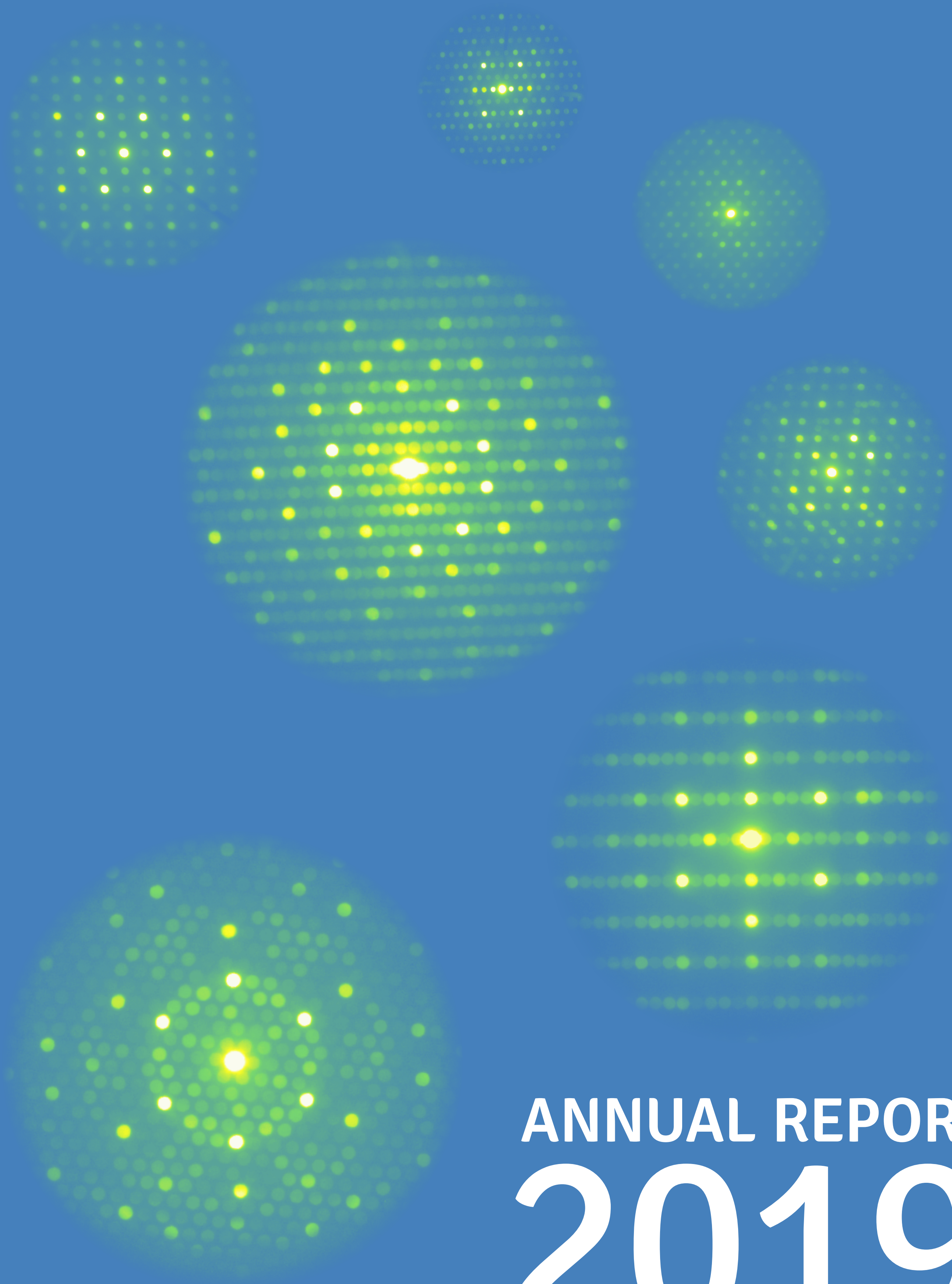
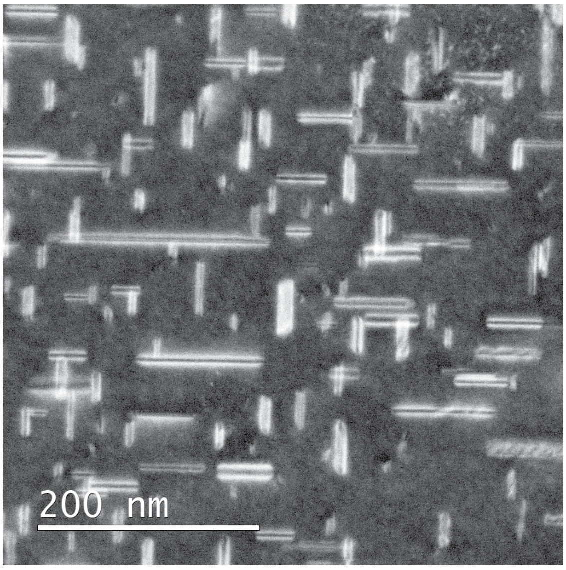
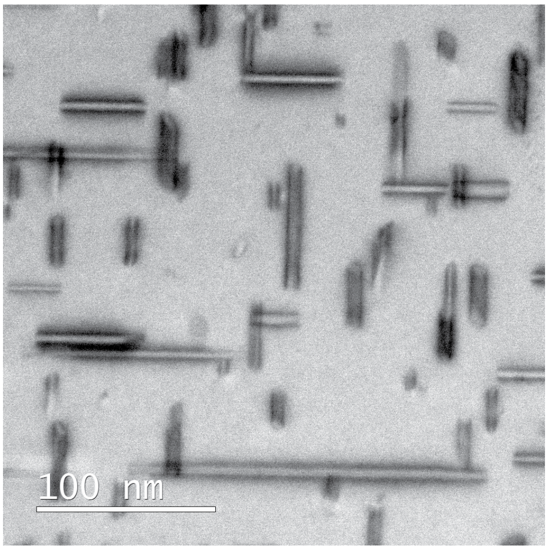


ANNUAL REPORT 2019

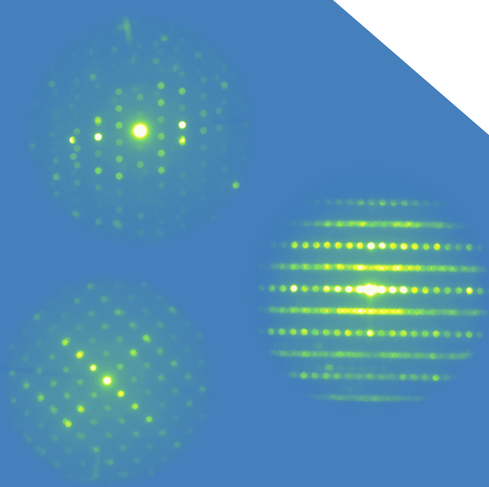
TEM GEMINI CENTRE

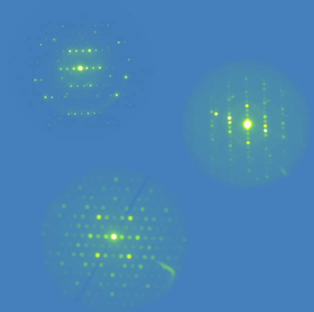
Department of Physics, NTNU;
Department of Materials Science and Engineering, NTNU;
Materials Physics, SINTEF Industry





HAADF- (top) and BF-STEM (bottom) images of a 6xxx aluminium alloy. The strain fields around the precipitates are visible. By Dipanwita Chatterjee.



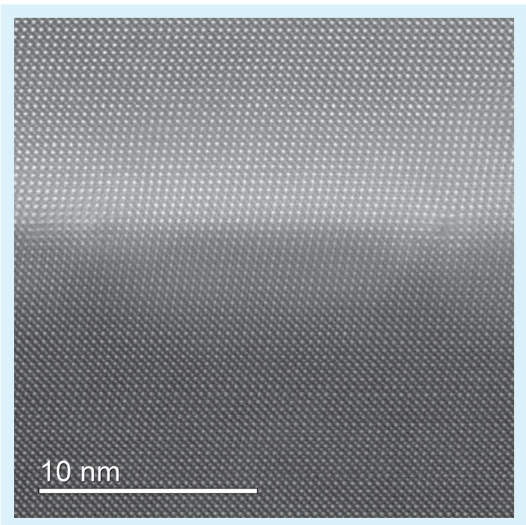


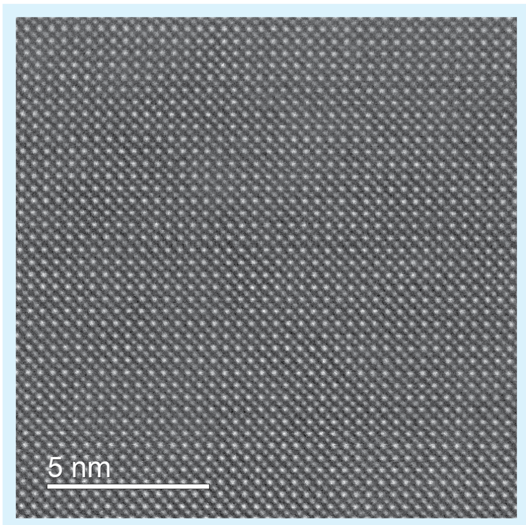
Graphic design: Tina Bergh.

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

Front and back page: Precession electron diffraction (PED) patterns of intermetallic phases aquired in nanobeam diffraction mode by Tina Bergh.

CONTENT





HAADF-STEM images of a $SrTiO_3$/$BaTiO_3$ (STO/BTO) thin film. Specimen preparation by Inger-Emma Nylund. Images by Dipanwita Chatterjee.

INTRODUCTION

2019 has again been a good year with further growth for the TEM Gemini Centre. We see that the structured work on building up routines, extending and further applying/ refining our competence around the NORTEM Trondheim node instruments gives return. We have more users of the infrastructure, more output in scientific papers and more projects than ever before. As a team we manage the big responsibility and long-term project to establish and have an effective role within Norway and in the NTNU/ SINTEF landscape for giving access to such expensive and advanced equipment. We see the consistent trend of increasingly high levels of use and quality of scientific and educational output achieved during the last years. Participation in the EU Horizon 2020 network ESTEEM3 has increased our visibility and international collaborations. 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. Furthermore, we organized a successful diffraction workshop Trondheim, the third in recent years, in June.

The total cost model for lab infrastructure introduced by NTNU is fully implemented in NORTEM. NTNU owns and runs the infrastructure, with SINTEF as an important user. The NORTEM project is a partnership between NTNU, UiO, and SINTEF, financed by the Research Council of Norway and the partners in 2011. In 2018 we submitted an application for a follow-up project NORTEM II, which unfortunately was not granted. Work is underway for a new proposal that will be submitted in 2020.

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 three ongoing KPN projects on aluminium with Norwegian aluminium industry – FICAL, AMPERE and SumAl. 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 is ongoing (to 2022) exchanging Japanese and Norwegian student between the two countries. In 2019 there were 2 Norwegian and 3 Japanese students participating.

As documented in this report, the Centre had 42 active hands-on users/operators, and served around 110 different projects, whose results have contributed to 51 journal publications (plus 6 in press) in 2019. 31 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, 2 PhD students and 8 MSc candidates were educated with TEM as a substantial part of their theses in 2019. The positive trend that the infrastructure gets more NTNU users from outside the Physics department continued in 2019. Three NTNU courses, with a total of approximately 130 students, used the facility. In the second half of 2019 we had nearly every week visitors or projects from abroad using the facilities. The yearly TEM introduction course was again held in September and had 24 participants. We had group meetings with presentations, and about 20 participants almost every week. Many guided tours for high school students and visitors to the microscopes took place. Most core members of the TEM Gemini Centre participated in international conferences and meetings in 2019.

Looking ahead into 2020 we warmly welcome Magnus Nord as a new associate professor in TEM based at Department of Physics and the Gemini Centre from April! We are happy that Magnus accepted the offer and looking forward to the new research activities (e.g. field mapping in functional materials) he will initiate and support. In March, we will take in use Norway's first direct electron detection camera, dedicated for electron diffraction. Furthermore, more detailed discussions are started on the Campus project and how rebuilding and constructions at Gløshaugen will affect our infrastructure. We will work strategically towards the long term aim of getting a new dedicated building for sensitive equipment at Gløshaugen campus.

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 in 2019. For more details, see our home page: ntnu.edu/ geminicentre/tem

- TEM Gemini Centre management, February 2020.

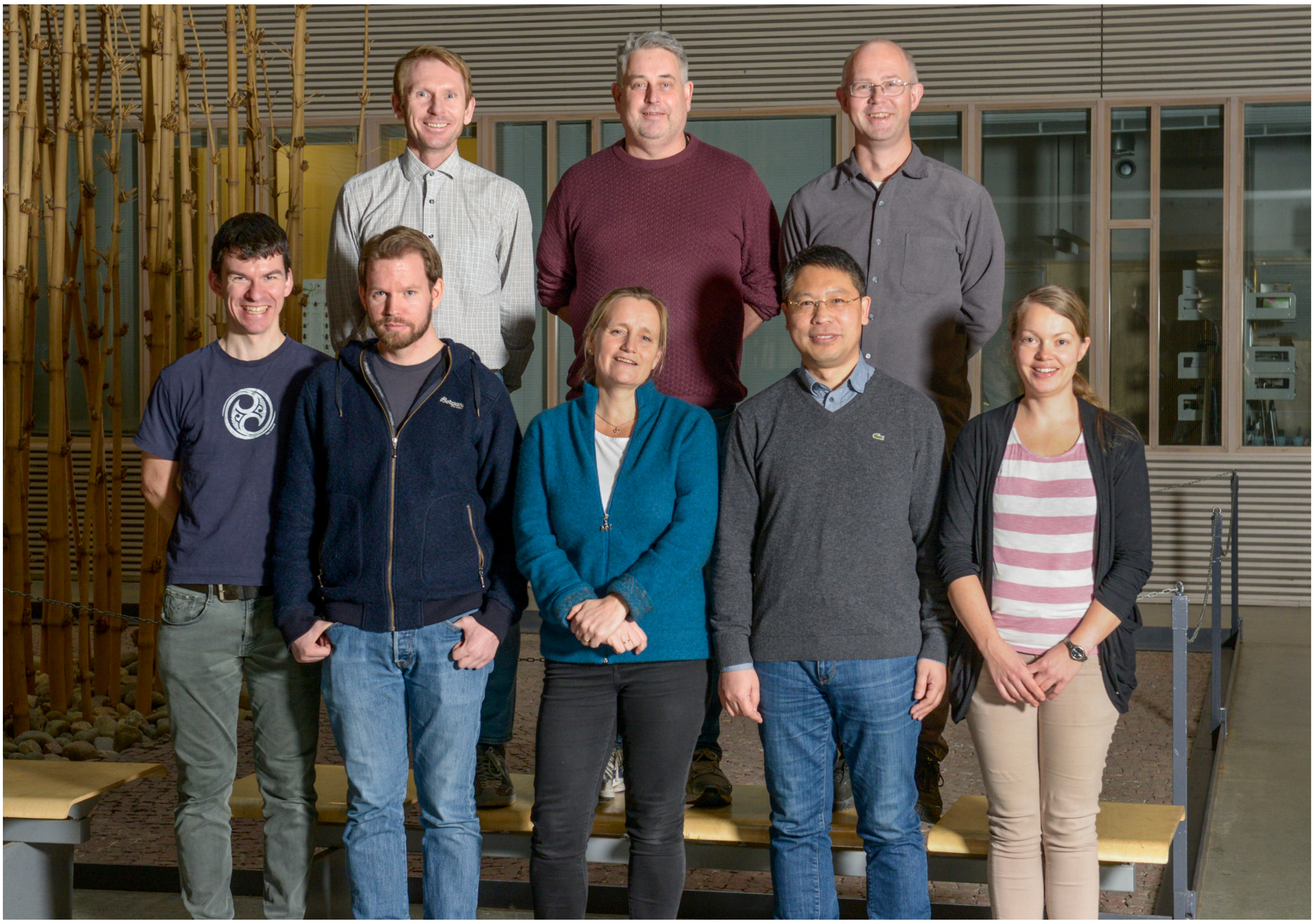
BOARD AND MANAGEMENT

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
- **Ruben Bjørge**, Materials Physics, SINTEF Industry (from September)



From left top: Per Erik Vullum (Research scientist/Associate Professor, Materials Physics, SINTEF Industry); Ragnar Fagerberg (Research manager, Materials Physics, SINTEF Industry); Ton van Helvoort (Professor, Physics, NTNU). From left bottom: Ruben Bjørge (Research scientict, Materials Physics, SINTEF Industry); Bjørn Gunnar Soleim (Senior engineer, Physics, NTNU); Randi Holmestad (Professor, Physics, NTNU, leader); Yanjun Li (Professor, Materials Science and Engineering, NTNU); Ragnhild Sæterli (Senior engineer, Physics, NTNU). Photo by Inger-Emma Nylund.

THE NORTEM PROJECT

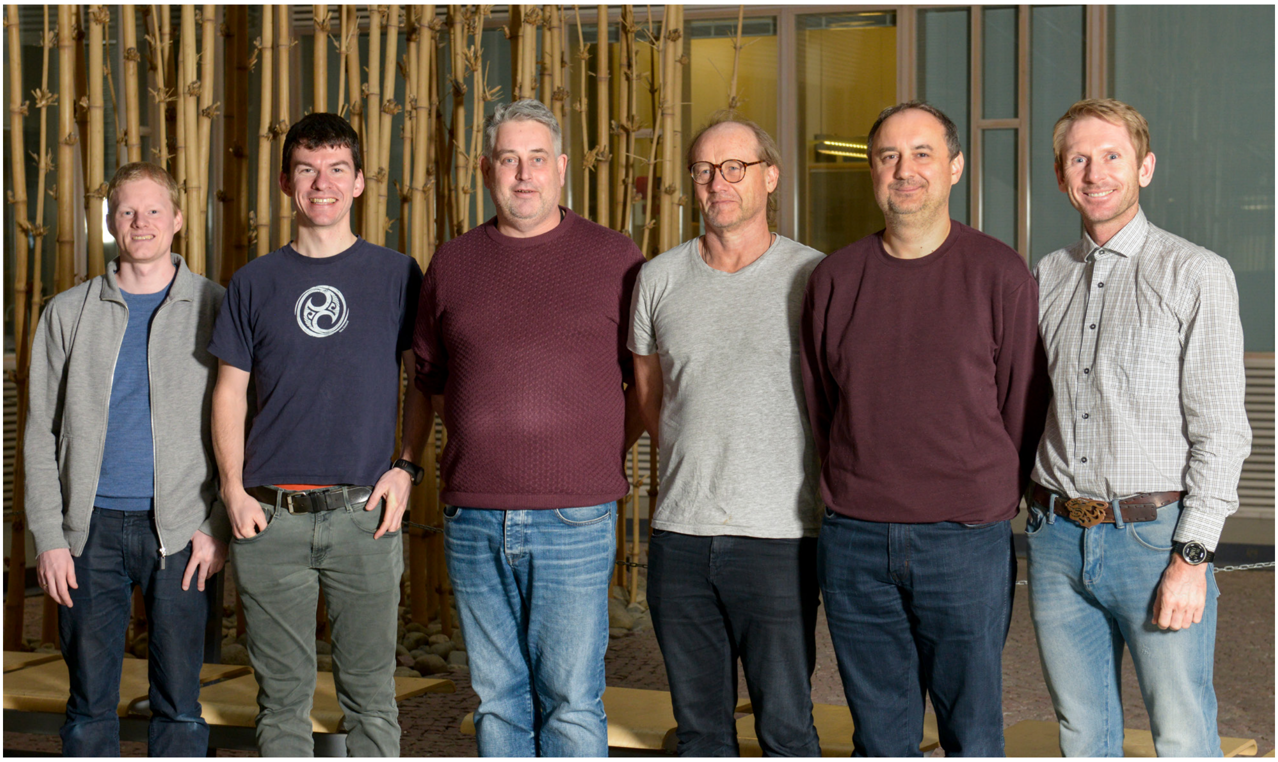
NORTEM (Norwegian Centre for Transmission Electron Microscopy) is a nationally coordinated large-scale infrastructure project (2011-2020) with three partners - SINTEF, NTNU and UiO, funded by the Research Council of Norway and the three partners. The budget for new equipment and the re-building in the project was about 75 MNOK for the two geographical nodes, Trondheim and Oslo. We have now been running the facility for close to six 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 included upgrading and new investments in both nodes. In the Trondheim node, we applied for detector upgrades on the existing instruments, in addition to a new TEM instrument specialised for spectroscopy. Unfortunately, this proposal was not granted. The Centre is working on a new proposal for NFRs INFRA program that will be handed in in 2020.

