	2013-2018
Partners: NTNU, SINTEF			
NTNU Digital Transformation	**Rational Alloy Design - ALLDESIGN**	1 PhD	2018-2021
Partners; 4 departments at NTNU; Physics, Materials Science and Engineering, Mechanical Engineering, Mechanical and Industrial Engineering			
FP/ENERGIX	**High Efficiency Quantum Dot Intermediate Band Solar Cells (HighQ-IB)**	1 PhD, SINTEF	2012–2018
Partners: NTNU, SINTEF			
IPN/BIA	**Integrated Hardening and Sheet Press-forming of Aluminium (I-Pal)**	SINTEF	2016-2019
Partners: SINTEF, Hydro, SAPA, Raufoss Technology Neumann, AP&T			
KPN/BIA	**Aluminium alloys with mechanical properties and electrical conductivity at elevated temperatures (AMPERE)**	1-2 PhDs, SINTEF	2015-2020
Partners: NTNU, SINTEF, Hydro, Nexans, Raufoss Neuman, SAPA, Grnges			
KPN/BIA	**Fundamentals of Intergranular Corrosion in Aluminum Alloys (FICAL)**	1 PhD, SINTEF	2015-2020
Partners: NTNU, SINTEF, Hydro, Benteler, Steertec, Gränges			
IFP/Nano2021	**GRANASOL - Low Cost, Ultra-High Efficiency Graphene Nanowire Solar Cells**	1 PhD	2014-2019
Partners: NTNU, Sejong University, Aalto University, CRAYONANO AS			
INTPART	**Norwegian-Japanese Aluminium alloy Research and Education Collaboration (NJALC)**	Travel, exchange students	2015-2018
Partners: NTNU, SINTEF, Hydro, University of Toyama, Tokyo Institute of Technology			
FP/PETROMAKS2	**Fatigue and hydrogen degradation of steels (HyF-Lex)**	SINTEF	2015-2018
Partners: NTNU, SINTEF			
FP/FRINATEK	**Oxide Intermediate Band Photovoltaics (Ox-IB)**	1PhD, SINTEF	2015-2020
Partners: NTNU, SINTEF			
FP/ENERGIX	**In-Situ characterization and Simulation of Defect Evolution in Silicon (INSIDES)**	1 Postdoc, SINTEF	2016-2019
Partners: NTNU, SINTEF			

INTERNATIONAL COLLABORATION

As can be seen from the map and the publication list, the TEM Gemini Centre has relations to many research institutions and researchers across the world. Some are long term collaborations, others are new initiatives. The main step forward for international collaborations is ESTEEM3, which effectively starts in Q1 of 2019. The map illustrates the direct scientific collaborations. We have yearly several visitors from abroad that use the TEM facility in Trondheim. An example of a long-standing international cooperation is the contact with Toyama University and Tokyo Institute of Technology in Japan, now supported by the new INTPART project (see p. 18). People from the group were chairs and invited contributors to international conferences and acted as PhD examiners abroad.

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

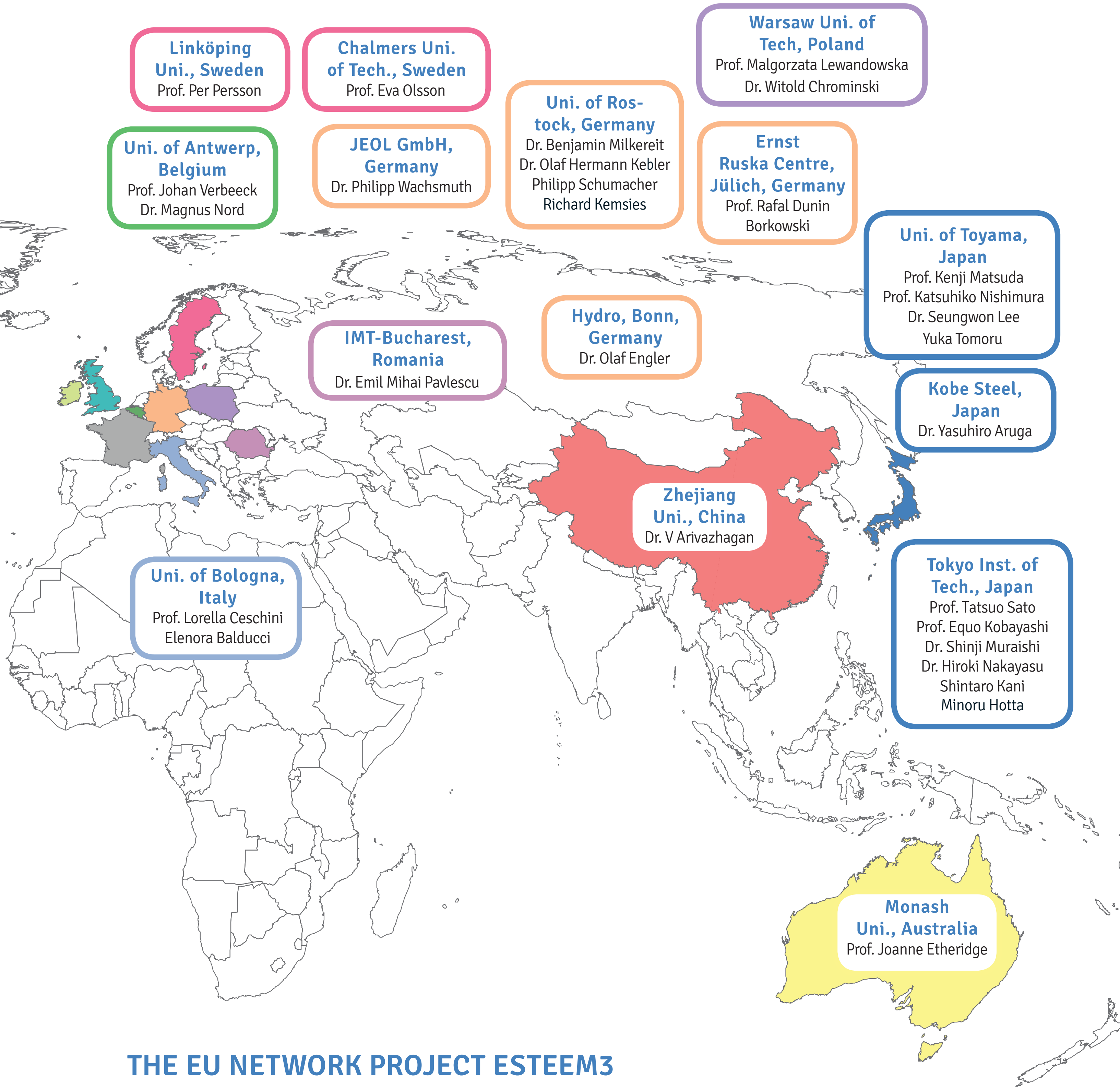


NORDIC HUB

The Gemini Centre took in 2017 the initiative to and chaired a proposal to NordForsk for a Nordic University Hub within TEM. Seven partners from Norway (NTNU and UiO), Sweden (Lindköping, Stockholm, Chalmers), Denmark (DTU) and Finland (Aalto) were in. Unfortunately, the Hub was not granted. However, NTNU organized a lab management seminar in Trondheim in May 2018, where best-practice and organization around lab management were discussed. We intend to follow up this successful meeting in 2019.