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.

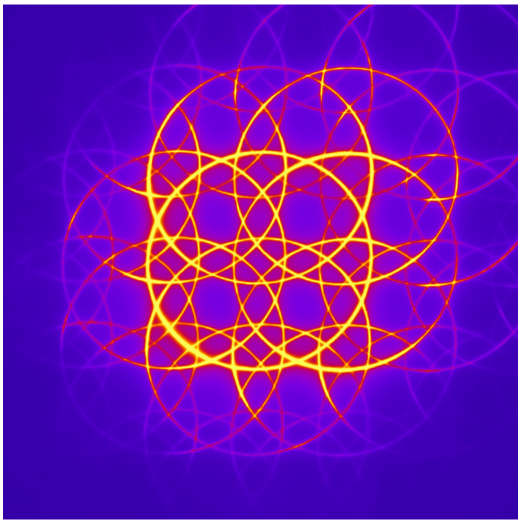
The combination of a research lab and a user facility requires a clear and sustainable running model, and the TEM Gemini Centre has spent considerable effort during the last years to establish a sound running model for the infrastructure. This is established and running well for some years now. NTNU 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 in the best way and the necessary future upgrades.

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 and Sigurd Wenner have been working as adjunct (affiliated) professors, 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. Photo by Inger-Emma Nylund.



'Precession on, descan off' in [001] Al by Tina Bergh.

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 Industry.

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.

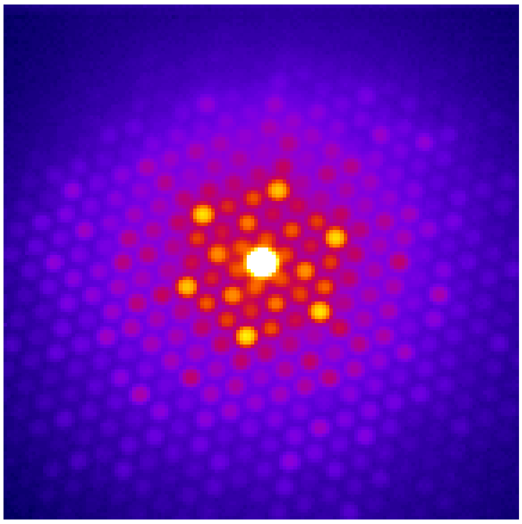
The Centre's research groups work within materials physics and materials science, studying a broad range of materials down to the nanometre and atomic level, where the main tool is the transmission electron microscope (TEM). The overall objective of the TEM Gemini Centre is to build and secure a robust 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 broader identity to the Centre's TEM infrastructure.

THE GEMINI CENTRE CONCEPT

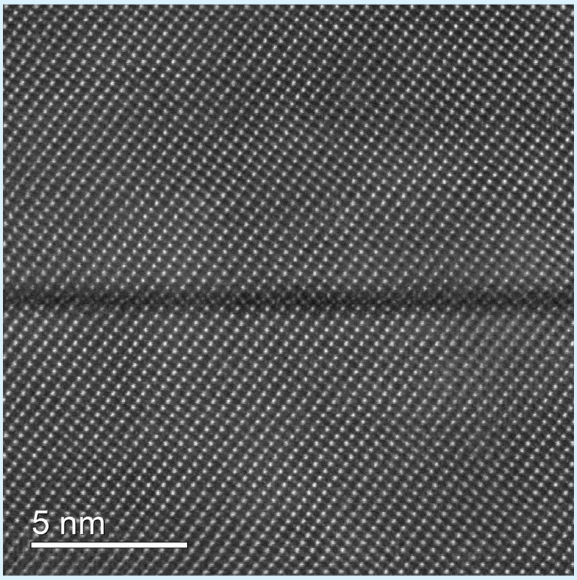
Gemini collaborations in general represent a model for strategic research coordination between parallel research groups at SINTEF, NTNU and UiO. The aim is to develop large-scale technical centers that produce higher quality results collectively than the individual groups would achieve independently. 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:

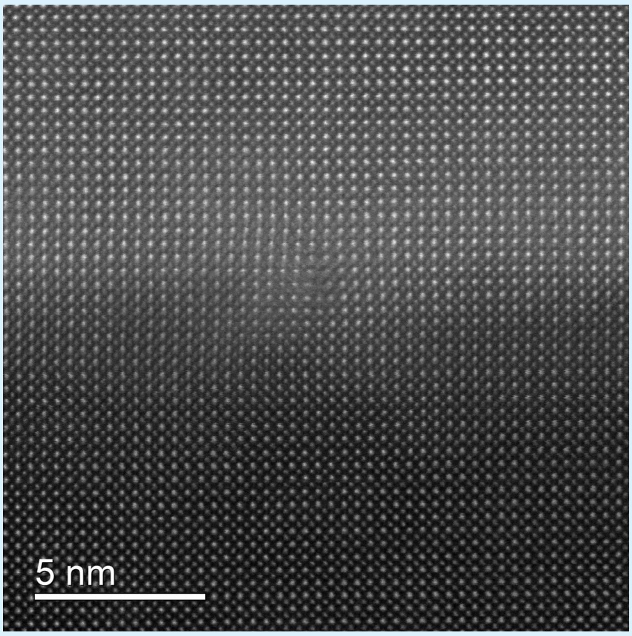
"Global excellence together".

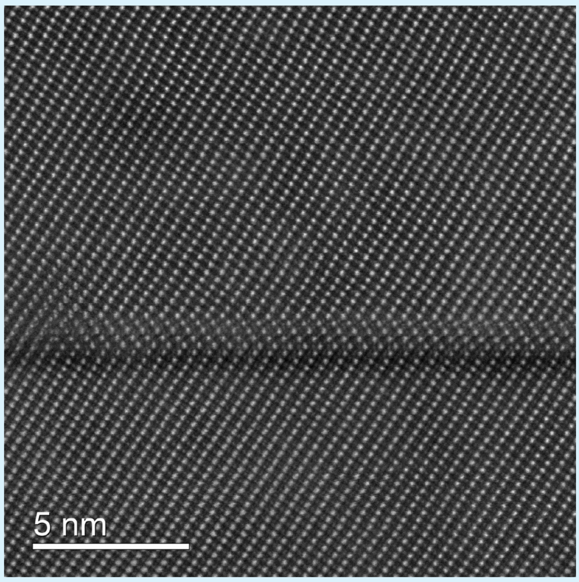
In order for the collaboration to work, the groups must undertake to adhere to joint strategic processes as the basis for their research planning, technical coordination in connection with large- scale projects, joint fora for concept development and information exchange, the collective presentation 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.



Summed SPED pattern of ferroelectric $Pb_5Ge_3O_{11}$ on [001] zone. Specimen preparation Kasper Hunnestad. Pattern by Ton van Helvoort.

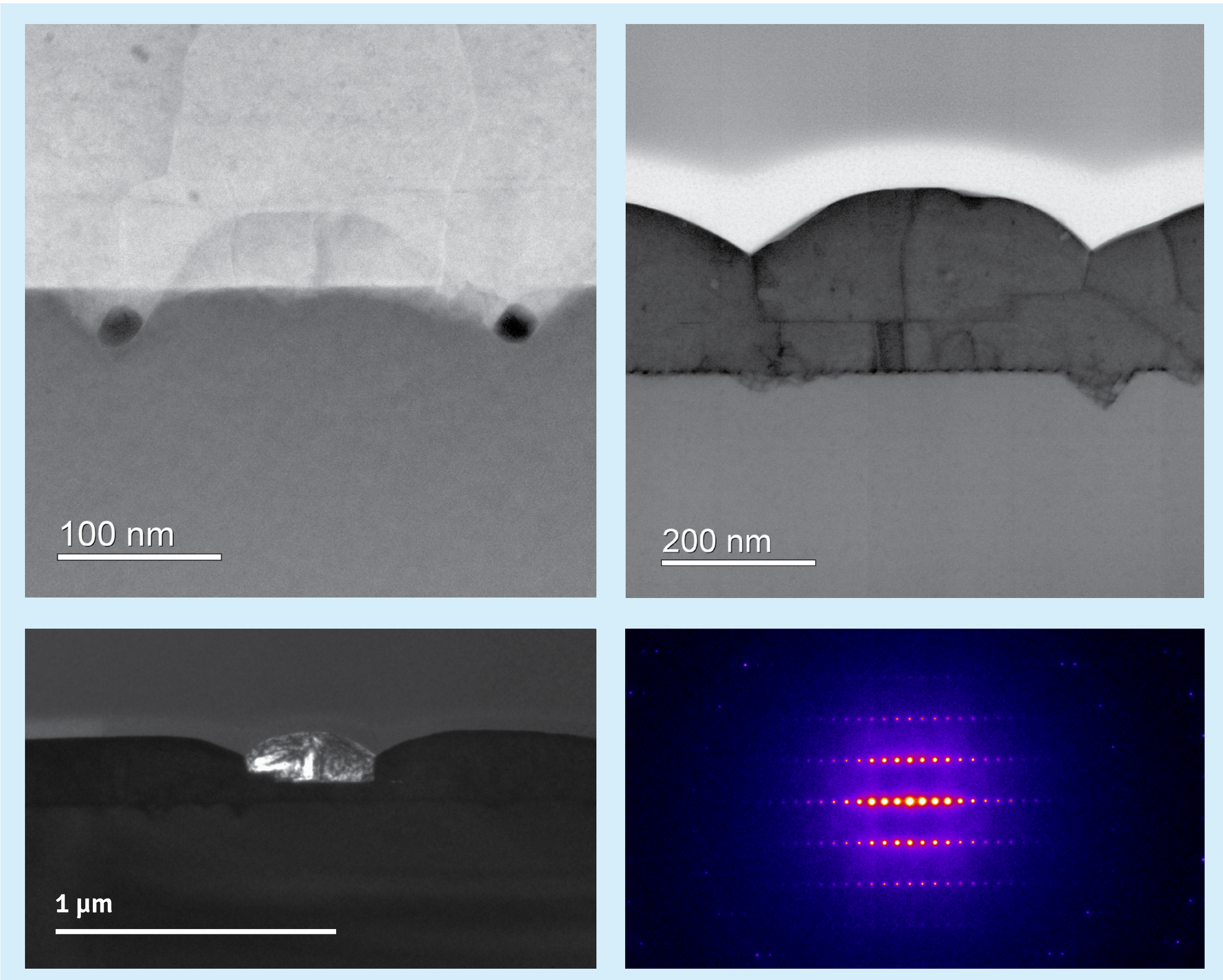






$SrTiO_3$ substrate with a chemical solution deposited $BaTiO_3$ thin film on top. Top and bottom: [111] oriented $BaTiO_3$ thin film with Ba-deficiency and a non-continuous crystal structure across an internal interface (due to the layered deposition technique). In the bottom image, the internal interface is thicker and has a twin structure. Middle: [001] oriented $SrTiO_3$ / $BaTiO_3$ interface with an edge dislocation. By Inger-Emma Nylund.

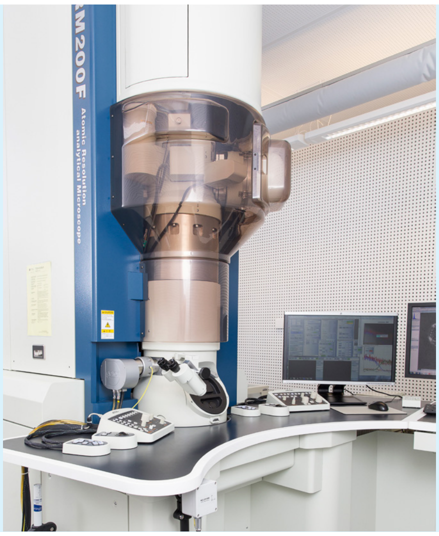
INSTRUMENTATION



Chemical solution deposited $BaTiO_3$ thin film on a $SrTiO_3$ substrate oriented to [111]: top left; defective SrTiO3 substrate surface, and top right and bottom left; BF-STEM and DF-TEM images of the same grain . Bottom right: SAED pattern of $Gd_2(MoO_4)_3$. By Inger-Emma Nylund.

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 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
- Medipix/QD TEM/STEM direct detector (from March 2020)

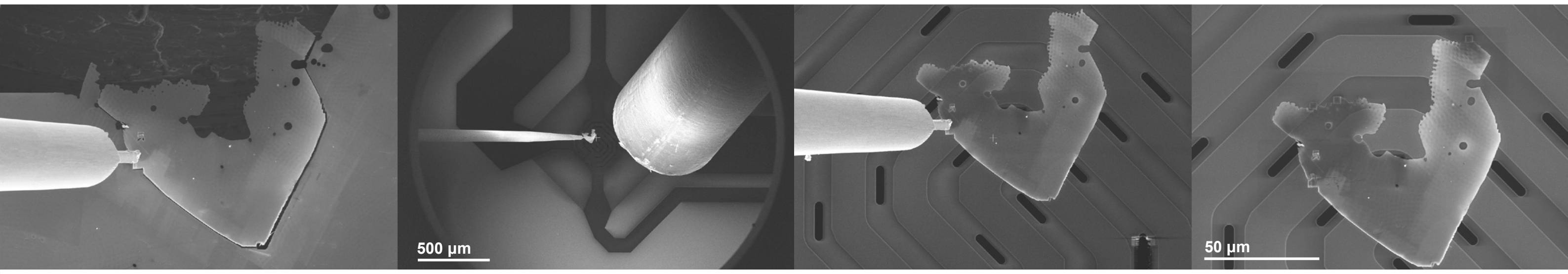
JEOL JEM-2100

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)

Photos by Ole Morten Melgård.



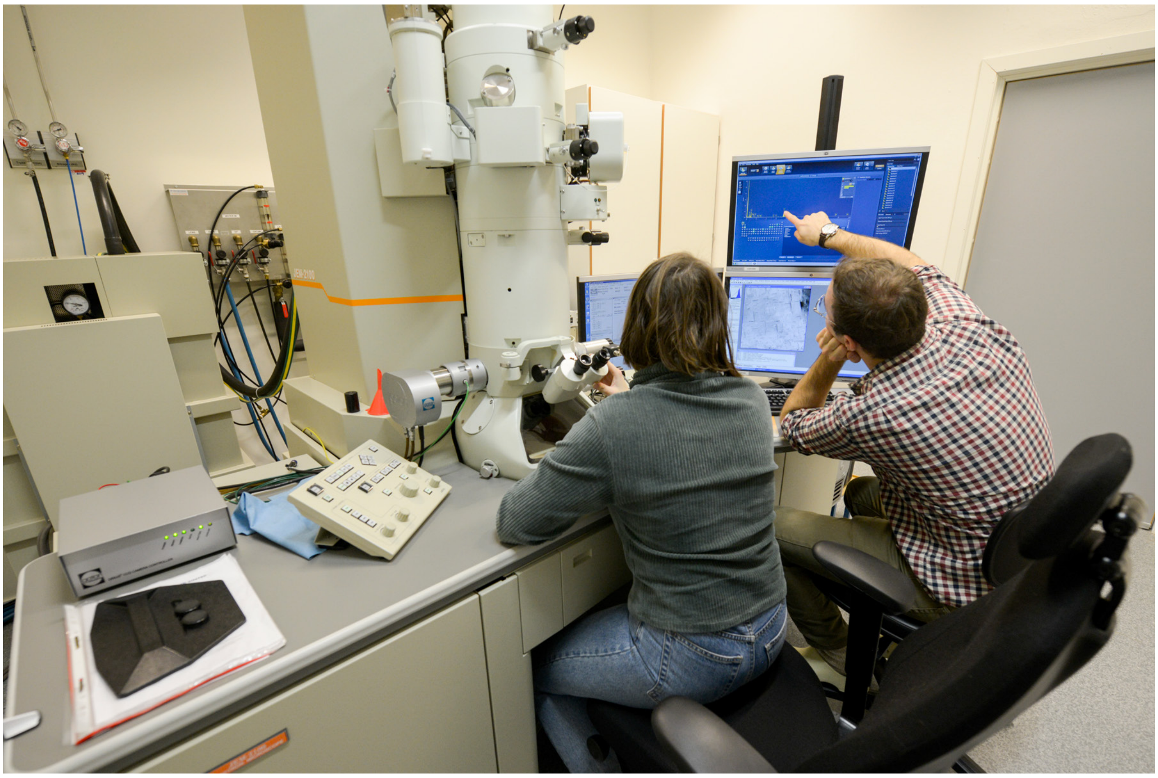
SEM images of attaching a [001] oriented grain from an electropolished 6082 Al alloy (by Jonas Sunde) to a DENS heating chip by using FIB. By Tina Bergh.

SPECIMEN PREPARATION

Given the high resolution of the TEM instruments, specimen quality is often the limiting factor. Also special holders require a dedicated specimen shape for optimal performance. 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. Investments were made in a new semi-automatic grinder and a new highspeed saw.

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. In 2019 an inert transfer set-up for FIB-based TEM prep became operative. The investment was done together with NTNU NanoLab and a special interest application area is advanced characterization of battery materials.

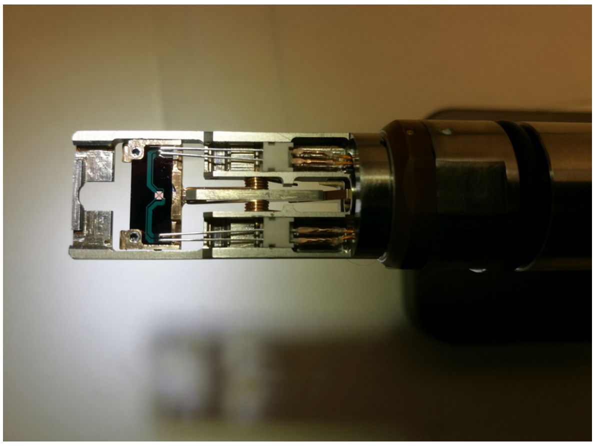
The electropolisher at DP is essential in producing high quality Al TEM specimens. A semi-automatic tripod polishing set-up is available for large area preparation of hard materials. For soft materials, such as polymers, ultramicrotomy is an essential technique. This equipment is also used for TEM preparation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



PhD students Elisabeth Thronsen and Christoph Hell at the 2100. Photo by Inger-Emma Nylund.

SPECIMEN HOLDERS

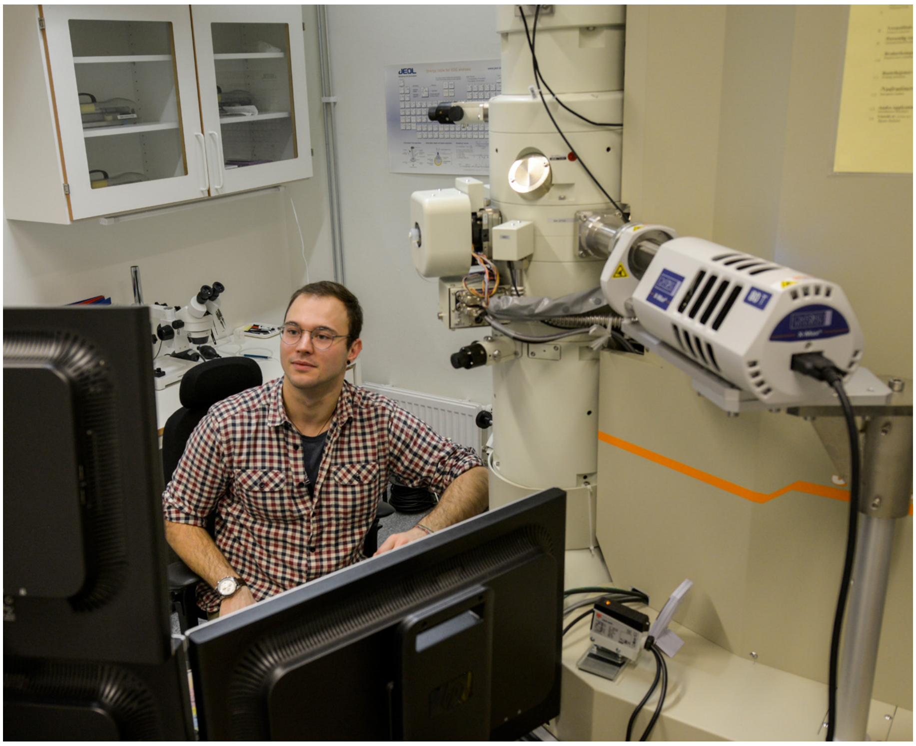
Each TEM has its own set of single and double tilt holders. A broad range of additional holders is available for use on all three microscopes. This includes a cold stage holder, a conventional heating holder, an environmental cell holder, an inert transfer holder, several tomography holders, two tilt-rotation holders and back-up double tilt holders. Another noteworthy holder is the 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. In 2019 a holder tip was manufactured for TEM characterization of atom probe tomography needles, to enable correlated structural and chemical studies of 100 nm sized volumes.