INTERNATIONAL VISITORS AND VISITS IN 2018

- Katsuhiko Nishimura, University of Toyama, 6-10. February 2018
- Artenis Bendo, University of Toyama, 15-20. August 2018
- Maxmillian Engelhart, University of Leoben, 1. May-25. June 2018
- Sigurd Ofstad, Tokyo Tech and Nippon Light Metals (Kanbara, Shizuoka), Japan, February- May 2018
- Jonas K. Sunde, Tokyo Tech and KOBELCO (Moka, Tochigi), Japan, March-May 2018
- Adrian Lervik, Tokyo Tech and UACJ (Nagoya, Aichi), Japan, March-May 2018



THE EU NETWORK PROJECT ESTEEM3

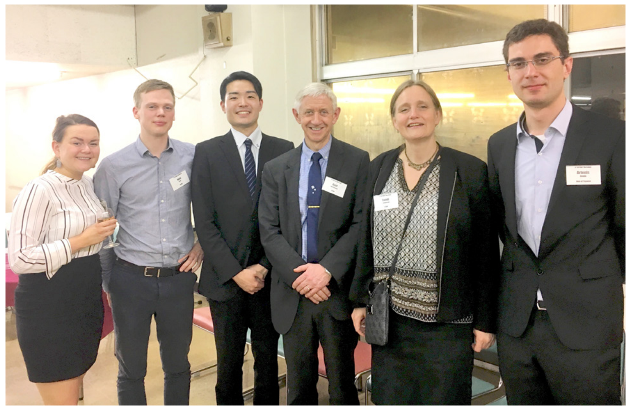
The TEM Gemini Centre is glad to be a partner in the newly granted EU Horizon 2020 INFRAIA initiative ESTEEM3. ESTEEM3 is a European Network for Electron Microscopy among the leading European TEM groups, integrating activity for electron microscopy, and providing access, facilitating and extending transnational access (TA) services. The project started in the beginning of 2019, and NTNU is involved in several work packages; training (microscopy schools), outreach (in particular industrial outreach) and in the joint research area 'Materials for transport', in the last topic together with AGH in Krakow, Poland. In addition, we are affiliated to the work packages Diffraction and Data analysis. The main part of ESTEEM3 is that we can welcome researchers for transnational access, and we hope to get many visitors through this access program. TA-exchanges do not only include work on the TEM, but can also be data handling. The website of ESTEEM3 (esteem3.eu) gives more details on how to get access through TA. One 4 years postdoc is hired on the project and starts in March 2019.

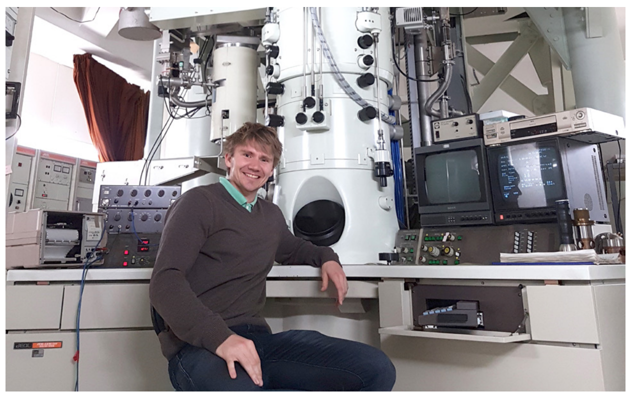
INTPART PROJECT WITH JAPAN

A 3-year International Partnership (INTPART) project funded by the Norwegian Research Council and the Norwegian Centre for International Cooperation in Education (SIU) called "The Norwegian-Japanese Aluminium alloy Research and Education Collaboration" ended in 2018. In addition to NTNU and SINTEF, Hydro Aluminium, University of Toyama and Tokyo Institute of Technology were partners. The objective of this project has been to continue the fruitful partnership we obtained through the earlier BILAT project, and also include and formalize educational issues, such as guest lecturers, workshops joint courses and internships. Furthermore, exchange of students on Master/ PhD levels between the university partners with research close to the aluminium industry, will ensure strong and long-lasting international collaboration.

During 2018 there has been a large activity in the INTPART project. We have had two visits from University of Toyama to NTNU, we had three internships in Japanese aluminium industry and two visits to Japan from the aluinium group in the TEM Gemini Centre. In May, 11 people from Trondheim were in Japan, and we organized a two days workshop at Tokyo Tech with 40 participants. Also people from the NTNU administration followed, to make more formalized connections to Tokyo Tech and University of Toyama. In October, 4 researchers from Trondheim visited Toyama to give lectures, and participated in two conferences.

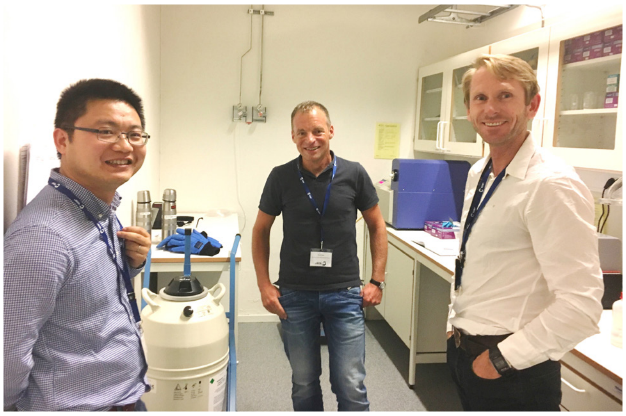
In 2018 we applied for a continuation of this project, and got granted a phase II at the end of 2018, with duration 2018-2022. In addition to the original partners we had in phase I, Kyushu University in Fukuoka has been added into the collaboration. We are happy to be able to continue this fruitful collaboration with Japan!





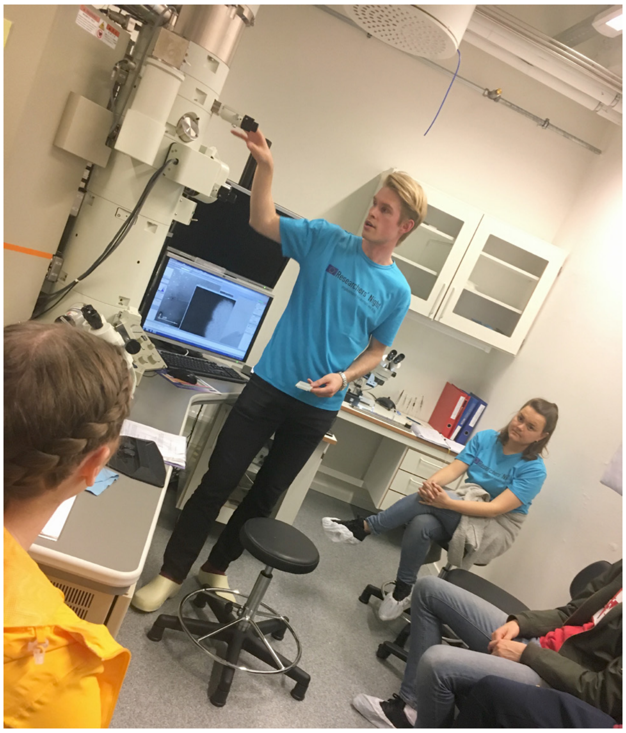
ARM OWNER MEETING IN TRONDHEIM

The 5th European user meeting for ARM owners was organized at Realfagbygget in Trondheim 15th-16th of June 2018. The meeting had 46 participants from 10 countries. Invited speakers from the ARM labs were Angus Kirkland (Oxford), Ton van Helvoort (NTNU), Alan Craven (Glasgow), Ian Griffiths (Oxford), Dogan Ozkaya (MTC), Eva Olsson (Chalmers), Ying Wang (MPI), and Richard Beanland (Warwick). In addition to JEOL specialists Hiroki Hashiguchi and Ichiro Ohnishi informing about the last developments on ARM microscopes. The TEM Gemini Centre organized lab tours and demos on our three JEOL instruments. Discussions were extended during a nice dinner at Ringve Museum. We are honored to be allowed to organize this meeting, and think it brought up nice discussions on front research being done on the ARM instruments around in Europe as well as stimulated collaborations between users based across Europe. Please note that the TEM Gemini Centre and JEOL have since 2013 a cooperation agreement (JEOL Competence Centre (JCC)) to develop the facility and its users' competence.