The DENS heating and biasing double tilt holder.

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. It also includes a more powerful workstation, dedicated to more demanding data processing. All acquisition software is accessible via offline licenses in the computer room. In addition, the TEM facility has dedicated a share in the NTNU's IDUN cluster for the most demanding simulations and processing the larger TEM data sets created at the facility.



PhD student Christoph Hell at the 2100. Photo by Inger-Emma Nylund.

MICROSCOPES IN THE TEM GEMINI CENTRE

Instrument	Location	Configuration	Installed
JEOL 2100	DP, Chemistry building I	LaB_6, 200 kV, STEM, GIF, EDS, Orius camera	2013
JEOL 2100F	DP, Chemistry building I	FEG, 200 kV, EDS, 2k CCD camera, ASTAR, tomography aquisition software	2013
JEOL ARM-200F	DP, Chemistry building I	Cold FEG,image and probe corrected, Quantum GIF, Centurio EDS, CCD cameras	2013

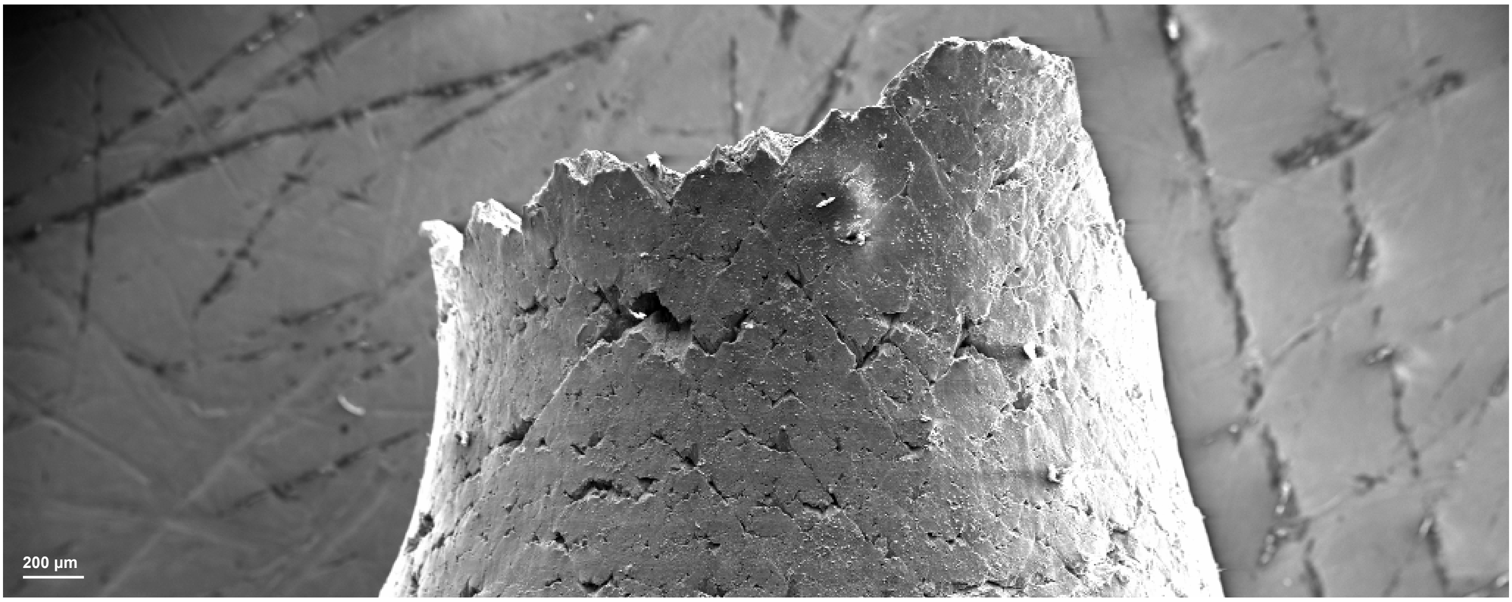
USER STATISTICS IN 2019

The total registered used time for the three instruments in 2019 was 4075 hours, including 125 non-paid hours used for testing, competence development, demonstrations and guided tours. Of the 3950 paid hours, the use by NTNU corresponds to 81 %, externals (with NTNU operator) 4 %, and SINTEF 15 %. NTNUs use is divided over seven departments, where the main use is from Department of Physics (79 % of NTNUs paid hours). About 110 different projects used TEM in 2019.

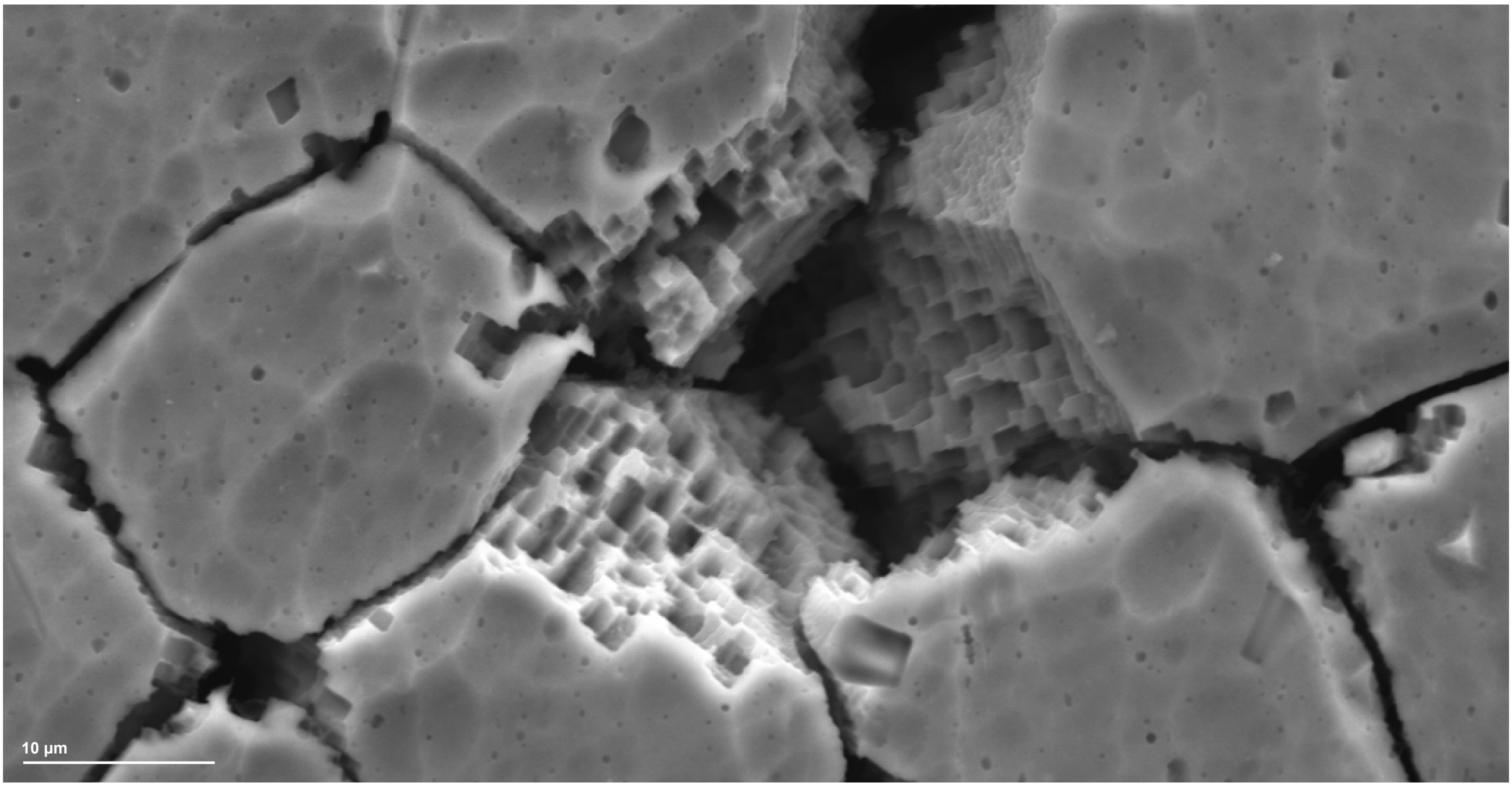
The infrastructure had 42 hands-on users in 2019, where 4 were based at SINTEF, 16 were PhD candidates and 13 Master students. The use from outside the Physics department has experienced an increase: 7 PhD candidates from other departments used the microscopes of the infrastructure in 2019.

Postdoc Dipanwita Chatterjee inserting a specimen in a double tilt holder.

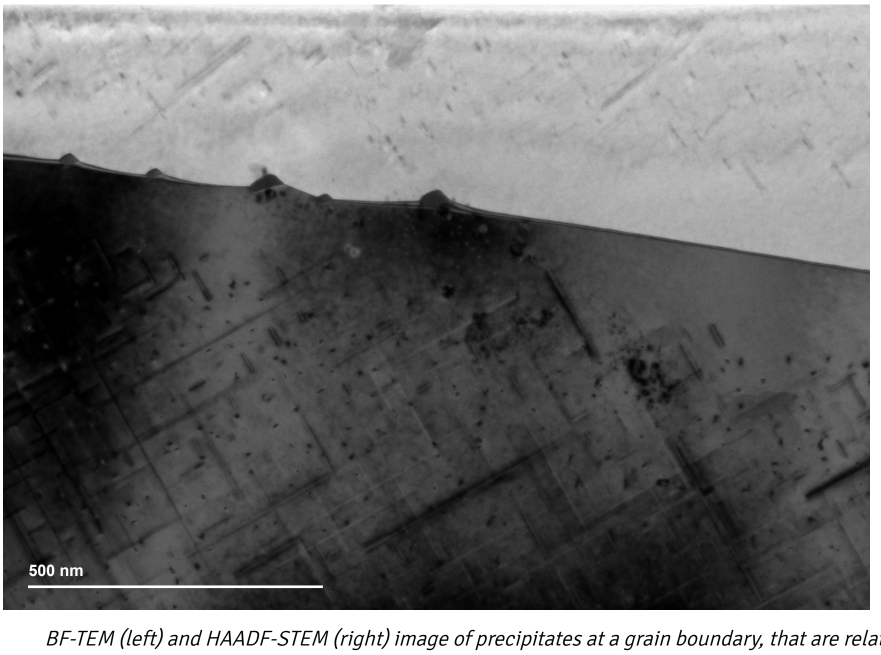
Microscope use in hours	ARM-200F	2100F	2100	SUM
SINTEF	375	96	141	612
NTNU – Physics	542	1115	720	2377
NTNU – Other departments	177	189	293	659
NTNU – Visitors from abroad	45	13	8	66
NTNU – Teaching lab	19	65	11	95
External	56	77	8	141
NTNU – Set-up/testing/training/demonstrations	13	84	28	125
Total use	**1227**	**1639**	**1209**	**4075**

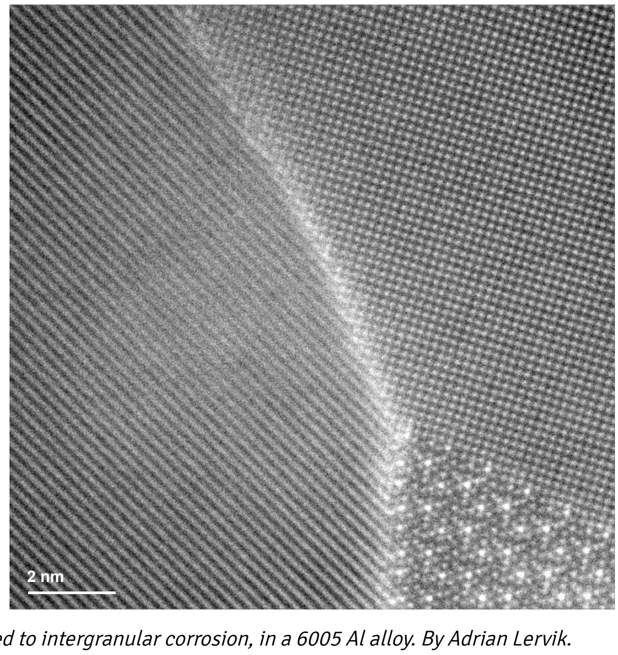


SEM image of an Al sample after a slow strain rate tensile test in a 3.5% NaCl solution. By Adrian Lervik.



SEM image of intergranular corrosion attacks in a 6005 Al alloy. By Adrian Lervik.

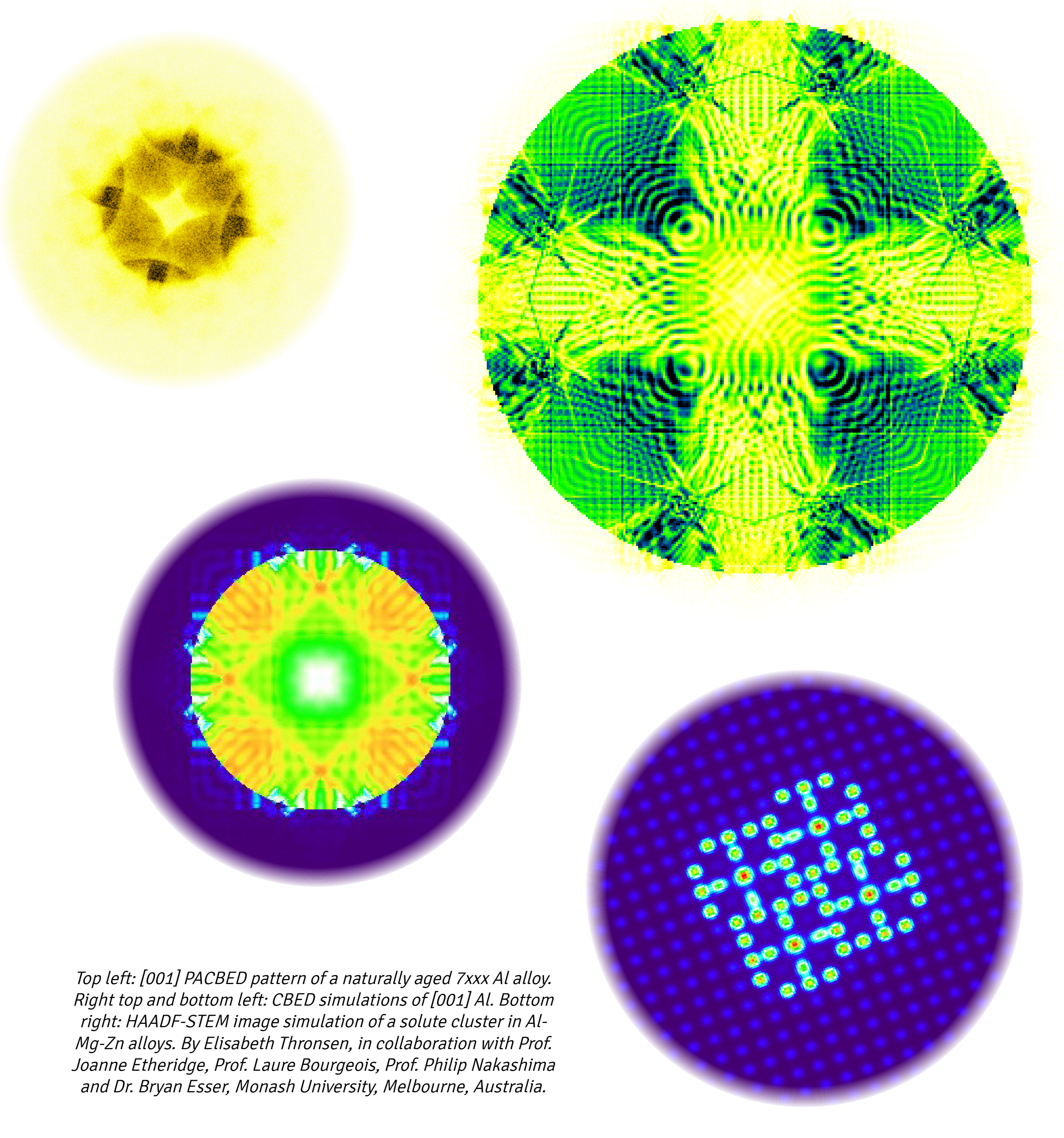




BF-TEM (left) and HAADF-STEM (right) image of precipitates at a grain boundary, that are related to intergranular corrosion, in a 6005 Al alloy. By Adrian Lervik.

ACTIVITIES

RESEARCH AND EVENTS



Top left: [001] PACBED pattern of a naturally aged 7xxx Al alloy. Right top and bottom left: CBED simulations of [001] Al. Bottom right: HAADF-STEM image simulation of a solute cluster in Al-Mg-Zn alloys. By Elisabeth Thronsen, in collaboration with Prof. Joanne Etheridge, Prof. Laure Bourgeois, Prof. Philip Nakashima and Dr. Bryan Esser, Monash University, Melbourne, Australia.

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 2019. The next sections describe these activities. Activities in aluminium alloy research are the largest. In all areas the use of advanced data processing has gained significance.

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 2019 we have been involved in 2 SFI Centers, 3 competence projects (KPN) and one Digitalization project in aluminium research, in addition to the INTPART project with Japanese universities and aluminium industry.

In SFI CASA, headed by Prof. Magnus Langseth at the Structural 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 finished his PhD thesis on TEM studies of deformed aluminium alloys in September, revealing connections between the materials microstructure and their mechanical behaviour. Emil is still hired in the CASA project, as a postdoc in the Structural Engineering department, doing TEM on CASA materials. Jonas Frafjord is hired to work on the modelling side on the lower scale. He is doing density functional theory (DFT) and molecular dynamics in combination with other higher scale methods to explore dislocation behaviour in Al alloys. Project leader of the SINTEF part of CASA Lower scale is Inga Ringdalen. SFI CASA has made a promotion video titled "Centre for Advanced Structural Analysis | NTNU" (youtube.com/watch?v=mQXCU9uNLUI) where TEM on aluminium has a central part.
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 joining techniques, including joints made by the start-up company HyBond. Tina uses conventional and advanced TEM techniques and also works on electron diffraction data analysis (pyXem).

Three 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 a 5 years project, ending in 2020, that has the objective of establishing new fundamental understanding of the mechanisms of intergranular corrosion (IGC). Industrial funding is provided by a consortium of four aluminium companies; Hydro Aluminium, Benteler, Gränges and Steertec, representing the entire aluminium value chain. The mechanisms of IGC are studied at the nm-scale utilizing advanced laboratory infrastructure, especially TEM, plus modelling. Adrian Lervik is working as a PhD student in the FICAL project and focuses on quantitative understanding of nanoscale structure and chemistry related to IGC. One concrete case studied is stress corrosion cracking in 7xxx alloys from Benteler Automotive.

The second competence project with aluminium industry is the project '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. Hydro Aluminium, Gränges, Nexans and Neuman are partners. The project aims at providing new advances in experimental technologies, experimental databases and developing modelling tools for combinations of aluminium properties. Jonas K. Sunde is a PhD student on this project and has been studying

People working with aluminium and TEM at NTNU/SINTEF in 2019.

the effects of very small Cu additions to the 6082 alloy by combining advanced TEM techniques, such as scanning precession electron diffraction (SPED) and HAADF-STEM.