NORTEM II APPLICATION

The NORTEM project (page 4) was granted in 2011 and the funding ended in 2016. To be able to be in the research front, we now see needs for upgrades and investments to keep the facility at the technological forefront. The partners (SINTEF, NTNU and UiO) therefore submitted a NORTEM II proposal for the large scale infrastructure call in October 2018. Included in the proposal for the Trondheim node are upgrades of detectors on the ARM200 and the 2100F, in addition to a new instrument with GIF, dedicated to spectroscopy. The result of the application will be announced before summer 2019. Fingers crossed!

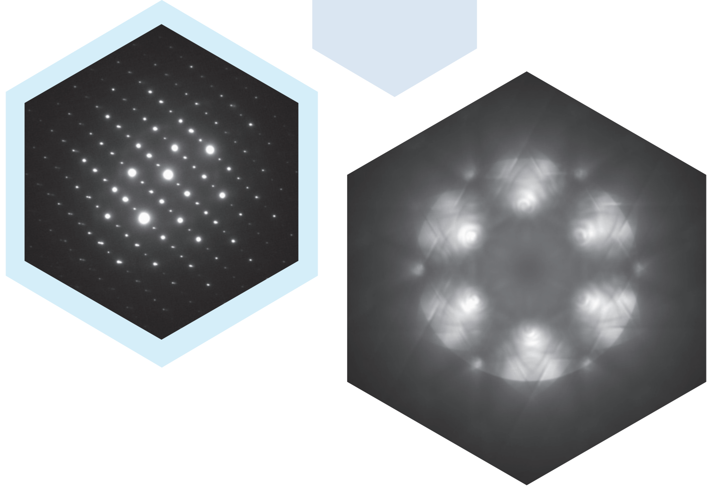


RESEARCHERS' NIGHT, SCHOOL VISITS AND OUTREACH

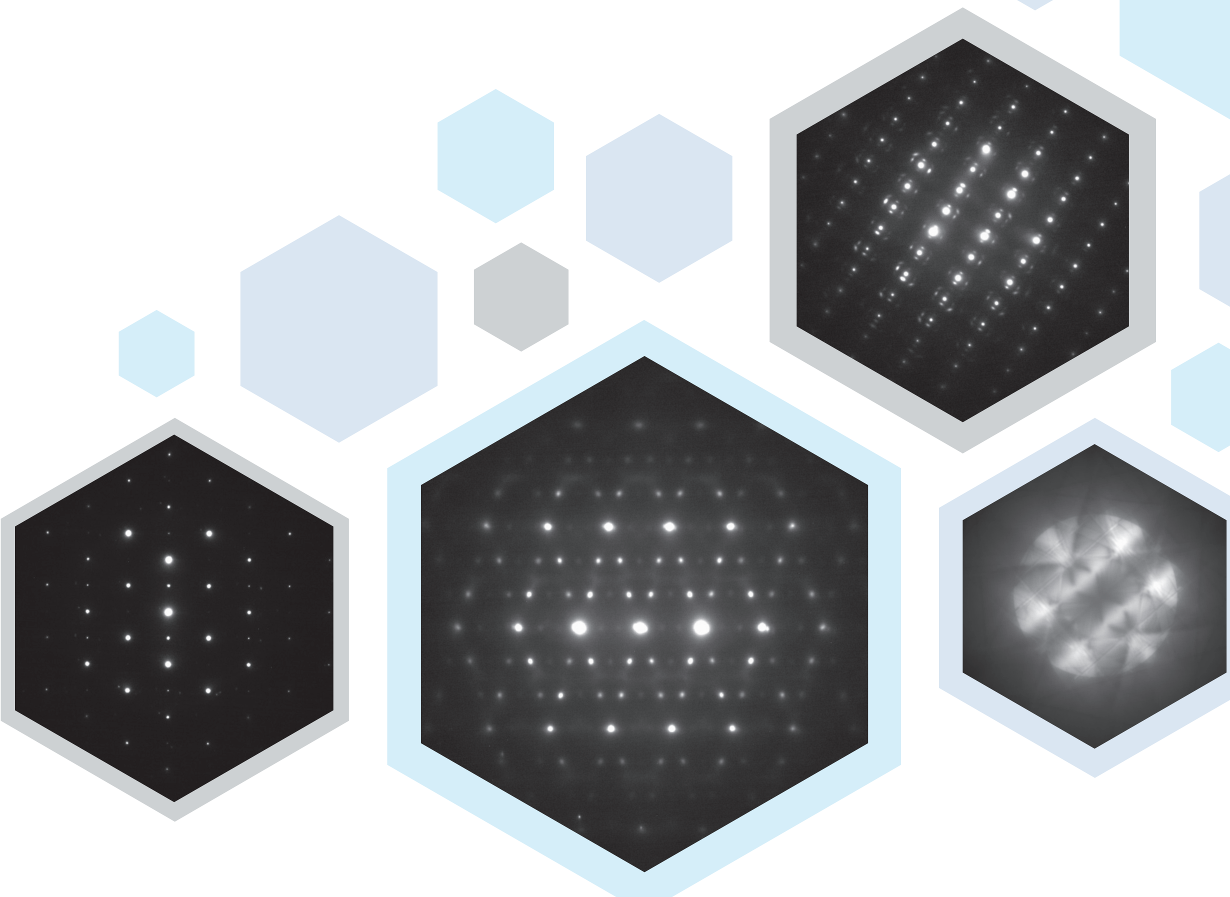
As in earlier years, the TEM Gemini Centre has contributed to a large number of high school visits through 2018. In September we had a stand about TEM and gave lab tour to pupils attending the Researchers Night in the Natural Science building. Further, a substantial number of international visitors had a tour of our facilities.

PEOPLE IN THE TEM GEMINI CENTRE IN 2018

Randi Holmestad (Prof., DP, NTNU / Leader TEM Gemini Centre)
Ton van Helvoort (Prof., DP, NTNU)
Knut Marthinsen (Prof., DMSE, NTNU)
Yanjun Li (Prof., DMSE, NTNU)
Per Erik Vullum (Research scientist, SINTEF and Assoc. Prof. II, DP, NTNU)
Bjørn Gunnar Soleim (Senior engineer, DP, NTNU)
Ragnhild Sæterli (Senior engineer, DP, NTNU)
Yingda Yu (Senior engineer, DMSE, NTNU)
Sigmund J. Andersen (Senior research scientist, SINTEF)
Jesper Friis (Senior research scientist, SINTEF)
Calin Marioara (Senior research scientist, SINTEF)
Ruben Bjørge (Research scientist, SINTEF)
Sigurd Wenner (Research scientist, SINTEF)
Eva Anne Mørtsell (Postdoc, DMSE, NTNU)
Marat Gazizov (Postdoc, DP, NTNU)
Maria Tsoutsouva (Postdoc, DP, NTNU)
Maryam Vatanparast (PhD student, DP, NTNU)
Julie Stene Nilsen (PhD student, DP, NTNU)
Aleksander Mosberg (PhD student, DP, NTNU)
Emil Christiansen (PhD student, DP, NTNU)
Adrian Lervik (PhD student, DP, NTNU)
Jonas Frafjord (PhD student, DP, NTNU)
Tina Bergh (PhD student, DP, NTNU)
Jonas Sunde (PhD student, DP, NTNU)
Hogne Lysne (PhD student, DP, NTNU)
Håkon Wiik Ånes (PhD student, DMSE, NTNU)
Inger-Emma Nylund (PhD student, DMSE, NTNU)
Johanna Neumann (Master student, DP, NTNU)
Susanne Araya (Master student, DP, NTNU)
Sigurd Ofstad (Master student, DP, NTNU)
Elisabeth Thronsen (Master/PhD student, DP, NTNU)
Andreas Toresen (Master student, DP, NTNU)
Jørgen Sørhaug (Project student, DP, NTNU)
Gregory Nordahl (Project student, DP, NTNU)
Edwin Nongba Traore (Project student, DP, NTNU)
Haakon Tvedt (Project student, DP, NTNU)
Kasper Aas Hunnestad (Project student, DP, NTNU)
Daniel Martin Lundeby (Project student, DP, NTNU)
Ingvild Hansen (Project student, DP, NTNU)
Simon Høgås (Project student, DP, NTNU)
Yngve Maximilian Ender (Project student, DP, NTNU)