The third competence project on Aluminium is SumAl (Solute cluster manipulation for optimized properties in Al-Mg-Si based Al alloys) with industry partners from Norway (Hydro, Benteler and Neuman), Austria (Neuman), Sweden (Hydro) and Germany (Hydro). The primary objective of SumAl is to establish an in-depth understanding of early stage solute ordering and atomic clustering by advanced experiments and modelling, and how these structures relate to the development of hardening precipitates and materials properties. Randi Holmestad is project leader, and there will be both TEM experiments and modelling within the TEM Gemini Centre in the years to come. A new PhD student, Christoph Hell is hired from January 2020.

NAPIC (NTNU aluminium product innovation Centre) was established in 2017, and Håkon Wiik Ånes is working as a PhD student in this centre, based in DMSE to study nucleation of recrystallization using SEM and TEM.

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.

An aluminium project that started in 2018 is the NTNU-financed Digitalization project AllDesign with Jaakko Akola as project leader. AllDesign provides 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 is a PhD student on this project and work on TEM of early stage clustering and precipitation. She stayed two months at Monash university, Melbourne Australia in 2019, to do diffraction experiments from clusters on a Titan microscope with an EMPAD detector.

Dr Marat Gazizov was hired as a postoc in the TEM Gemini Centre up to February 2019, supported by the Natural Sciences Faculty.

As seen from the publication list, we had in 2019 several invited talks about aluminium activities at international conferences, both material and microscopy conferences – AMT in Krakow, EMAG in Manchester, M&M in Portland, EUROMAT in Stockholm. In 2020 we will have also more invited talks (ACMM, ICAA, Thermec). In addition, we had popular science articles dedicated to our activities in Teknisk Ukeblad and Gemini.

The last three years we have started to study titanium alloys in the TEM, both providing feedback on processing parameters to industry and investigating fundamental precipitation phenomena at nm-scales. In 2019 Gregory Nordahl finished his MSc thesis on additively manufactured titanium grade 5 in collaboration with Norsk Titanium.

ENERGY MATERIALS – SOLAR CELLS

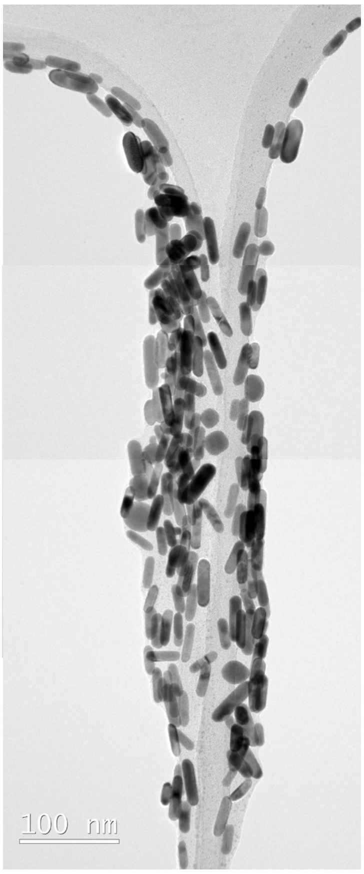
TEM has proven to be a crucial characterization tool to understand and improve the efficiency of both conventional and novel types of solar cells. The TEM Gemini Centre activities within solar cells include both types and a large range of materials. The Gemini Centre is participating in the FME SUSOLTECH (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 defended her PhD in June 2019. She worked 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. We also had a MSc student on this project in 2019.

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 finished her postdoc in the project at the end of Nov 2019. She combined 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.

SINTEF has worked together with ELKEM and IFE in three consecutive IPN projects within production of tailored Si powders for use in Li-ion batteries. The present project, "Silicon-based anodes towards market penetration (SiCANODE)" started in 2019. 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. Another IPN project, "Den Optimale Veien til Robuste Elektroder (DOVRE)" ended in 2019, but will continue through a new IPN project, "HAST" from 2020. The project owner CENATE, a spin-off company to Dynatec, 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. At the end of 2019 a new IPN project, "Surface treatment of Artificial Graphite for Anodes in Lithium-ion Batteries (SAGA)", was funded by RCN. The project owner, Elkem Carbon AS, aims to develop graphite materials for anodes in Li-ion batteries. TEM will here be a central tool to characterize the graphite powders, coatings and build-up of various solid electrolyte interphases as a function of production parameters and cycling conditions.



BF-TEM image of gold nanoparticles by Tina Bergh.

TEM is also used in a number of other projects related to development of Li-ion battery technologies. In the KMB project "Silicon on the Road", with SINTEF, IFE and UiO as academic partners and Cenate, Equinor, Borregaard and Beyonder as industry partners, TEM is used to characterize battery electrodes as a function of syntheses and cycling conditions. In the KPN project "SiBEC" TEM is one of the important characterization tools to understand the behaviour of the cathode material as a function of synthesis and cycling conditions. In the FME MoZEES TEM is also used to characterize and understand the fundamental behaviour of the battery electrodes as a function of electrode and electrolyte compositions, synthesis and cycling conditions.

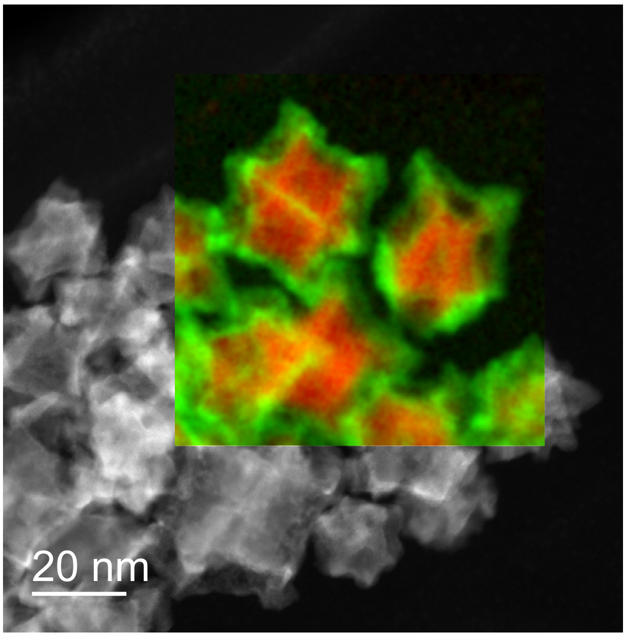
NANOTECHNOLOGY

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 analysed. 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 neighbour and many of the TEM operators also use equipment in the cleanroom. Especially the FIBs are important for the TEM Gemini Centre. We developed together with collaborators in Trondheim methods for correlated studies where TEM is directly combined with SEM, EBSD, Cathodeluminescence, Photoluminescence and Scanning Probe Microscopy. Hereby, more all-round characterization of nanomaterials is realized.

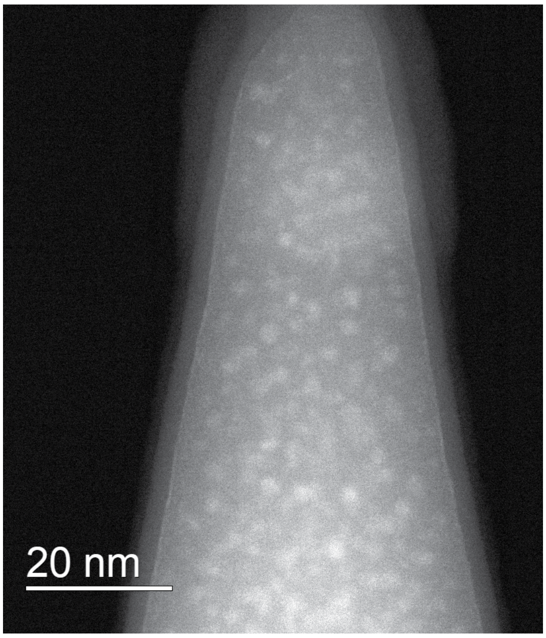
NorFab is an important partner for the TEM Gemini Centre. PhD Aleksander Mosberg is funded by NTNU's "Enabling technologies: Nanotechnology" and is based in the TEM Gemini Centre. Mosberg's PhD is focused on using FIB for nanostructuring. TEM is used to understand how the ion beam alters the materials. He uses TEM, as element in correlated studies, 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 had close ties with the Norwegian PhD Network on Nanotechnology for Microsystems and NTNU NANO. As can be seen from the publication list, many TEM studies on nanomaterials resulted in journal publications in 2019.

With the new atom probe tomography (APT) instrument installed at NTNU in 2018, the TEM group is participating in building up competence on APT and work on correlative techniques between TEM and APT.



Core-shell Ni-Ir nanoparticles for catalysis. The coloured inset shows a STEM-EELS map where red is Ni and green is Ir. By Sigurd Wenner.



Atom probe tomography needle of an Al-Zn-Mg alloy containing nanoclusters prepared by FIB. By Sigurd Wenner.

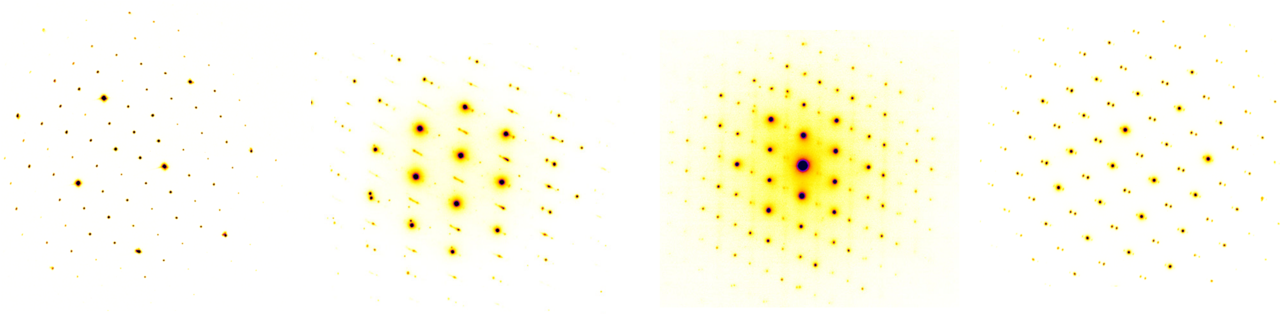
CHEMICAL ENGINEERING - CATALYSIS AND MEMBRANE MATERIALS

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. In the H2020 project eForFuel, Ir-based catalyst nanoparticles were investigated for their potential use in producing formic acid from carbon dioxide and water.

ADVANCED DATA PROCESSING

The ongoing revolution within TEM research is digital and data driven. Big/smart data, machine learning, open source, digital transformation, etc. are some of the current "hot topics". In 2019 we worked further on developing transparent procedures to handle larger TEM data sets and automation for more robust TEM studies. There were MSc, PhD and SINTEF projects dedicated on improving data handling. At the end of 2019 of the TEM Gemini Centre bought a share in the NTNU cluster IDUN for the coming 5 years. The common effort in establishing cluster-based TEM data handling will be continued. In 2019 a powerful local workstation became available for all TEM users In March 2020 Norway's first direct detection TEM detector will become available. We are confident we have the data infrastructure to make full use of the new possibilities this detector will give the facility users.

In the application of advanced TEM, especially multidimensional data set acquisition and handling, data processing transparency and dynamic in-situ studies are further pushed. 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 be handled in the same user interface. Also within SINTEF advanced data handling and incorporation of data TEM activities were further developed. Most of our MSc and PhD students are familiar with data handling in a Python environment. We have students who are dedicated towards modelling (e.g. DFT) or for whom scattering simulations are an essential part of their work.

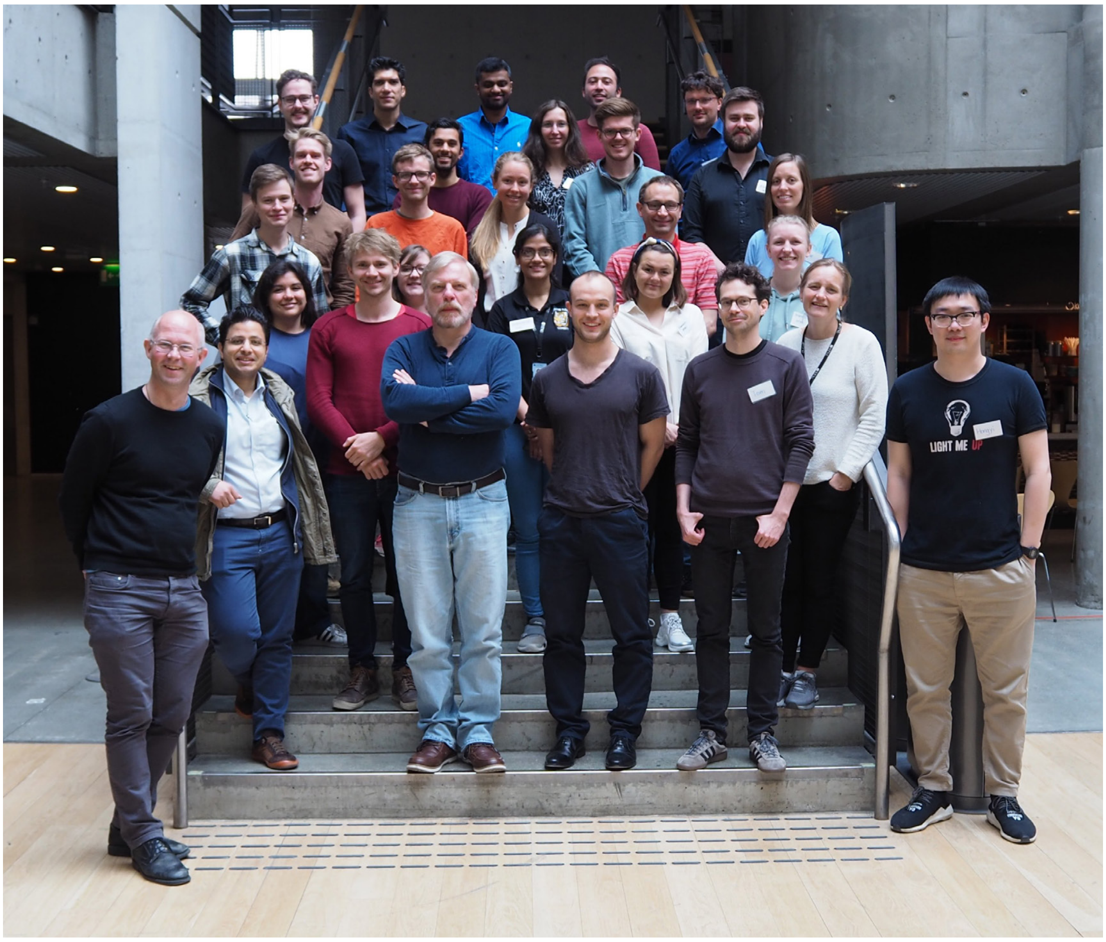


RESEARCHERS' NIGHT, SCHOOL VISITS AND OUTREACH

As in earlier years, the TEM Gemini Centre has contributed to a large number of high school visits through 2019. 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 not affiliated to TEM had a tour of our facilities.

WORKSHOP ON DIGITAL ELECTRON DIFFRACTION

15-16 May 2019 the TEM Gemini Centre organised a workshop on Digital electron diffraction with four external lecturers: Marc de Graef, Duncan Johnstone, Tom Wilhammar and Hongyi Xu. 24 participants, half from abroad (10 different labs) and half based in Trondheim, enjoyed a dedicated and diverse program addressing different aspects of electron diffraction and ongoing developments in electron diffraction data analysis. There were two full days with lectures on basic crystallography and electron scattering, modelling of electron diffraction processes in the SEM, structure determination by single-crystal electron diffraction, tomography and scanning electron diffraction microscopy in a (S)TEM, a lab demo on scanning precession electron diffraction and hands-on computer labs on packages as RED, EMsoft and pyXem. This was the third international workshop on advanced TEM the TEM Gemini Center organized in recent years.

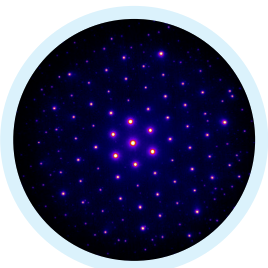
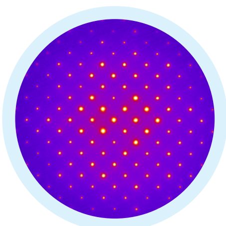
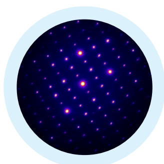
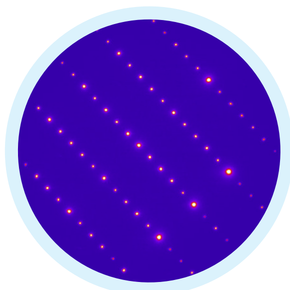
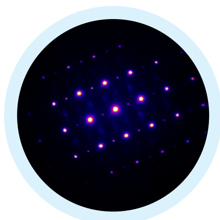


Participants at the 'Digital Diffraction' workshop in Trondheim, with invited lecturers in the front middle and front right.

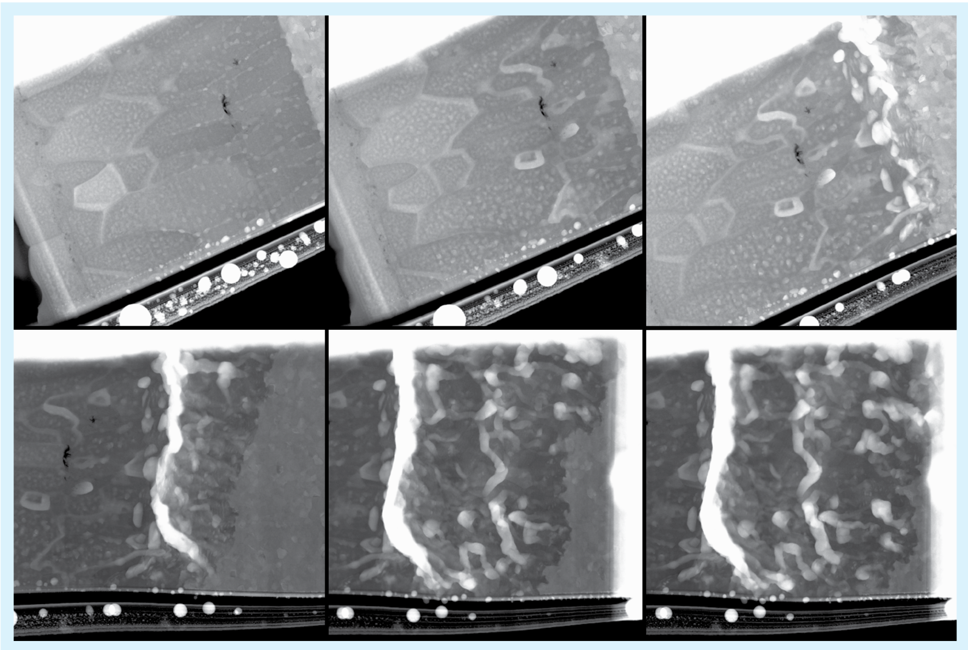
ACTIVE PROJECTS 2019

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. Smaller projects are not listed, both academic and with direct industrial support, run in parallel. In total the Centre had 110 different projects using the facilities in 2019.

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, 1 PD, 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, 1 PD, SINTEF, UiO	2009-2025
	Partners: IFE, NTNU, SINTEF, University of Oslo (UiO), CleanSi, Dynatec, Elkem Solar, Mosaic, Norsun, Norwegian Crystals, Quartz Corp, REC Silicon, REC Solar, Semilab		
FME	**Mobility Zero Emission Energy Systems - MoZEES**	SINTEF	2015-2023
	Partners: 7 research institutions including both SINTEF and NTNU, 7 public bodies, 26 industrial partners		
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	2012–2019
	Partners: NTNU, SINTEF		

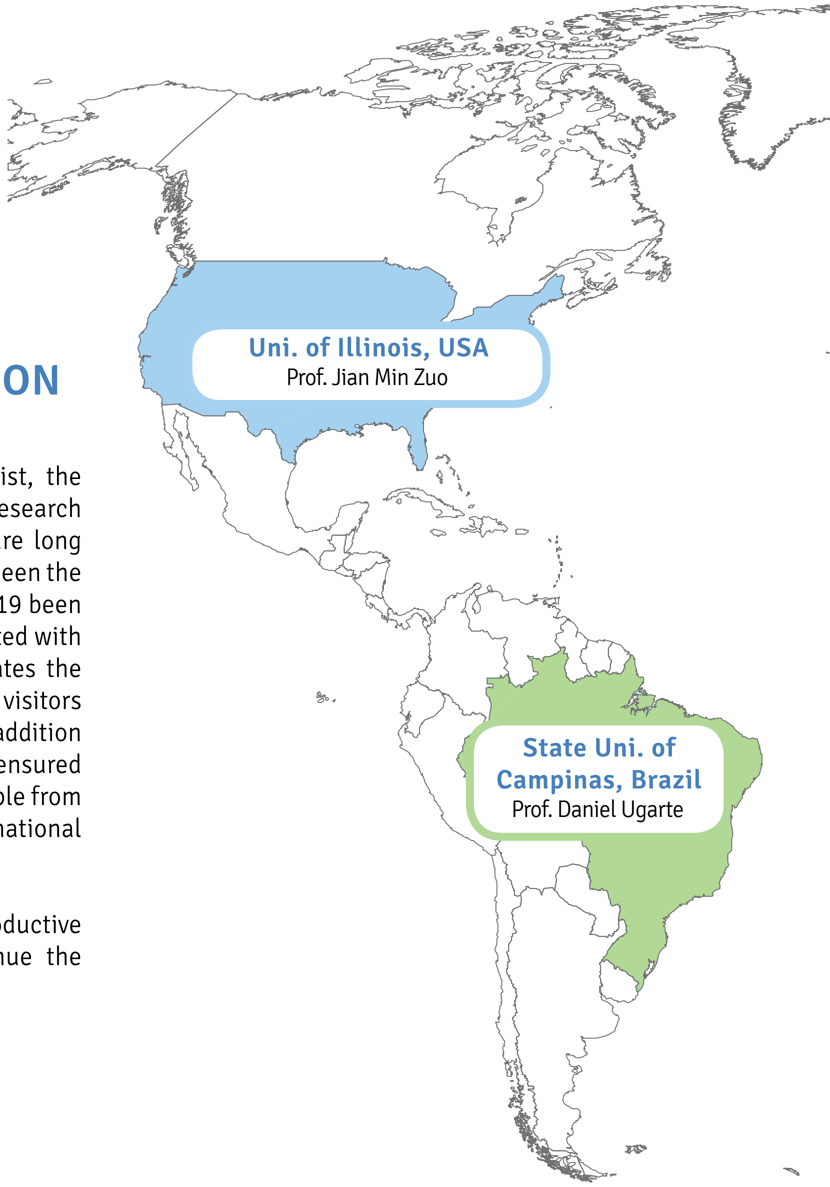






IPN/BIA	**Integrated Hardening and Sheet Press-forming of Aluminium (I-Pal)**	SINTEF	2016-2019
Partners: SINTEF, Hydro, SAPA, Raufoss Neuman, AP&T			
IPN/Nano2021	**Nanowire UV LEDs on graphene**	SINTEF	2019-2021
Partners: CrayoNano, SINTEF, NTNU			
IPN/ENERGIX	**Den Optimale Veien til Robuste Elektroder – DOVRE**	SINTEF	2018-2019
Partners; Cenate, Dynatec, IFE, SINTEF			
IPN/ENERGIX	**Silicon-based anodes towards market penetration – SiBanode**	SINTEF	2019-2021
Partners: Elkem Technology AS, IFE, SINTEF			
KPN/BIA	**Aluminium alloys with mechanical properties and electrical conductivity at elevated temperatures (AMPERE)**	1-2 PhDs, SINTEF	2015-2020
Partners: NTNU, SINTEF, Hydro, Nexans, Raufoss Neuman, SAPA, Gränges			
KPN/BIA	**Fundamentals of Intergranular Corrosion in Aluminum Alloys (FICAL)**	1 PhD, SINTEF	2015-2020
Partners: NTNU, SINTEF, Hydro, Benteler, Steertec, Gränges			
KPN/BIA	**SumAl (Solute cluster manipulation for optimized properties in Al-Mg-Si based Al alloys)**	1 PhD, SINTEF	2019-2024
Partners: NTNU, SINTEF, Hydro, Benteler, Neuman			
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) - II**	Travel, exchange students	2019-2023
Partners: NTNU, SINTEF, Hydro, University of Toyama, Tokyo Institute of Technology			
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 PD, SINTEF	2016-2019
Partners: NTNU, SINTEF			



ADF-STEM images aqcuired during in-situ heating of Al-Ni intermetallic phases by T. Bergh.

INTERNATIONAL COLLABORATION



As can be seen from the map and the publication list, the TEM Gemini Centre has productive relations to many research institutions and researchers across the world. Some are long term collaborators; others are new initiatives. 2019 has been the first year for ESTEEM3. Within ESTEEM3 we have in 2019 been granted seven transnational (TA) projects and collaborated with renowned researchers across Europe. The map illustrates the direct scientific collaborations. We have yearly several visitors from abroad that use the TEM facility in Trondheim. In addition to the ESTEEM3 project, the INTPART project has ensured international collaboration, in this case with Japan. People from the group were chairs and invited contributors to international conferences and acted as PhD examiners abroad.

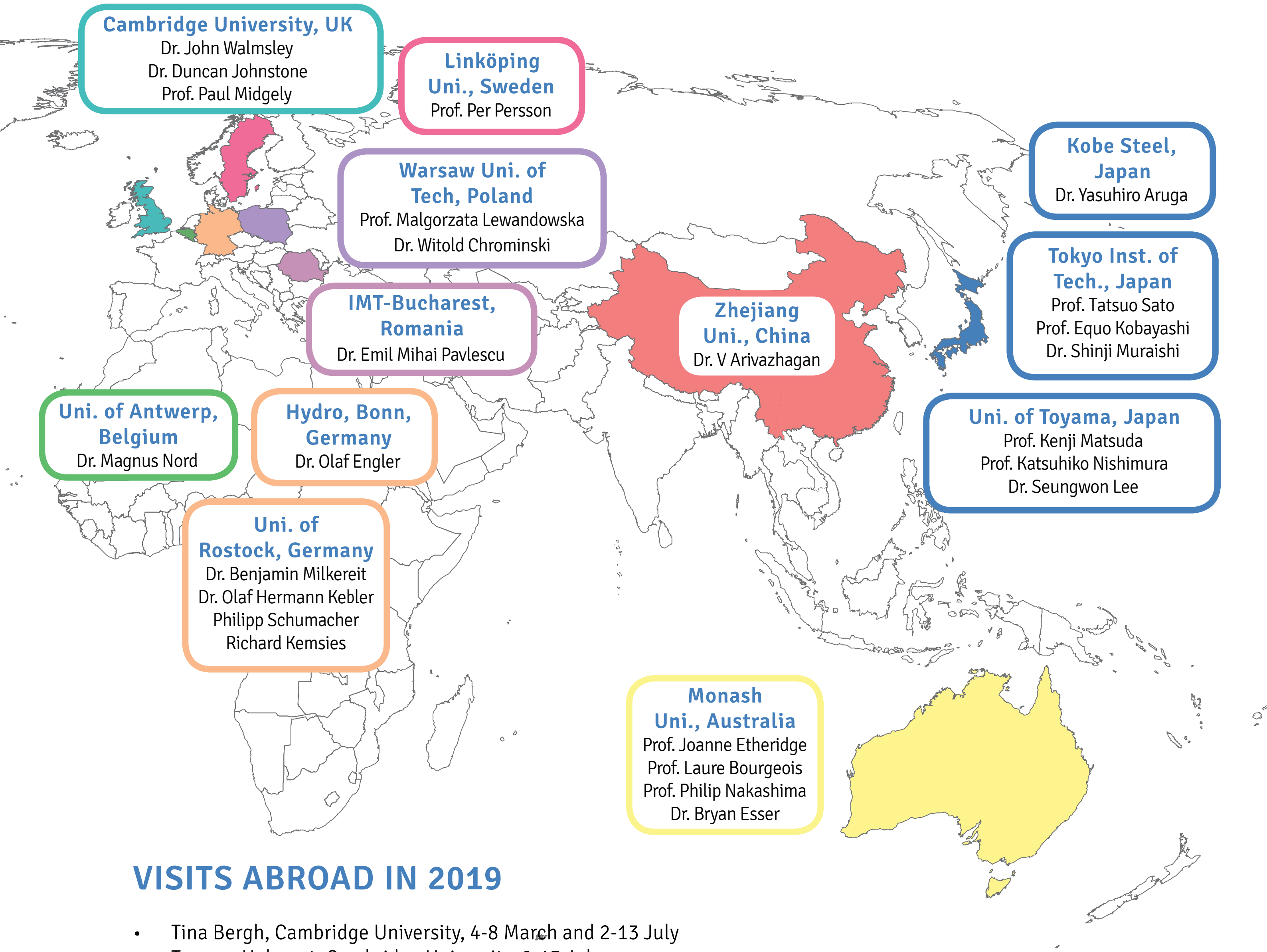
We thank all our international collaborators for the productive and stimulating interaction and hope we can continue the cooperation in the coming years!

NORDIC HUB

In 2019 the Gemini Centre took the initiative to a proposal to NordForsk for a Nordic University Hub within TEM. Per Persson from Linköping lead the final proposal. Seven partners from Norway (NTNU and UiO), Sweden (Linköping, Stockholm, Chalmers), Denmark (DTU) and Finland (Aalto) are in. If the proposal will be funded will be announced in the spring of 2020.

THE EU NETWORK PROJECT ESTEEM3

The TEM Gemini Centre is a partner in the 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 officially January 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 during 2019 we have already been granted 7 such projects. These projects were initiated from Germany, Romania, UK, Spain. TA exchanges do not only include data acquisition on the TEM, but also data handling. The website of ESTEEM3 (esteem3.eu) gives more details on how to get access through TA. Dr. Dipanwita Chatterjee is hired as a postdoc in this project, working on incoming TA activities (together with other TEM Gemini members) and the joint research activity Materials for Transport.



VISITS ABROAD IN 2019

- Tina Bergh, Cambridge University, 4-8 March and 2-13 July
- Ton van Helvoort, Cambridge University, 2-13 July
- Hanne Mørkeseth, YKK Kurobe, Japan, 14 Aug. - 14 Sept.
- Sander S. Jenssen, YKK Kurobe, Japan, 14 Aug. - 14 Sept.
- Randi Holmestad, Monash University, Australia, 8 Oct. – 6 Dec.
- Elisabeth Thronsen, Monash University, Australia, 8 Oct. - 30 Nov.

INTERNATIONAL VISITORS IN 2019

- Kazuhiko Kita (YKK, Kurobe, Japan) 13-16 March
- Tetsuya Katsumi (YKK, Kurobe, Japan) 13-16 March
- Kenji Matsuda (Toyama University, Japan) 14 – 17 March
- Marc de Graef (Carnegie Mellon University) 12-20 May
- Tom Wilhammar (Stockholm University) 13-18 May
- Hongyi Xu (Stockholm University) 13-18 May
- Duncan Johnstone (University of Cambridge) 13-18 May
- Jonas Werner (RWTH-aachen, Germany) 24-30 May
- Ana Sanchez (University of Warwick, UK) 5-8 June
- Eiji Abe (Tokyo University, Japan) 23. Sept.
- Kenji Kaneko (Kyushu University, Japan) 25-29 Sept.
- Daehan Kim (Tokyo Institute of Technology, Japan) 13 Oct. – 31 Dec.
- Xuanling Chen (Tokyo Institute of Technology, Japan) 13. Oct. – 14 Dec.
- Yamato Takeuchi (Tokyo Institute of Technology, Japan) 13 Oct. – 14 Dec.
- Jan Hajer (Würzburg University, Germany) 10 – 16 Nov.
- Mario Heinig (Danish Technical University (DTU), Denmark) 1 – 13 Dec.
- Simon Hogg (Loughborough University, UK) 14. -20. Dec.

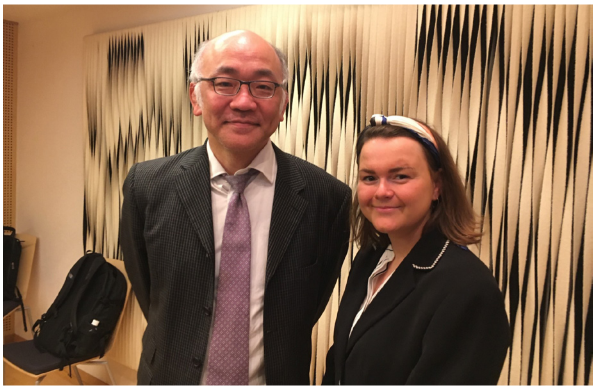
In addition, the facility was used, without collaborators present, for samples from Poland, Romania and Germany.

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 2019. In addition to NTNU and SINTEF, Hydro Aluminium, University of Toyama and Tokyo Institute of Technology were partners. A phase II of this was granted in 2019, with the same partners, except one additional university, Kyushu University in Fukuoka. The objective of this project has been to continue the fruitful partnership we obtained through earlier projects, and also include and formalize educational issues, such as guest lecturers, workshops joint courses and internships. Furthermore, exchange of MSc and PhD students on internships in Japanese and Norwegian aluminium industry and universities have been a prioritised activity.

During 2019 there has been a large activity in the INTPART project. We have had three visitors from Tokyo Tech to NTNU, and we had two Norwegian students on internships in Japanese aluminium industry and two visits to Japan from the aluminium group in the TEM Gemini Centre. In March, we had a visit to Trondheim from YKK, in May a delegation of 5 from the TEM Gemini Centre visited Tokyo, Toyama and Kyushu universities. In October, 3 researchers from Trondheim visited Toyama to give lectures, and participated in two conferences.

From the banquet of the CAMRIC5 conference in Toyama, October 2019. Dr. Calin Marioara (SINTEF), Prof. Knut Marthinsen (NTNU), Dr. Takeshi Sato (Hydro) and Prof. Randi Holmestad (NTNU) can be seen among the participants.



From the signing of the new INTPART-II project, May 2019, Prof. Kenji Kaneko (Kyushu University) and PhD student Elisabeth Thronsen (NTNU).

From the signing of the new INTPART-II project, outside the Norwegian Embassy, May 2019. From left; Prof. Katsuhiko Nishimura (Uni. of Toyama), Prof. Randi Holmestad (NTNU), Dr. Sigmund Andersen (SINTEF), Prof. Knut Marthinsen (NTNU), Dr. Takeshi Saito (Hydro) and Prof. Erik Wahlström (NTNU).

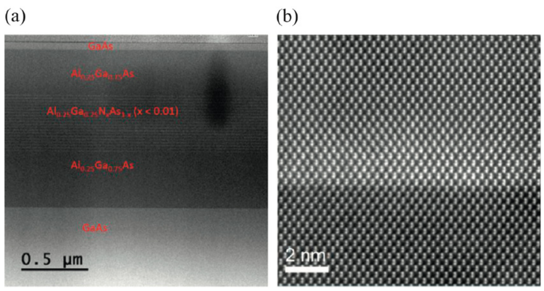
From left Prof. Knut Marthinsen (NTNU), Prof. Kenji Kaneko (Kyushu University) and Peter Karlsaune (NTNU) visiting the lab facilities in CASA, Structural engineering, NTNU, September 2019.

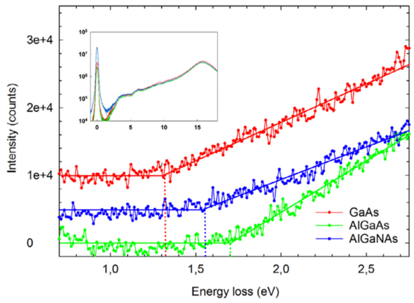
The three visiting students from Tokyo Institute of Technology at Bybroen, Trondheim, September 2019. From Left; Dr. Sigurd Wenner (SINTEF), Postdoc Emil Christiansen (NTNU), PhD student Jonas K. Sunde (NTNU), Xuanling Chen (Tokyo Tech), Daehan Kim (Tokyo Tech), Yamato Takeuchi (Tokyo Tech) and Postdoc Dipanwita Chatterjee (NTNU).

PHD DEFENSES
IN THE TEM GEMINI CENTRE 2019

MARYAM VATANPARAST - JUNE 7th

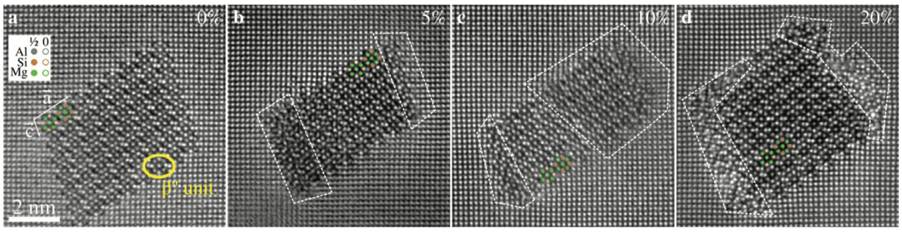
Maryam Vatanparast's PhD work was dedicated to GaAs based materials for intermediate band solar cells (IBSCs) - studied by TEM and electron energy loss spectroscopy (EELS). TEM allowed to quantitatively study the quantum dot compositions, dislocations in different thin layers and strain down to sub-Ångström resolution, and even dielectric properties, including the bandgaps with nanometer resolution. Determination of bandgaps in high refractive index materials as GaAs can be quite difficult to study by high energy electrons. EELS with high spatial resolution over optical methods motivated us to develop a method to control the retardation losses and measure the bandgaps. The thesis shows that TEM is a strong tool to study GaAs based materials for IBSCs and that advanced TEM can give valuable feedback and information on the nanometer scale, both for those who grow the materials, and also on more fundamental materials properties such as bandgaps.





Left figure: (a) HAADF- STEM image of GaAs with a $Al_{0.25}Ga_{0.75}As$ layer and a stack of 20 InAs quantum dot (QD) layers separated by $Al_{0.25}Ga_{0.75}N_xAs_{1-x}$ spacer layers. (b) HAADF-STEM image of a QD. Right figure: Background subtracted electron energy loss spectra from GaAs, AlGaAs and AlGaNAs acquired in Low-Mag off-axis set-up, showing their bandgaps.

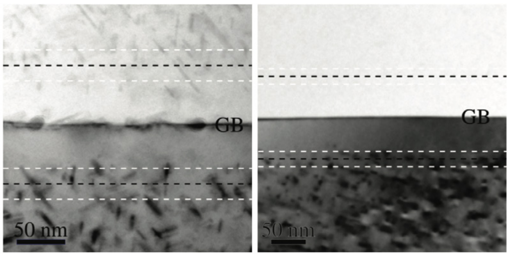
Left image: Maryam with supervisors. From left Randi Holmestad, NTNU; Per Erik Vullum, NTNU/SINTEF and Bjørn Ove Fimland, NTNU. Turid D. Reenaas, NTNU was not present. Right image: From left Steinar Raaen, NTNU (administrator); Ana Sanchez, University of Warwick (opponent); Maryam Vatanparast (PhD!) and Annett Thøgersen, SINTEF (opponent).

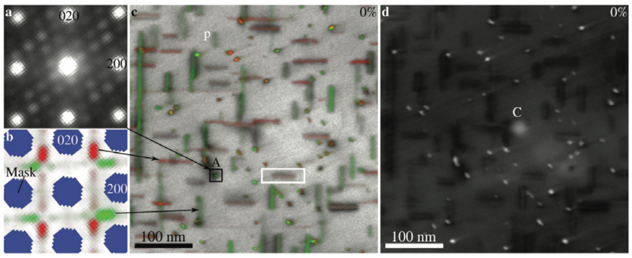


Smart-aligned and filtered [001]Al zone axis HAADF STEM images of precipitates in (a) undeformed, and after (b) 5%, (c) 10%, and (d) 20% compressive engineering strain.