SELECTED SCIENTIFIC PAPERS



NANO LETTERS

Letter

Cite This: *Nano Lett.* 2018, 18, 2304–2310

pubs.acs.org/NanoLett

Single-Mode Near-Infrared Lasing in a GaAsSb-Based Nanowire Superlattice at Room Temperature

Dingding Ren,[†] Lyubomir Ahtapodov,[†] Julie S. Nilsen,[‡] Jianfeng Yang,[§] Anders Gustafsson,[∥] Junghwan Huh,[†] Gavin J. Conibeer,[§] Antonius T.J. van Helvoort,[‡] Bjørn-Ove Fimland,[†] and Helge Weman*,[†]

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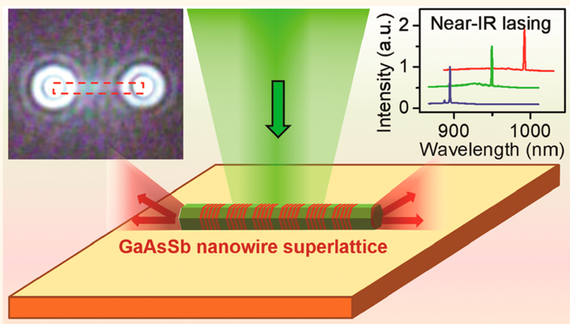
[§]Australian Centre for Advanced Photovoltaics, University of New South Wales, Sydney, New South Wales 2052, Australia

[∥]Solid-State Physics and NanoLund, Lund University, Box 118, SE-22100 Lund, Sweden

Supporting Information

ABSTRACT: Semiconductor nanowire lasers can produce guided coherent light emission with miniaturized geometry, bringing about new possibilities for a variety of applications including nanophotonic circuits, optical sensing, and on-chip and chip-to-chip optical communications. Here, we report on the realization of single-mode and room-temperature lasing from 890 to 990 nm, utilizing a novel design of single nanowires with GaAsSb-based multiple axial superlattices as a gain medium under optical pumping. The control of lasing wavelength via compositional tuning with excellent room-temperature lasing performance is shown to result from the unique nanowire structure with efficient gain material, which delivers a low lasing threshold of ~6 kW/cm^2 (75 μJ/cm^2 per pulse), a lasing quality factor as high as 1250, and a high characteristic temperature of ~129 K. These results present a major advancement for the design and synthesis of nanowire laser structures, which can pave the way toward future nanoscale integrated optoelectronic systems with superior performance.



KEYWORDS: *Nanowire laser, GaAsSb, superlattice, molecular beam epitaxy*

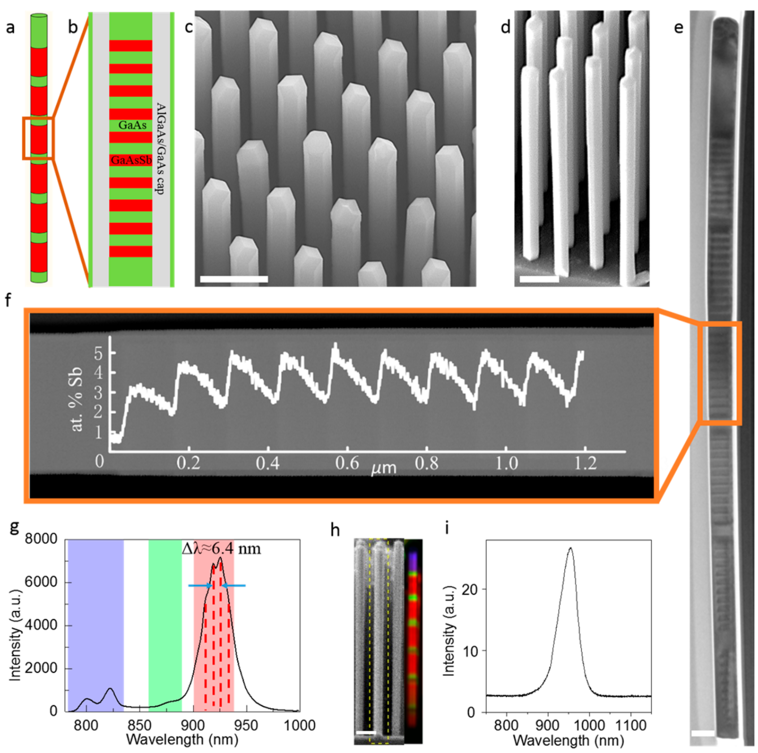


Figure 1. Structural and optical properties of the NW superlattice laser (sample B). (a) Schematics of the NW consisting of six periodic GaAsSb- based superlattices (red) separated by GaAs spacers (green) and (b) schematic design of each superlattice. Tilted-view SEM images at (c) 20° and (d) 30° of an as-grown NW array on a Si(111) substrate with an EBL patterned oxide mask from the center and cleaved edge, respectively. (e) Cross-section BF-TEM image of a representative NW. (f) Cross-section HAADF-STEM with the correlated EDX Sb composition profile of the fourth NW superlattice, marked by the orange rectangle in panel e. (g) Average low-temperature CL spectrum of the as-grown NW array with color coding for three different spectral regions as used to construct the CL false-color image in panel h. The vertical red dashed lines represent the periodic peaks of the spontaneous emission originating from the FP modes of the NW laser cavity. (h) Cross-section SEM image of the as-grown NWs and the corresponding low-temperature CL false-color image. (i) Room-temperature μ-PL spectrum of the as-grown NW array at a pumping and excitation power density of 125 W cm^{-2}. The scale bars are 1 μm, 1 μm, 500 nm, and 1 μm in panels c, d, e, and h, respectively.

ACS NANO
Cite This: ACS Nano 2018, 12, 2998−3009
www.acsnano.org

High Interfacial Charge Storage Capability of Carbonaceous Cathodes for Mg Batteries

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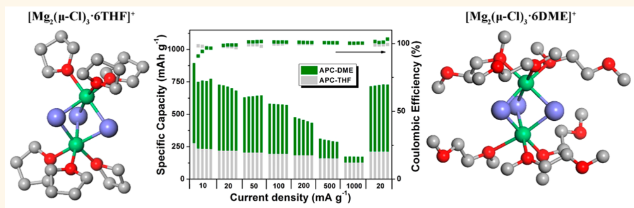
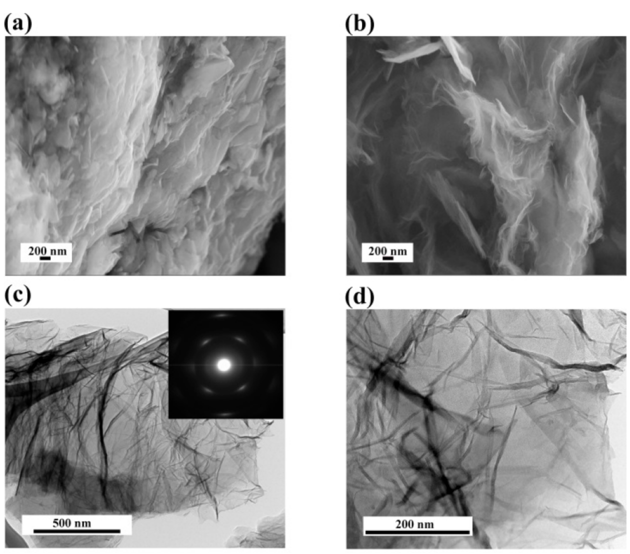
[∥]The Sixth Element Materials Technology Co., Ltd., 213000 Changzhou, China

Supporting Information



ABSTRACT: A rechargeable Mg battery where the capacity mainly originates from reversible reactions occurring at the electrode/electrolyte interface efficiently avoids the challenge of sluggish Mg intercalation encountered in conventional Mg batteries. The interfacial reactions in a cell based on microwave-exfoliated graphite oxide (MEGO) as the cathode and all phenyl complex (APC) as electrolyte are identified by quantitative kinetics analysis as a combination of diffusion-controlled reactions involving ether solvents (*esols*) and capacitive processes. During magnesiation, *esols* in APC electrolytes can significantly affect the electrochemical reactions and charge transfer resistances at the electrode/electrolyte interface and thus govern the charge storage properties of the MEGO cathode. In APC−tetrahydrofuran (THF) electrolyte, MEGO exhibits a reversible capacity of ∼220 mAh g^{-1} at 10 mA g^{-1}, while a reversible capacity of ∼750 mAh g^{-1} at 10 mA g^{-1} was obtained in APC-1,2-dimethoxyethane (DME) electrolyte. The high capacity improvement not only points to the important role of the *esols* in the APC electrolytes but also presents a Mg battery with high interfacial charge storage capability as a very promising and viable competitor to the conventional intercalation-based batteries.

KEYWORDS: *Mg battery, microwave-exfoliated graphite oxide, diffusion-controlled reactions, ether solvents, capacitive processes*



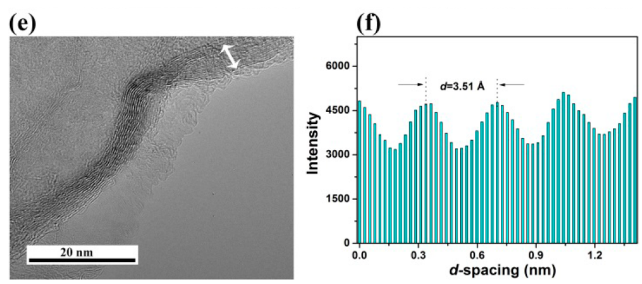


Figure 1. Secondary electron images of (a) graphite and (b) MEGO. (c and d) Typical bright-field (BF) TEM images of the MEGO nanosheets. The inset in (c) shows the electron diffraction pattern from the region shown in the BF TEM image. (e) BF TEM image of nanosheets in the MEGO sample where the edges of the nanosheets are bent such that the atomic planes are parallel to the electron beam. (f) Intensity profile across some of the atomic planes that are oriented in cross-section showing the interatomic distances.

Materials Characterization 142 (2018) 458–469

Contents lists available at ScienceDirect

Materials Characterization

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The evolution of precipitate crystal structures in an Al-Mg-Si(-Cu) alloy studied by a combined HAADF-STEM and SPED approach

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This work presents a detailed investigati5on into the effect of a low Cu addition (0.01 at.%) on precipitation in an Al-0.80Mg-0.85Si alloy during ageing. The precipitate crystal structures were assessed by scanning transmission electron microscopy combined with a novel scanning precession electron diffraction approach, which includes machine learning. The combination of techniques enabled evaluation of the atomic arrangement within individual precipitates, as well as an improved estimate of precipitate phase fractions at each ageing condition, through analysis of a statistically significant number of precipitates. Based on the obtained results, the total amount of solute atoms locked inside precipitates could be approximated. It was shown that even with a Cu content close to impurity levels, the Al-Mg-Si system precipitation was significantly affected with overageing. The principal change was due to a gradually increasing phase fraction of the Cu-containing Q′-phase, which eventually was seen to dominate the precipitate structures. The structural overtake could be explained based on a continuous formation of the thermally stable Q′-phase, with Cu atomic columns incorporating less Cu than what could potentially be accommodated.

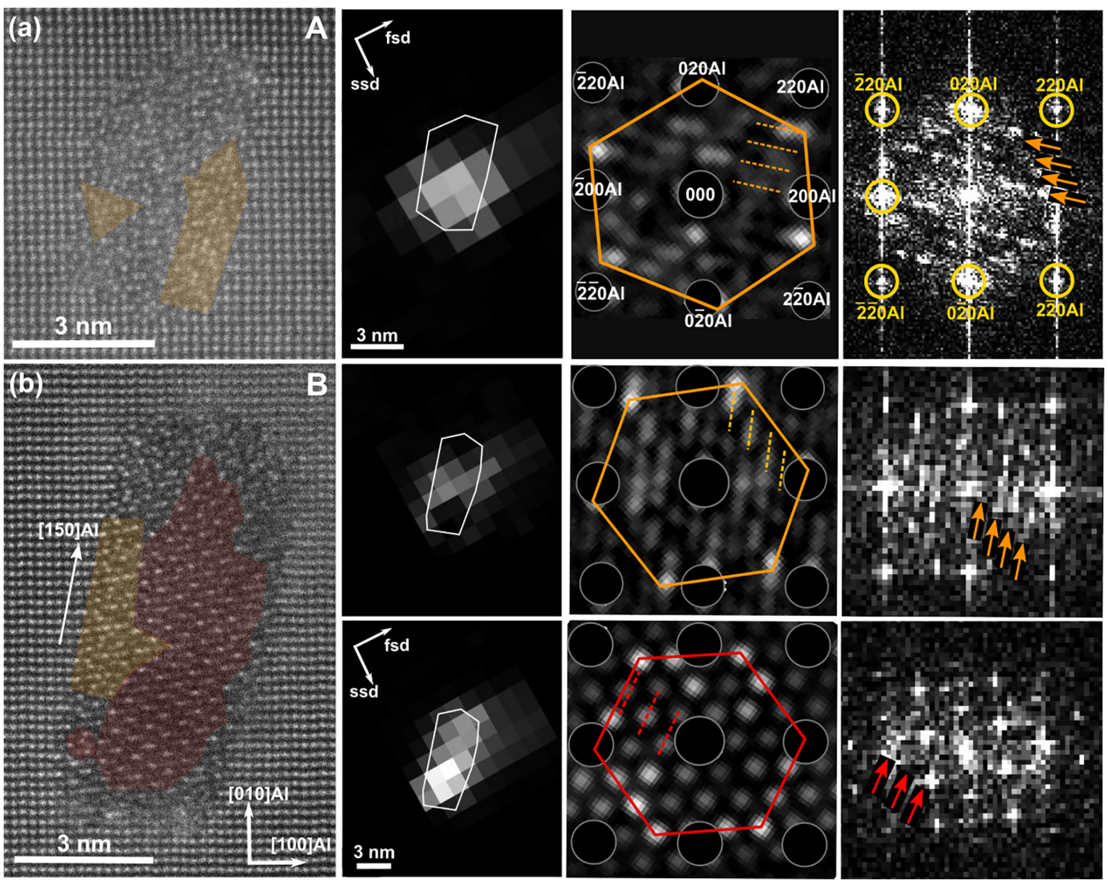

Fig. 7. (a) and (b) show HAADF-STEM lattice images and SPED decomposition results for precipitates labeled **A** and **B** in Fig. 6. The first column shows precipitate lattice images. The second and third columns show the main loading maps and associated component patterns from NMF decomposition, respectively. The fourth column shows FFTs of the lattice image areas highlighted. The red and orange highlighted regions show unit cells/sub-units of β'-2 and Q′, respectively. The outlines of the precipitate cross-sections are overlaid on the loading maps. Characteristic precipitate features in component patterns and image FFTs are indicated.