EMIL CHRISTIANSEN - SEPTEMBER 27th

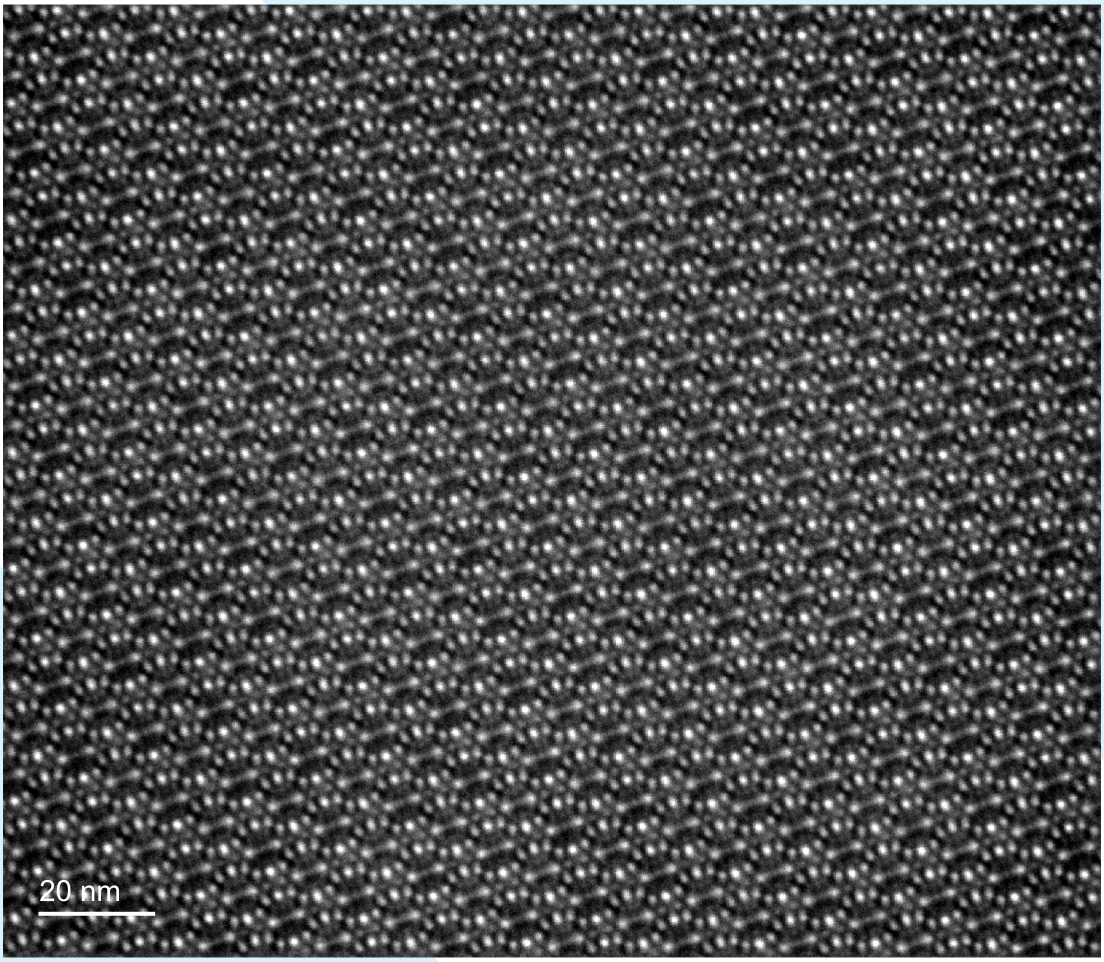
In his PhD work, Emil Christiansen used TEM to study deformed Al-Mg-Si alloys. In the first study, he found that that very small subgrains in the order of 200 nm in diameter can form when strain localises in precipitate free zones (PFZs) in materials artificially aged to the peak hardness condition. These subgrains will likely strengthen the PFZ by the Hall-Petch mechanism and might reduce the amount of strain localisation. In another study, he looked at how the quench rate during thermo-mechanical processing influences precipitation and mechanical properties of three different Al-Mg-Si alloys with different texture and grain structure. This study showed that dense alloys can be very quench sensitive if they have a very small grain size, as the PFZs around grain boundaries and dispersoids overlap and create a very weak alloy. Finally, he studied how the needle-like " precipitates that form in Al-Mg-Si alloys in peak hardness are sheared by dislocations.

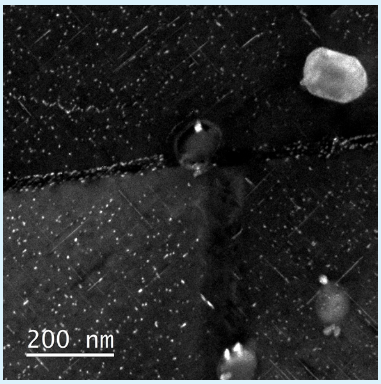


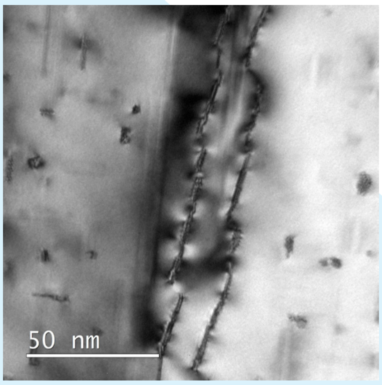


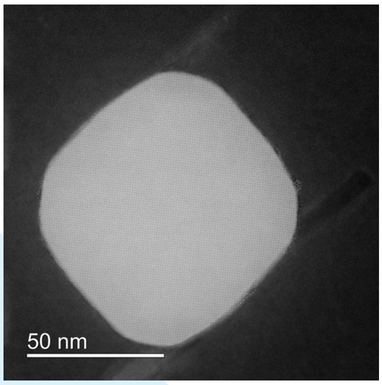
Left figure: TEM BF images of grain boundaries and surrounding PFZs. Right figure: [001]Al zone axis SPED results. (a) Single-pixel PED pattern of a precipitate. (b) NMF factors from in-plane precipitates. (c) VBF image with overlaid normalised NMF loadings. (d) VADF image.

Left image: From left: Randi Holmestad, NTNU (supervisor); Kenji Kaneko, Kyushu University (opponent); Emil Christiansen (PhD!); Aude Simar, UCLouvain (opponent); Geir Ringen, NTNU (administrator) and Odd Sture Hopperstad, NTNU (supervisor). Calin Marioara, SINTEF (supervisor) was not present. Right image: Emil Christiansen presenting.





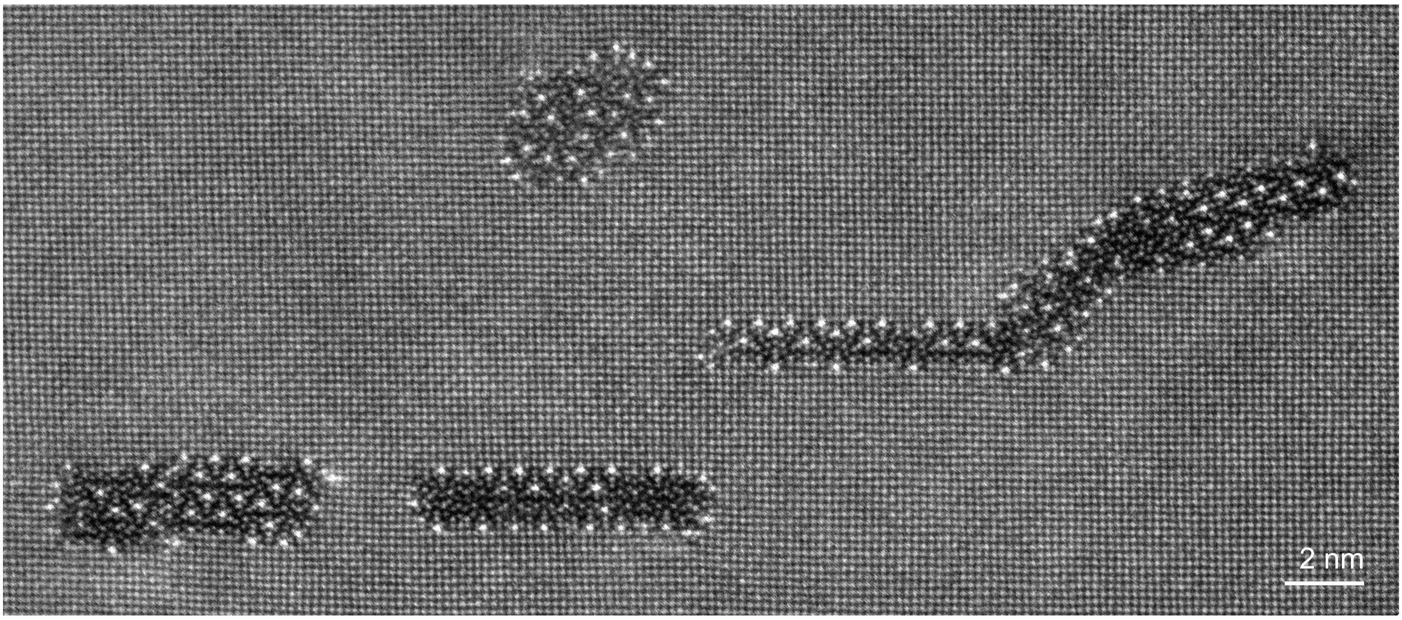


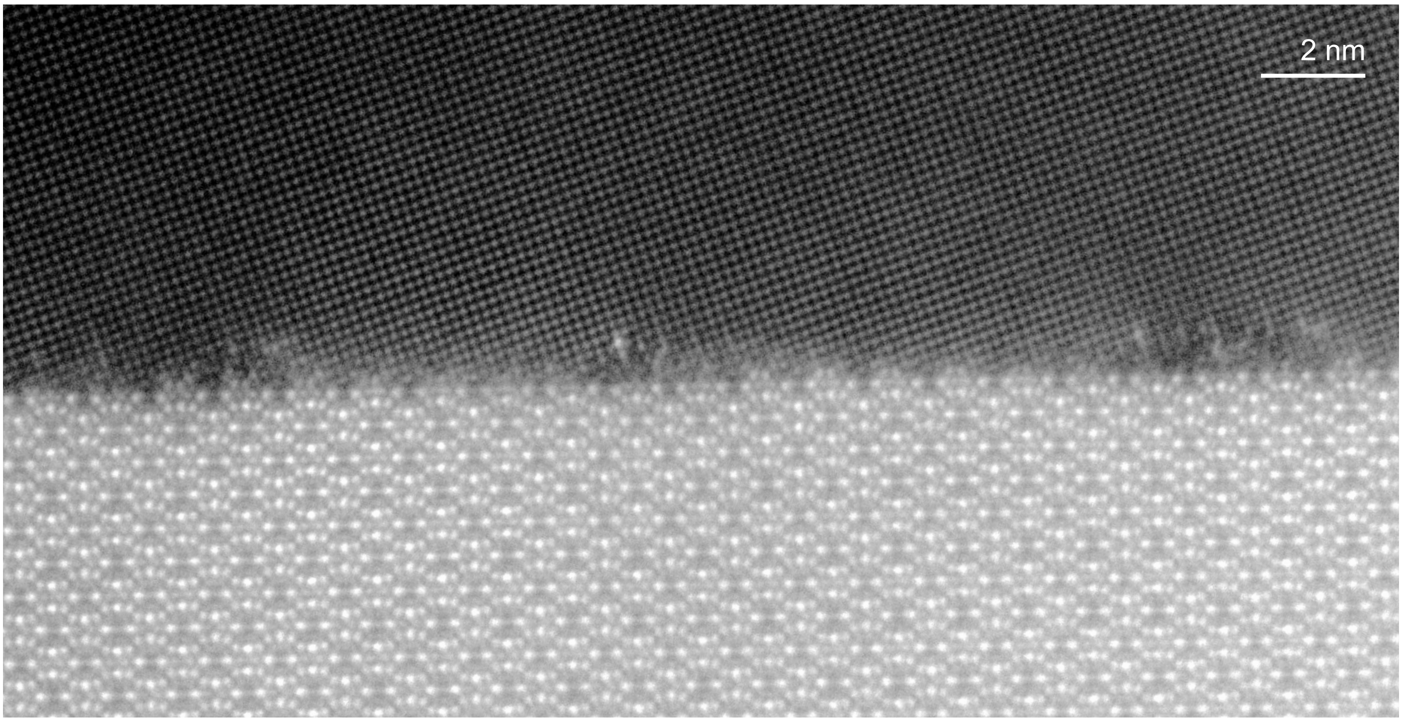


Top and bottom right: HAADF-STEM images of an alpha phase in an Al-Mg-Si-Cu alloy. Bottom left: DF-TEM of a low angle grain boundary. Bottom middle: BF-TEM image of precipitates on dislocations. By Adrian Lervik.

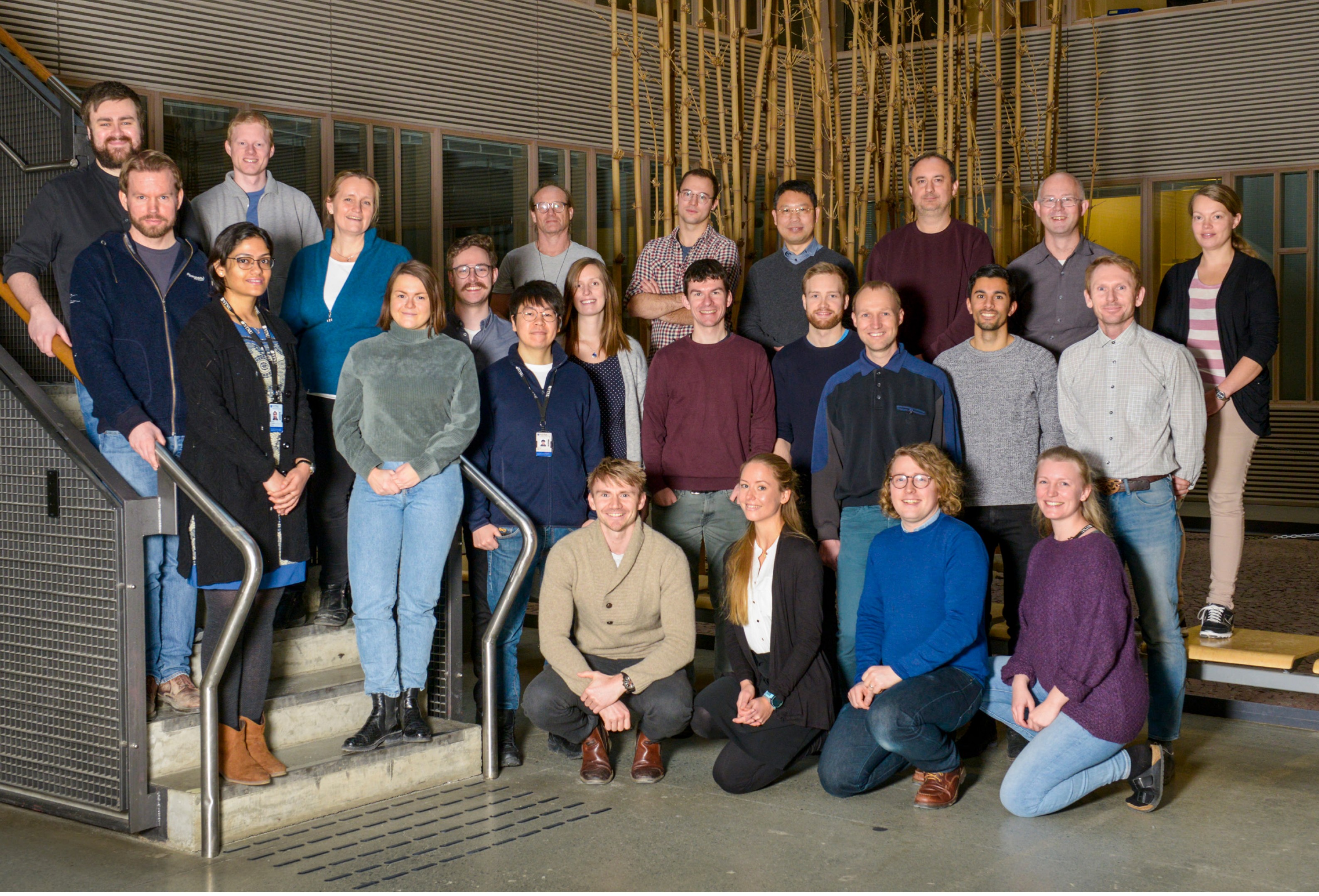
PUBLICATIONS 2019

PEOPLE IN THE TEM GEMINI CENTRE ARE HIGHLIGHTED





HAADF-STEM images of (top) precipitates formed on dislocations in an Al-Mg-Si-Cu alloy, and (bottom) the interface between Al and an alpha dispersoid. By Adrian Lervik.



PEOPLE IN THE TEM GEMINI CENTRE IN 2019

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 (Senior 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 and Assoc. Prof. II, DP, NTNU)
Calin Marioara (Senior research scientist, SINTEF)
Ruben Bjørge (Research scientist, SINTEF)
Sigurd Wenner (Research scientist, SINTEF and Assoc. Prof. II, DP, NTNU)
Marat Gazizov (Postdoc, DP, NTNU)
Maria Tsoutsouva (Postdoc, DP, NTNU)
Dipanwita Chatterjee (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)
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Elisabeth Thronsen (PhD student, DP, NTNU)
Kasper Aas Hunnestad (PhD student, DMSE and Master student, DP, NTNU)
Jørgen Sørhaug (Master student, DP, NTNU)
Gregory Nordahl (Master student, DP, NTNU)
Edwin Nongba Traore (Master student, DP, NTNU)
Haakon Tvedt (Summer student, DP, NTNU)
Daniel Martin Lundeby (Master student, DP, NTNU)
Ingvild Hansen (Master student, DP, NTNU)
Simon Høgås (Master student, DP, NTNU)
Yngve Maximilian Ender (Master student, DP, NTNU)
Tor Inge Thorsen (Master student, DP, NTNU)
Hanne Mørkeseth (Project student, DP, NTNU)
Sander S. Jenssen (Project student, DP, NTNU)
Hursanay Turgun (Project student, DP, NTNU)
Ragna Bakke (Project student, DP, NTNU)
Sigrid Wanvik Haugen (Project student, DP, NTNU)
Endre Jacobsen (Project student, DP, NTNU)
Eirik Opheim (Project student, DP, NTNU)
Øystein Rolstad (Project student, DP, NTNU)

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H. F. Andersen, C. E. L. Foss, J. Voje, R. Tronstad, T. Mokkelbost, P. E. **Vullum**, A. Ulvestad, M. Kirkengen, and J. P. Mæhlen, “Silicon-Carbon composite anodes from industrial battery grade silicon”, *Scientific Reports* **9**, 14814 (2019).

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J. K. **Sunde**, E. **Christiansen**, E. **Thronsen**, S. **Wenner**, C. D. **Marioara**, A. T. J. **van Helvoort**, and R. **Holmestad**, “Scanning Precession Electron Diffraction to aid Aluminum Alloy Development”, *Microscopy and Microanalysis,* **25**, 1920-1921 (2019).

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The annual hike in Bymarka with the TEM group organized by Ton van Helvoort. Photos by Randi Holmestad.

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T. **Bergh**, D. N. Johnstone, P. Crout, S. **Høgås**, P. A. Midgley, R. **Holmestad**, P. E. **Vullum**, and A. T. J. **van Helvoort**, “Nanocrystal segmentation in scanning precession electron diffraction data”, *Journal of Microscopy,* in press (2019). doi.org/10.1111/jmi.12850

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POPULAR SCIENCE

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Sikrere biler og bygg starter på nano-nivå, *Gemini*, 14-10-2019

PhD students Jonas K. Sunde, Tina Bergh and Emil Christiansen, Prof. Randi Holmestad and collaborator Dr. Witold Chrominski decided to go for an ‘assassinations’ photo session theme at the banquet for the EMAG MMC conference in Manchester, UK.