REVIEW

Al–Mg–Si Alloys

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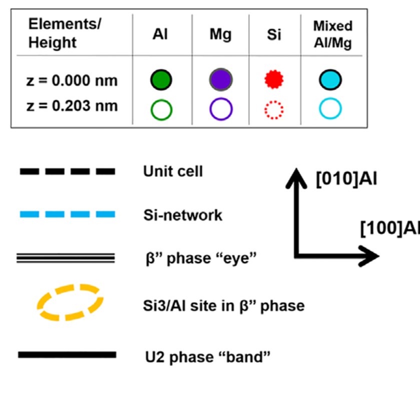
Atomic Structures of Precipitates in Al–Mg–Si Alloys with Small Additions of Other Elements

Takeshi Saito, Eva A. Mørtsell, Sigurd Wenner, Calin D. Marioara, Sigmund J. Andersen, Jesper Friis, Kenji Matsuda, and Randi Holmestad*

In Al–Mg–Si alloys, additions of only a few weight percent of Mg and Si enable formation of hardening precipitates during heat treatment. The precipitation is complex and is influenced by chemical compositions and thermo-mechanical treatment. Structural analysis at the atomic scale has played an important role for understanding the Al–Mg–Si system. This review paper gives a summary of the influence of elements on the precipitate structures of Al–Mg–Si alloys at the atomic scale. The structures are modified by small additions of different elements, but all the encountered precipitates are structurally connected with the Si network, except for the main hardening phase which exhibit a partially discontinuous Si network. The influence of the selected elements (Li, Cu, Zn, Ge, Ag, Ni, Co, and Au) is discussed in detail.



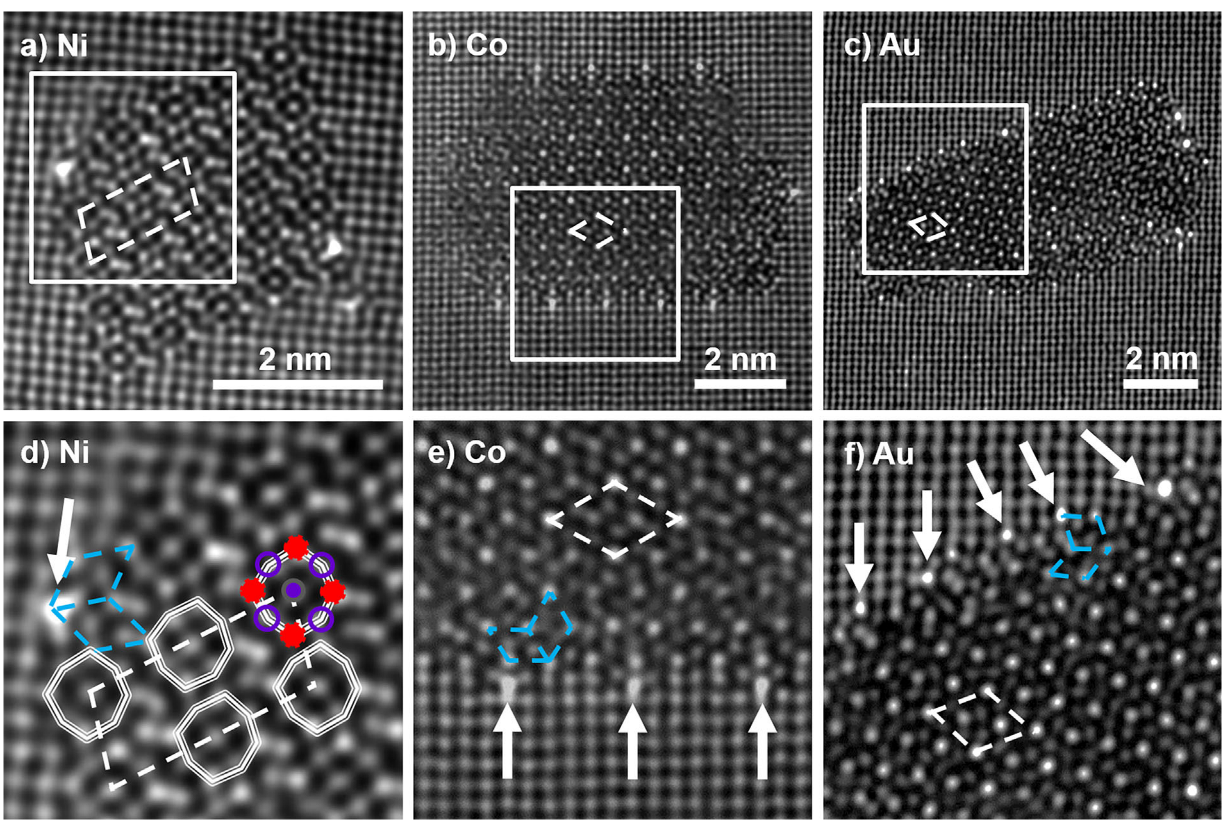


Figure 17. HAADF-STEM image of a) Ni, b) Co, and c) Au containing precipitate cross sections, taken from Al–1.0 wt%Mg2Si alloys having 0.1 wt%Ni, 0.1 wt%Co, and 0.01 wt%Au, respectively, and d–f) corresponding to the magnified images in white solid square of a–c), respectively. The images have been filtered using Fast Fourier transform with a circular band pass mask that removed all periods (noise) shorter than 0.15 nm. White arrows show Ni, Co, or Au containing atomic sites in each image. White dashed lines show unit cell of β" in a) and d) and unit cell of β' in b), c), e) and f). Blue dashed lines show the Si network. See the unit cells and the legend in **Figure 4**.

Materials Characterization 144 (2018) 522–531

Contents lists available at ScienceDirect

Materials Characterization

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Lattice rotations in precipitate free zones in an Al-Mg-Si alloy

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Scanning precession electron diffraction and automated crystal orientation mapping in a transmission electron microscope (TEM) have been applied to quantitatively study the crystal orientation of precipitate free zones (PFZs) of four GB regions in an AA6060 alloy in peak aged condition (temper T6) after uniaxial compression to 20% engineering strain. The PFZ width in the alloy was found to be $w = 170 \pm 40$ nm. The results show that some PFZs develop significant misorientations relative to their parent grain, and represent, to the best knowledge of the authors, the first quantitative evidence of this. This misorientation may either be constant inside a particular PFZ, making it appear like a band or a very elongated subgrain, or be partitioned in discrete regions with a diameter comparable to the PFZ width, making the former PFZ into a collection of small grains. The band-like PFZ observed in this work had a misorientation relative to its parent grain of ≈ 7°, while the grain-like PFZ had grains with misorientations between ≈ 12° and ≈ 20° relative to its parent grain. The other PFZs that were observed had only limited misorientations relative to their parent grains, and had either dislocations perpendicular to the GB plane or a dislocation wall at the transition region. A general TEM study of the material at various engineering strains was also conducted and suggests that grain-like PFZs are more frequent for larger strains, indicating that the different PFZ features are likely due to different strain localisation in individual PFZs. This localisation is expected to be influenced by the orientation of the loading axis relative to crystal orientations and GB planes. It is also suggested that the different PFZ features engender different work hardening rates and possibly affect nucleation of intergranular fracture. The results support previous studies on the microstructure evolution of PFZs in age-hardenable aluminium alloys during deformation.