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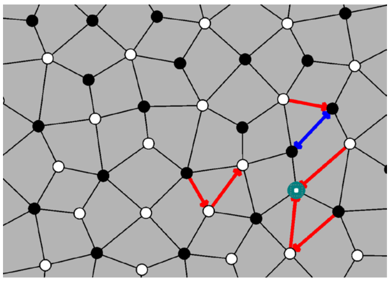
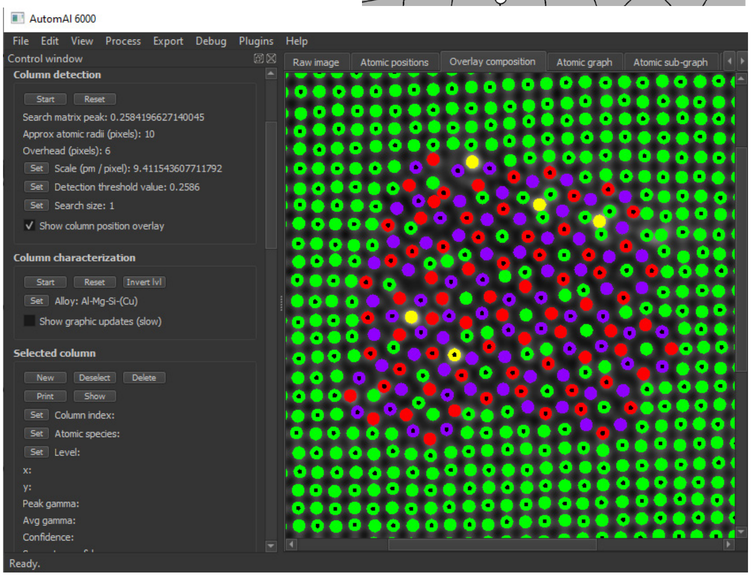
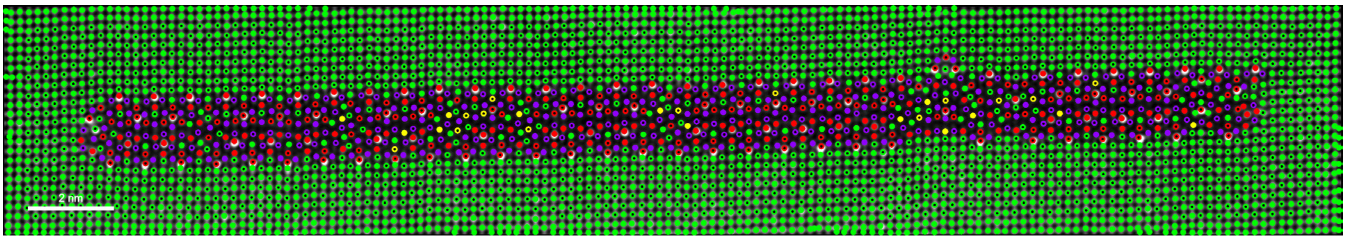
Yngve Maxmillian Ender, "Characterization of particles from dissolved 5049 aluminum alloys, and the microstructure from billet to extruded and drawn tubes", August 2019.
Ingvild Hansen, "Correlated Structural and Optical Characterisation of Individual AlGaN Nanowires on Graphene", June 2019.
Kasper Aas Hunnestad, "Visualizing Ferroelectric Domain Structures in $ErMnO_3$ and $Pb_5Ge_3O_{11}$ by Electron Microscopy", June 2019.
Simon Høgås, "Improvements to nm scale crystal phase and orientation mapping by physical model comparison", June 2019.
Daniel Martin Lundeby, "Improving the accuracy of TEM-EDX quantification by implementing the zeta-factor method", June 2019.
Gregory Nordahl, "Transmission electron microscopy characterization of heat treatment effects in additively manufactured Ti-6Al-4V", December 2019.
Jørgen Sørhaug, "TEM characterization of tungsten implanted silicon - A study of a potential intermediate band solar cell material", June 2019.
Edwin Traore, "Characterization of BTO thin films by electron diffraction techniques", June 2019.



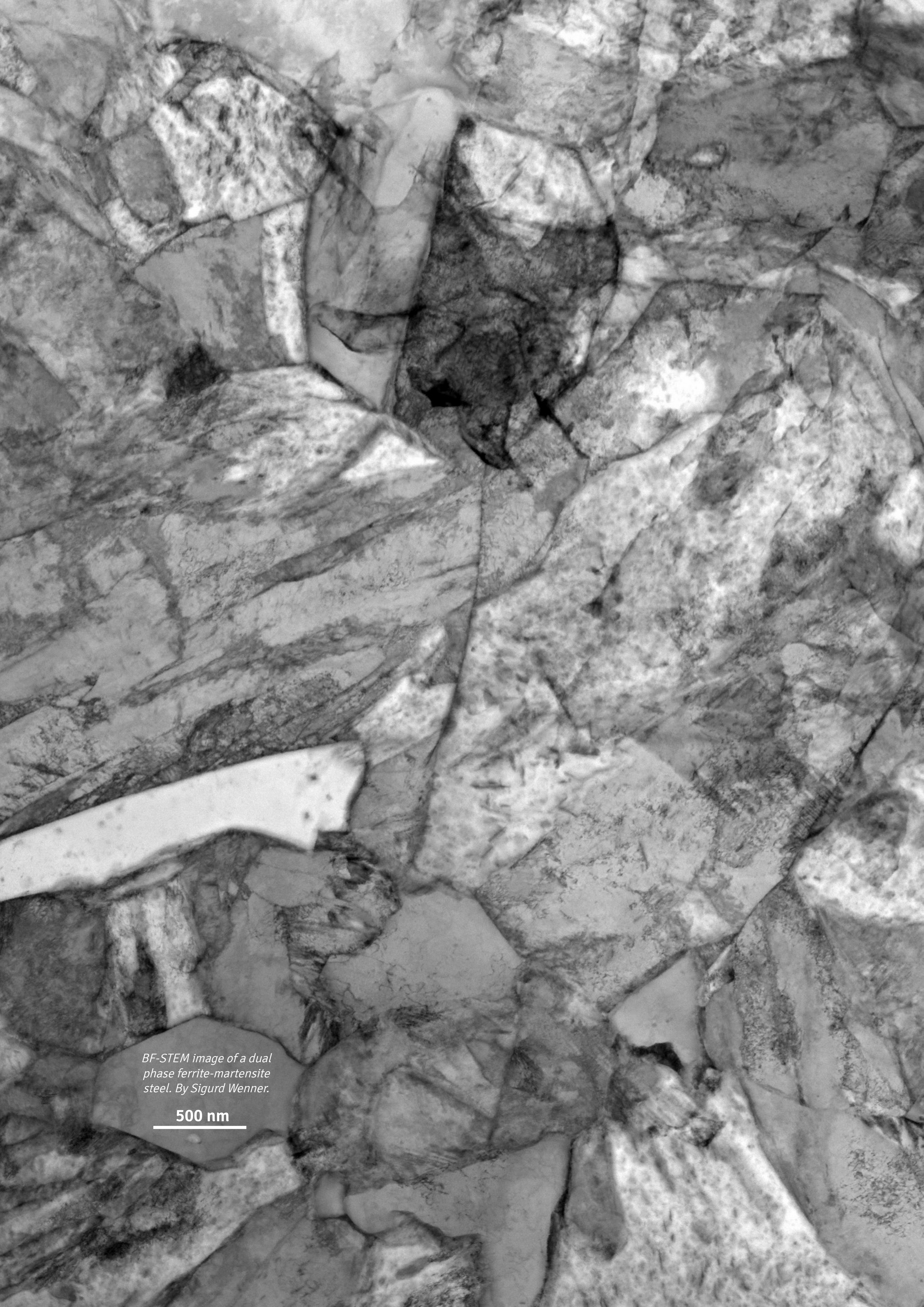
PROJECT THESES

Sigrid Wanvik Haugen, "Correlated SEM/TEM/CL of GaN/AlGaN nanowire based LEDs", December 2019.
Endre Jacobsen, "Scanning Precession Electron Diffraction Template Matching for Automated Phase Mapping of Precipitates in Aluminium Alloys", December 2019.
Hanne Mørkeseth, "The effect of Fe impurities on mechanical properties and microstructure in an Al-Cu-Mg-Si alloy" December 2019.
Eirik Opheim, "Evaluating template matching for simulated kinematic electron diffraction", December 2019.
Øystein Rolstad, "Determining elemental occupancy through EDX channeling analysis" (Project), Submitted December 2019.
Hursanay Turgun, "Electron microscopy characterization of hybrid metal extrusion and bonding joint", December 2019.



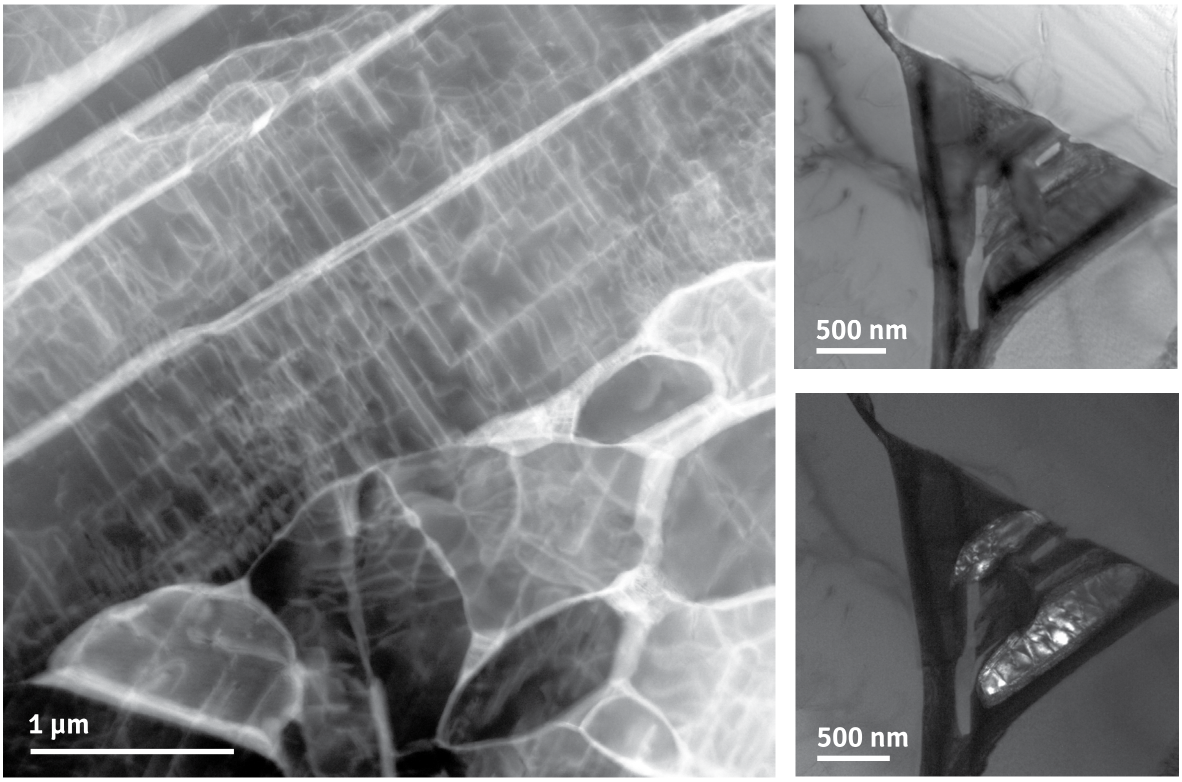


The software project AutomAl 6000 on statistical analysis and automatic atomic overlay of HAADF-STEM lattice images of precipitates in 6xxx Al alloys. Top: an 'atomic graph' that visualizes the underlying data structure of the software. Middle: the graphical user interface of the software. Bottom: a HAADF-STEM image of a large C-plate precipitate in an Al matrix overlayed by the software. By Haakon Tvedt.



BF-STEM image of a dual phase ferrite-martensite steel. By Sigurd Wenner.

SELECTED SCIENTIFIC PAPERS



Left: HAADF-STEM image of a lamellar Ti-6Al-4V microstructure, showing high dislocation density in alpha plates. Right: BF-TEM image (top) and DF-TEM image (bottom) of a triangular titanium beta phase grain containing several lamellae of an intermetallic phase. The DF-TEM image highlights of one of the observed orientations of the intermetallic phase. By Gregory Nordahl.

SCIENTIFIC REPORTS
natureresearch

Nano-scale characterisation of sheared β″ precipitates in a deformed Al-Mg-Si alloy

Emil Christiansen[1,2*], Calin Daniel Marioara[1,3], Bjørn Holmedal[1,4], Odd Sture Hopperstad[1,5] & Randi Holmestad[1,2]

This paper compares the nano-scale structure of β″ precipitates in a peak-aged Al-Mg-Si alloy before and after deformation. Three complementary advanced transmission electron microscopy techniques are used to reveal the structures and elucidate the interaction between dislocations and β″ precipitates. We show that the needle-like and semi-coherent β″ precipitates are sheared several times on different planes by dislocations during deformation, with no indications that they are bypassed or looped. Our results show that dislocations cut through precipitates and leave behind planar defects lying on planes inclined to ⟨100⟩ directions inside the precipitates. The results also indicate that precipitates are sheared in single steps, and the implication of this observation is discussed in terms of slip behaviour.

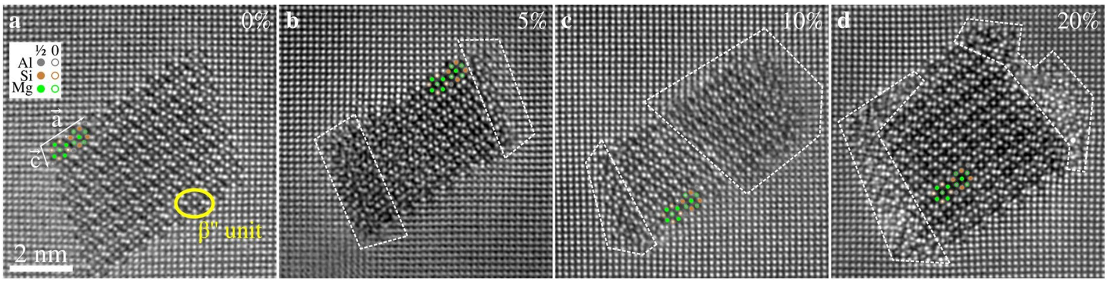


Figure 3. Smart-aligned and filtered $[001]_{Al}$ zone axis HAADF STEM images of precipitates in (**a**) undeformed, and after (**b**) 5%, (**c**) 10%, and (**d**) 20% compressive engineering strain. An overlay of the β″ precipitate structure is also included, based on Wenner *et al.*[38]. The relative position of the overlaid atoms in the out-of-plane direction is indicated by full and empty circles, referring to $1/2 \cdot b_{\beta''}$ and $0 \cdot b_{\beta''}$, respectively. A β″ unit referred to as a β″ "eye" is circled in yellow in (**a**). Precipitates in deformed specimens typically have regions (dashed) with reduced contrast/resolution along some of the edges.

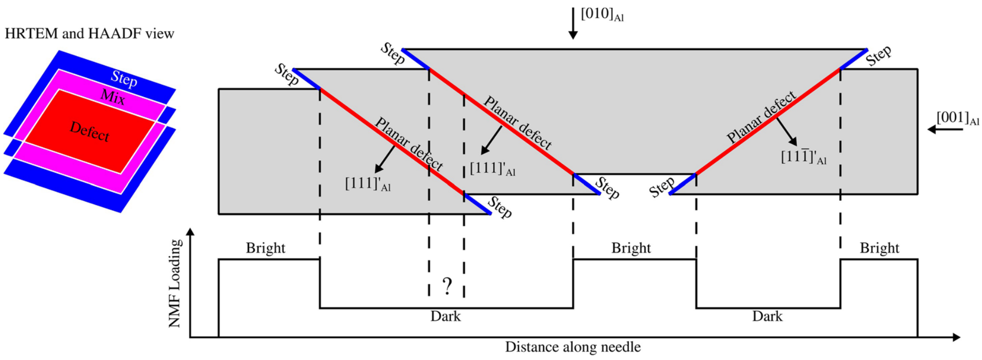
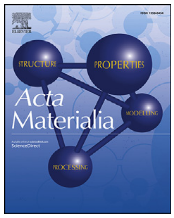


Figure 6. Schematic illustration of a $[100]_{Al}$ section of a sheared β″ precipitate and how deformation induced defects will appear in HRTEM, HAADF STEM and SPED. The precipitates are sheared on $\{111\}_{Al}$ planes (marked by the projection of their $\langle 111\rangle'_{Al}$ plane normals), and the shearing planes become planar defects because these are not slip planes in the β″ phase. Each shearing event also creates steps on the precipitate-matrix interface. Traces of the planar defects and steps are shown in red and blue, respectively. The cross-sectional view to the left is a projection of the needle along $[001]_{Al}$, and shows the defects and steps in projection, with overlap between planar defects and steps shown as magenta regions. The SPED NMF loading intensity variation of in-plane needles is shown schematically as well and is explained as the projection of the planar defects along $[010]_{Al}$. Whether the projection of the steps influence scattering is not known.

Crystallographic relationships of T-/S-phase aggregates in an Al–Cu–Mg–Ag alloy

Jonas K. Sunde [a, *], Duncan N. Johnstone [b], Sigurd Wenner [c], Antonius T.J. van Helvoort [a], Paul A. Midgley [b], Randi Holmestad [a]

T-($Al_{20}Cu_2Mn_3$) phase dispersoids are important for limiting recovery and controlling grain growth in Al-Cu alloys. However, these dispersoids can also reduce precipitation hardening by acting as heterogeneous nucleation sites and may lead to increased susceptibility towards pitting corrosion when galvanically coupled with S-(Al_2CuMg) phase precipitates. The interplay between T- and S-phases is therefore important for understanding their effect on the mechanical and electrochemical properties of Al-Cu-Mg alloys. Here, the crystallographic relationships between the T-phase, S-phase, and surrounding Al matrix were investigated in an Al-1.31Cu-1.14Mg-0.13Ag-0.10Fe-0.28Mn (at.%) alloy by combining scanning precession electron diffraction with misorientation analysis in 3-dimensional axis-angle space and high-resolution transmission electron microscopy. Orientation relationships are identified between all three phases, revealing S-T orientation relationships for the first time. Differences in S-Al orientation relationships for precipitates formed at T-phase interfaces compared to their non-interfacial counterparts were also identified. These insights provide a comprehensive assessment of the crystallographic relationships in T-/S-phase aggregates, which may guide future alloy design.



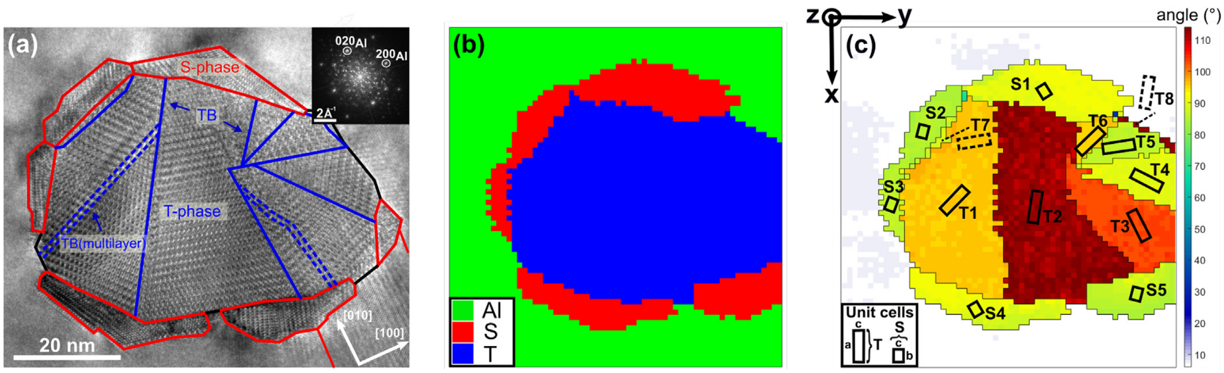


Fig. 4. Structure of a shell-shaped lath-shaped T-/S-phase aggregate. (a) HRTEM image of the aggregates with S-phases and T-phase TBs/TB multilayers indicated. Insert show the image fast fourier transform. (b) Phase map obtained via template matching of SPED data. (c) Orientation map showing the disorientation angle u taken about an axis r (Eq. (1)) at each probe position relative to the specimen reference frame (x,y,z). The average orientation of each labelled domain/phase is indicated by drawn unit cells of the T- and S-phase (not to scale).