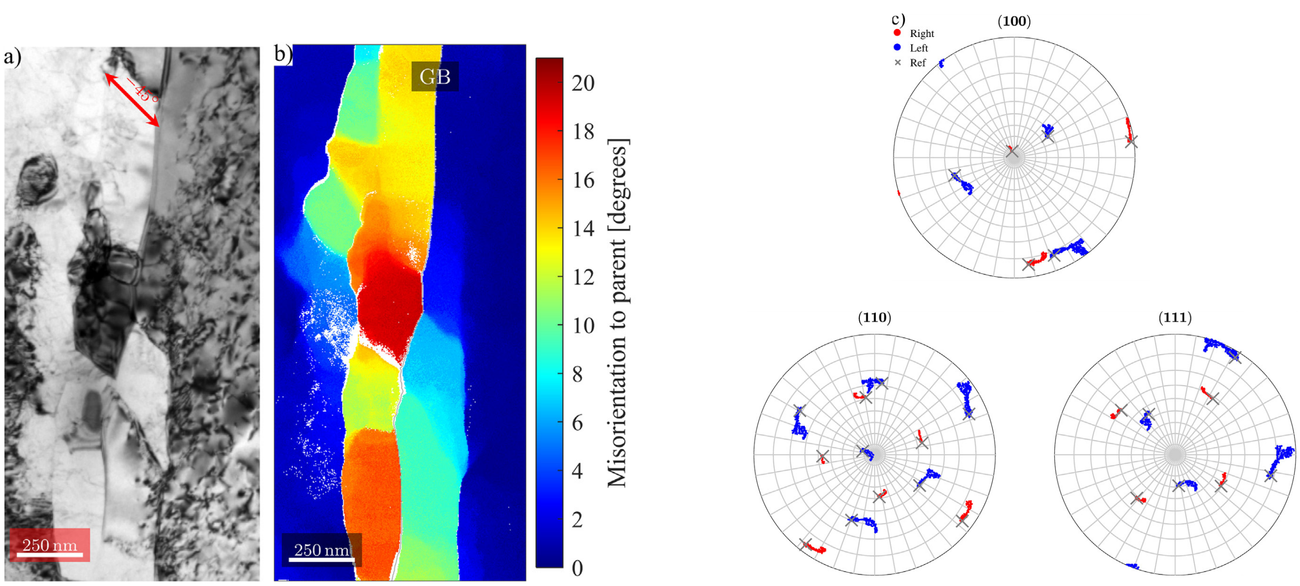

Fig. 8. PFZ with small regions of high misorientations relative to parent grain orientations, i.e. *grains*, observed by BF TEM and SPED orientation mapping. The parent-parent misorientation is $\varphi = 38°$, and the CA-GB angle is $\Phi = 45°$ as indicated by the double arrow in a). The PFZs to either side of the GB have formed regions of different contrast in the BF TEM image shown in a). In b), the misorientation angles in degrees of each grain are shown. The GB plane is shown as a thin grey line. Pole figures of the (100), (110), and (111) poles are given in c), where 103805 and 82316 data points are included for the left-hand and right-hand sides, respectively. The parent orientation of the two grains are marked by crosses.

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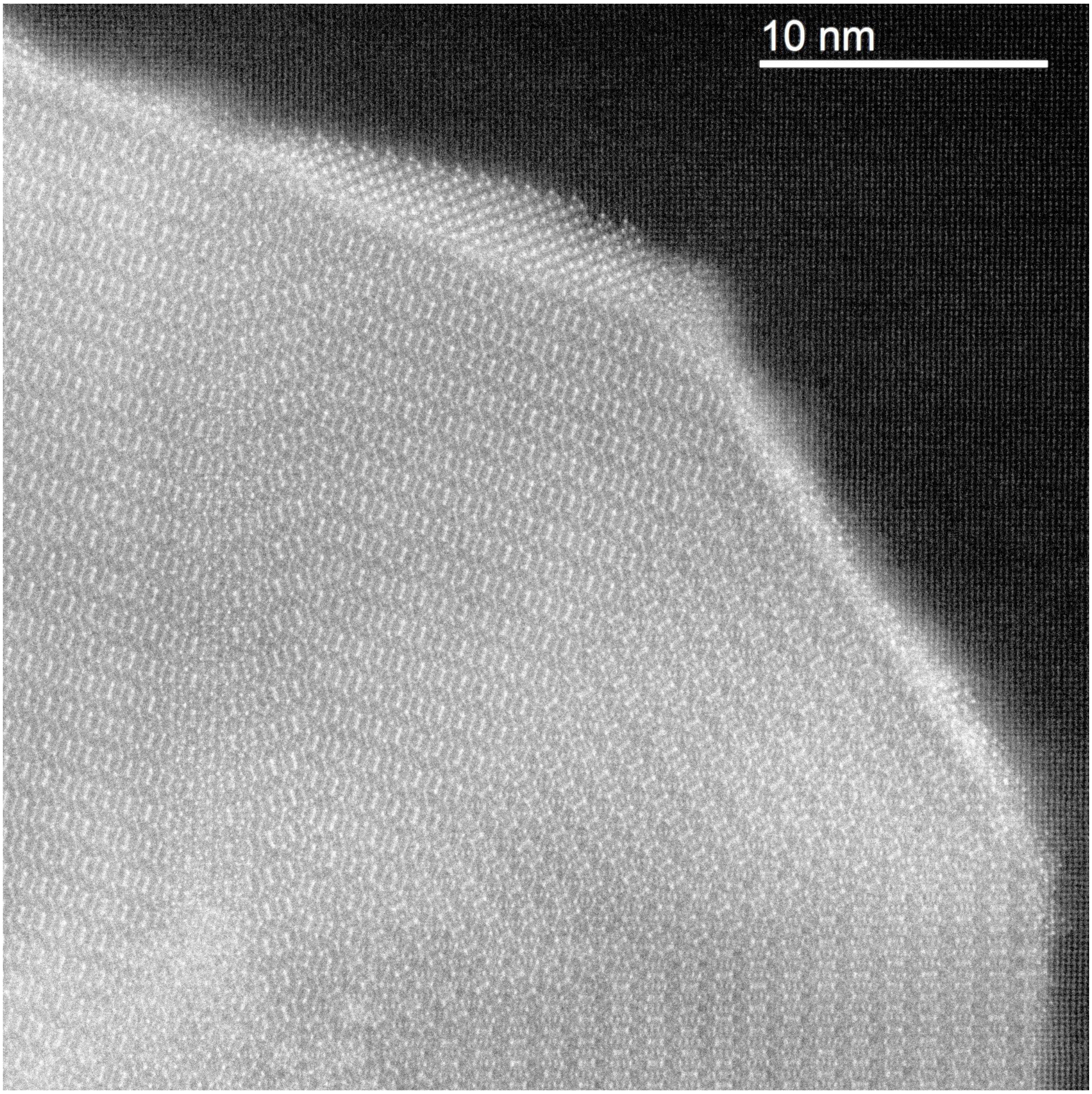

HAADF-STEM image of a T-($Al_{20}Cu_2Mn_3$) phase dispersoid exhibiting internal faulting, and with S-(Al_2CuMg) phase precipitates having formed at the interface towards Al by J.K. Sunde.

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J.K. **Sunde**, C.D. **Marioara**, A. **van Helvoort**, R. **Holmestad**, "Studying the effect of low Cu additions in 6082 alloys", *Third INTPART Workshop between NTNU*, University of Toyama and Tokyo Tech, Japan 7-8 May, 2018.

J.K. **Sunde**, C.D. **Marioara**, A.T.J. **van Helvoort**, and R. **Holmestad**, "Quantitative analysis of precipitate crystal structure evolution in Al-Mg-Si-Cu alloys using scanning precession electron diffraction", *International Microscopy Congress*, Melbourne, Australia, 9-14 September, 2018.

J.K. **Sunde**, C.D. **Marioara** and R. **Holmestad**, The effect of low Cu additions in overaged Al-Mg-Si alloys studied by advanced transmission electron microscopy techniques. *International Conference on Aluminium Alloys (ICAA16)*, Montreal (2018).

E. **Thronsen**, C.D. **Marioara**, J.K. **Sunde**, I. **Erga,** K. **Minakuchi**, T. **Katsumi,** K. **Marthinsen,** K. **Matsuda**, R. **Holmestad**. Characterizing precipitates in a heavily deformed Al alloy, a TEM study. Third INTPART Workshop between NTNU, University of Toyama and Tokyo Tech, Japan 7-8 May, 2018.

T. Yamaguchi, J.K. **Sunde** and K. Ihara, Effect of grain boundary properties on the intergranular precipitation in 6022 aluminum alloy. *135th Conference of Japan Institute of Light Metals*, Utsunomiya University (2018).

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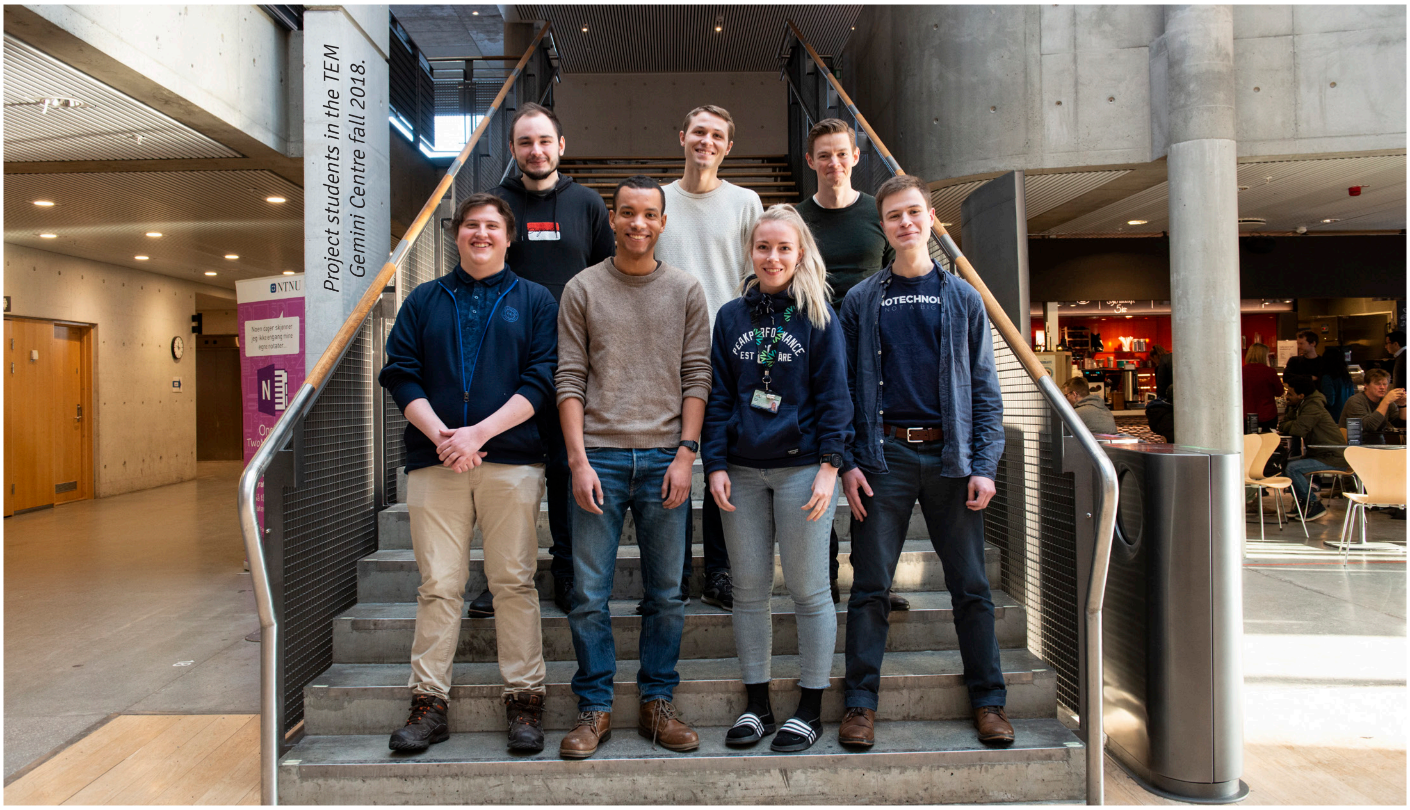

Project students in the TEM Gemini Centre fall 2018.

MASTER'S THESES

Susanne Araya, "Study of solid state phase transformations and precipitation in Ti-6Al-4V exposed to thermal cycling in additive manufacturing process", June 2018.
Elise Otterlei Brenne, "Atomic Resolution 3D Reconstruction of Crystal Grain Boundaries", June 2018.
Johanna Neumann, "The interface between graphene glass and self-catalyzed GaAsSb Nanowires", June 2018.
Inger-Emma Nylund, "Electron Microscopy Characterization of III-Nitride Nanowires grown on Graphene", January 2018
Sigurd Ofstad, "A Theoretic Study of β" Misfits and Strain in Al-Mg-Si Alloys - A Cluster-Based Approach Combining Density Functional Theory and Linear Elasticity", July 2018.
Elisabeth Thronsen, "The effect of deformation and natural ageing in an Al-Mg-Si-Cu alloy with high Cu-content: A transmission electron microscopy study", June 2018.
Andreas Toresen, "Transmission Electron Microscopy Characterisation of Lead-Free KNN Thin Films", June 2018.

PROJECT THESES

Yngve Maxmillian Ender, Characterization of Dispersoids and Precipitates in 5049 Aluminum Alloy, December 2018.
Ingvild Hansen, Correlated SEM/CL/TEM of MOCVD Grown GaN/AlGaN Nanowires on Graphene, December 2018.
Kasper Aas Hunnestad, TEM characterization of $ErMnO_3$, December 2018.
Simon Høgås, Towards large area semi-automatic crystallographic characterisation of semiconducting nanostructures by scanning precession electron diffraction, December 2018.
Daniel Martin Lundeby, Impact of data processing methods on TEM EDX quantification of $GaAs_{1-y}Sb_y$ nanowires, December 2018.
Gregory Nordahl, Transmission Electron Microscopy Characterization of As-Deposited and Heat Treated Additively Manufactured Ti-6Al-4V, December 2018.
Jørgen Andre Sørhaug, Transmission Electron Microscopy and Atomic Force Microscopy on Recrystallized Ag-implanted Si, December 2018.
Edwin Traore, A Transmission Electron Microscope Study of Precipitation Hardening in two Al-Mn-Cu-Mg-Si Alloys aimed for Brazed Heat Exchangers, December 2018.
Haakon Tvedt, Software implementation of methods for structural labeling of Al-Mg-Si-(Cu) alloy precipitates in HAADF-STEM images, December 2018.

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

http://www.ntnu.edu/geminicentre/tem