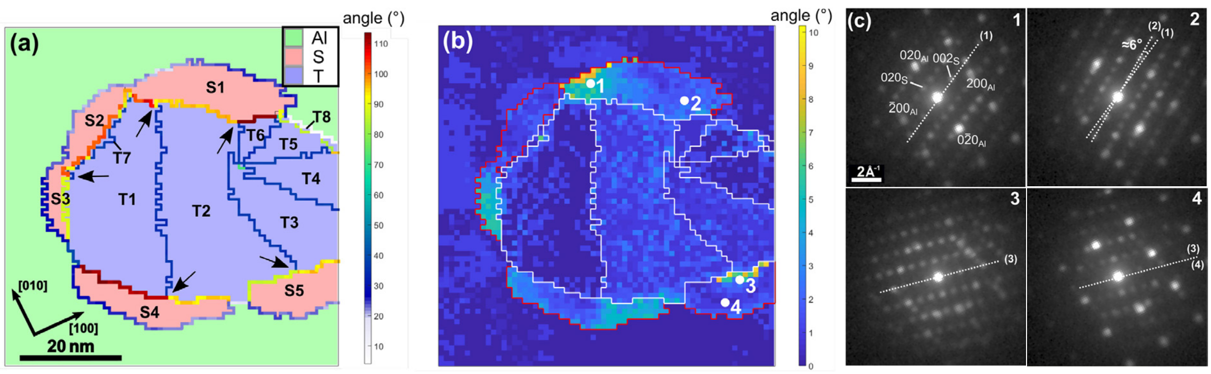


Fig. 9. (a) Phase map of a T-/S-phase aggregate with phase/domain boundaries coloured according to the misorientation angle between neighbouring pixels. Arrows highlight abrupt changes in misorientation angle across S-T boundaries. This is the same dispersoid aggragate as shown in Fig. 4(a). (b) Misorientation with respect to the mean for each phase/domain, respectively. T-phase domains are highlighted by white lines, and red lines mark S-phase cross-sections. (c) The PED patterns from the numbered pixels 1e4 in (b). Dashed lines (1e4) from the associated PED pattern 1e4 is drawn to compare orientations.

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Critically testing olivine-hosted putative martian biosignatures in the Yamato 000593 meteorite—Geobiological implications

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Abstract

On rocky planets such as Earth and Mars the serpentinization of olivine in ultramafic crust produces hydrogen that can act as a potential energy source for life. Direct evidence of fluid–rock interaction on Mars comes from iddingsite alteration veins found in martian meteorites. In the Yamato 000593 meteorite, putative biosignatures have been reported from altered olivines in the form of microtextures and associated organic material that have been compared to tubular bioalteration textures found in terrestrial sub-seafloor volcanic rocks. Here, we use a suite of correlative, high-sensitivity, in situ chemical, and morphological analyses to characterize and re-evaluate these microalteration textures in Yamato 000593, a clinopyroxenite from the shallow subsurface of Mars. We show that the altered olivine crystals have angular and micro-brecciated margins and are also highly strained due to impact-induced fracturing. The shape of the olivine microalteration textures is in no way comparable to microtunnels of inferred biological origin found in terrestrial volcanic glasses and dunites, and rather we argue that the Yamato 000593 microtextures are abiotic in origin. Vein filling iddingsite extends into the olivine microalteration textures and contains amorphous organic carbon occurring as bands and sub-spherical concentrations <300 nm across. We propose that a martian impact event produced the micro-brecciated olivine crystal margins that reacted with subsurface hydrothermal fluids to form iddingsite containing organic carbon derived from abiotic sources. These new data have implications for how we might seek potential biosignatures in ultramafic rocks and impact craters on both Mars and Earth.

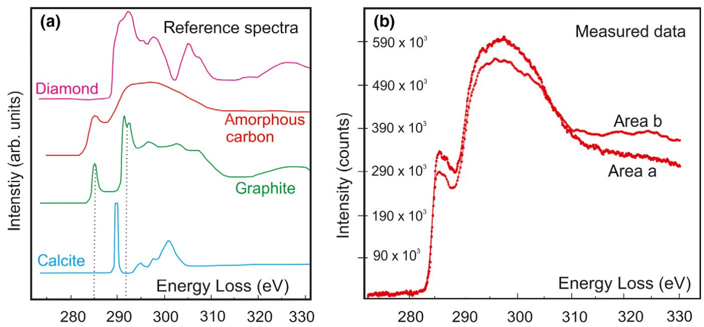

FIGURE 9 Structure of the organic carbon found in Y000593 revealed by Energy Electron Loss Spectroscopy (EELS). (a) carbon-K-edge EELS reference spectra for calcite, graphite, amorphous carbon, and diamond, with the dashed lines showing the 1s to ϖ* electronic transition at ~285 eV and the 1s to σ* exciton at ~292 eV for graphite. (b) carbon-K-edge spectra measured from the red boxes in areas a and b of FIB-lamellae- 3 closely match the amorphous carbon reference spectrum shown in (a). EELS carbon-K-edge reference spectra were taken from (Garvie & Busek, 2006).

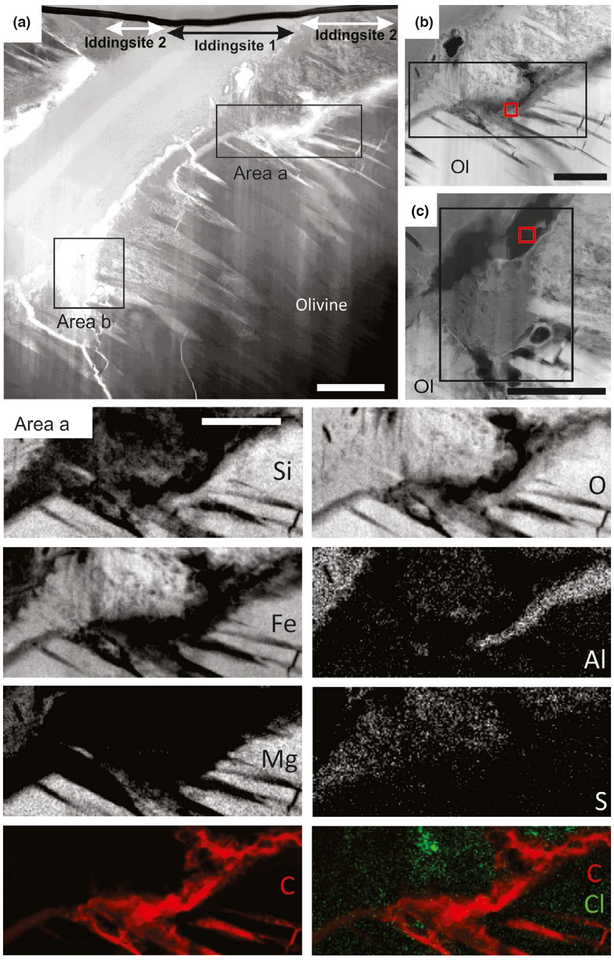

FIGURE 6 Distribution and elemental mapping of olivine (Ol), iddingsite alteration and organic carbon in Y000593. (a) BF (bright field) STEM image of FIB-lamella-3 (for location in sample see Figure 3b) the black boxes correspond to areas that were selected for element mapping. (b) High-angle annular dark field (HAADF) STEM image of Area a, contains fractured olivine (lower right) adjacent to iddingsite (upper left), element maps shown beneath. (c) HAADF STEM image of Area b on the margin of a brecciated and altered olivine crystal (lower right), elemental maps shown in Figure 7. Area a shows a pronounced band of carbon along the margins of the olivine crystal near the boundary with the iddingsite, also in the fractures that brecciate the olivine crystal margin. Crucially the C (red) and Cl (green) maps do not correlate, excluding epoxy as a source for the organic carbon. Element maps were measured using the EDS detector except for the carbon map which is a K-edge EELS map. Red boxes in b and c correspond to locations where Carbon EELS spectra were measured, see Figure 9. Scale bars are 2 μm in (a) and 1 μm in (b and c and the elemental maps) [Colour

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GaN/AlGaN Nanocolumn Ultraviolet Light-Emitting Diode Using Double-Layer Graphene as Substrate and Transparent Electrode

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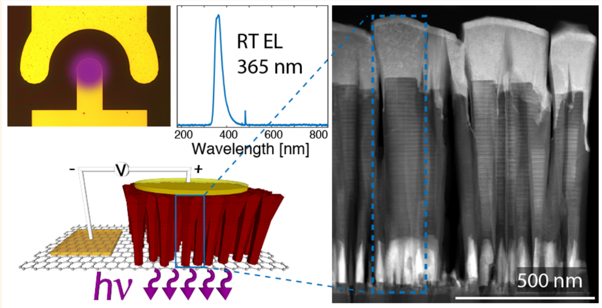
ABSTRACT: The many outstanding properties of graphene have impressed and intrigued scientists for the last few decades. Its transparency to light of all wavelengths combined with a low sheet resistance makes it a promising electrode material for novel optoelectronics. So far, no one has utilized graphene as both the substrate and transparent electrode of a functional optoelectronic device. Here, we demonstrate the use of double-layer graphene as a growth substrate and transparent conductive electrode for an ultraviolet light-emitting diode in a flip-chip configuration, where GaN/AlGaN nanocolumns are grown as the light-emitting structure using plasma-assisted molecular beam epitaxy. Although the sheet resistance is increased after nanocolumn growth compared with pristine double-layer graphene, our experiments show that the double-layer graphene functions adequately as an electrode. The GaN/AlGaN nanocolumns are found to exhibit a high crystal quality with no observable defects or stacking faults. Room-temperature electroluminescence measurements show a GaN related near bandgap emission peak at 365 nm and no defect-related yellow emission.



KEYWORDS: *Graphene, semiconductor nanocolumn, UV optoelectronics, LED, nitride-based devices, electrical injection*

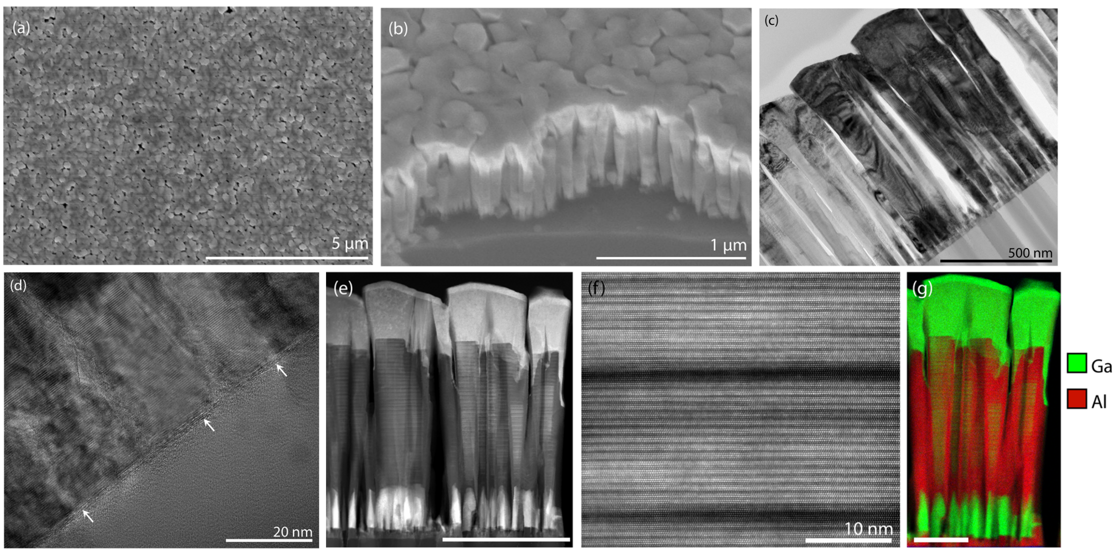


Figure 1. Overview of self-assembled GaN/AlGaN nanocolumns by SEM and their structural details by TEM. (a,b) Top- and bird-view SEM images of nanocolumns grown on DLG transferred onto amorphous silica glass. (c) BF TEM image of the nanocolumns shows local preferential orientation as several neighboring nanocolumns are simultaneously on-axis, indicating epitaxy with the graphene substrate. The nanocolumns grow in the [0001]-direction. (d) High-resolution BF TEM image showing the DLG (white arrows) between the amorphous silica glass support and the crystalline nanocolumns. (e) A superlattice-like structure can be observed by HAADF STEM (scale bar 500 nm). (f) High-resolution HAADF STEM image of alternating Al-rich/Ga-rich layers in the superlattice-like n-AlGaN nanocolumn segment. (g) Compositional mapping by EDS and EELS of the different GaN/AlGaN segments shows an actual nanocolumn heterostructure consisting of n-AlN/n-GaN/n-$Al_{0.76}Ga_{0.24}N$/p-$Al_{0.42}Ga_{0.58}N$/p-GaN (scale bar 200 nm).

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Nanocrystal segmentation in scanning precession electron diffraction data

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Summary

Scanning precession electron diffraction (SPED) enables the local crystallography of materials to be probed on the nanoscale by recording a two-dimensional precession electron diffraction (PED) pattern at every probe position as a dynamically rocking electron beam is scanned across the specimen. SPED data from nanocrystalline materials commonly contain some PED patterns in which diffraction is measured from multiple crystals. To analyse such data, it is important to perform *nanocrystal segmentation* to isolate both the location of each crystal and a corresponding representative diffraction signal. This also reduces data dimensionality significantly. Here, two approaches to nanocrystal segmentation are presented, the first based on virtual dark-field imaging and the second on non-negative matrix factorization. Relative merits and limitations are compared in application to SPED data obtained from partly overlapping nanoparticles, and particular challenges are highlighted associated with crystals exciting the same diffraction conditions. It is demonstrated that both strategies can be used for nanocrystal segmentation without prior knowledge of the crystal structures present, but also that segmentation artefacts can arise and must be considered carefully. The analysis workflows associated with this work are provided open-source.

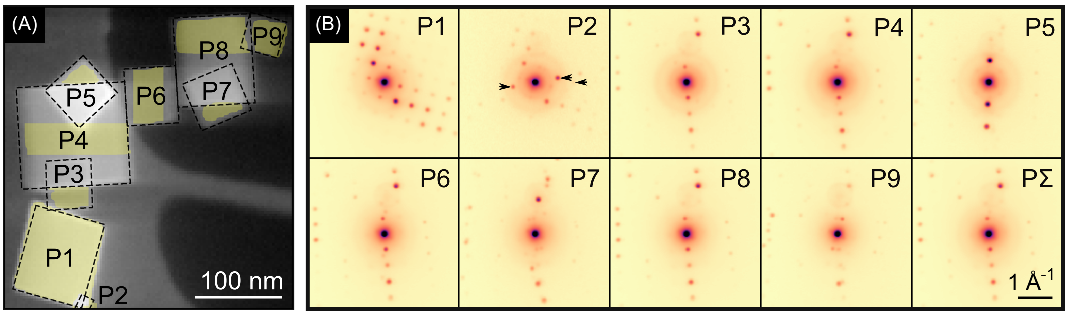

Fig. 3. (A) Annular virtual dark-field (VDF) image showing nine magnesium oxide (MgO) particles (grey), labelled P1–P9, lying on top of a holey amorphous carbon film (dark grey) or over vacuum (black). The outlines of the MgO particles are indicated by dashed rectangles. (B) Sum of PED patterns within the yellow areas in (A). The detected diffraction vectors of P2 are marked by black arrows. PΣ is the sum of P3, P4, P6 and P8.

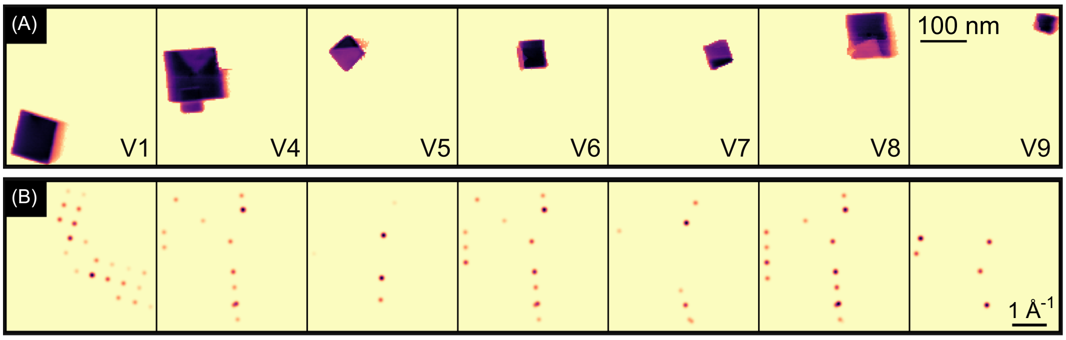

Fig. 4. Virtual dark-field (VDF) imaging-based segmentation results. (A) Summed VDF image segments (V_{SS}), labelled V1 and V4–V9 according to the particle numbering in Figure 3, and (B) corresponding virtual diffraction patterns (P_D).

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In-situ observations of dislocation recovery and low angle boundary formation in deformed aluminium

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Abstract. An experimental study of the recovery of dislocations and low angle boundary formation in aluminium is presented. By combining *in-situ* annealing with orientation mapping in the transmission electron microscope, maps of geometrically necessary dislocation estimates derived from orientation measurements and subgrain formation can be quantitatively analysed. A thin foil of a commercially pure aluminium alloy cold-rolled to a true strain of $\varepsilon = 2.3$ and annealed *in-situ* in four steps of increasing temperatures from 170 °C to 560 °C was studied. An increase in the subgrain size and low angle boundary misorientation was accompanied by a halving of the dislocation density from $1.2 \times 10^{16}\,\mathrm{m}^{-2}$ to $0.6 \times 10^{16}\,\mathrm{m}^{-2}$. Limited boundary migration was observed and the increased subgrain size was attributed to the dissolution of dislocations within the low angle boundaries upon annealing.

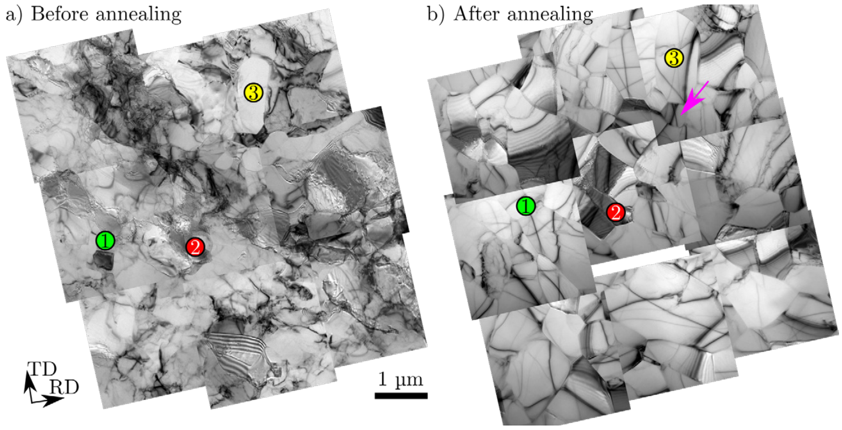

Figure 2. BF TEM images of the ROI (a) before annealing and (b) after the final annealing step. The numbered dots (1-3) indicate the same positions.

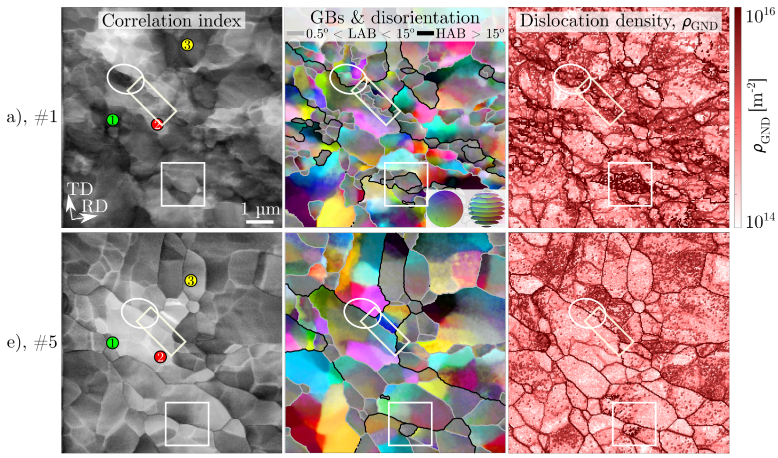

Figure 3. Substructure evolution from the PED scans (#1, #5). (Left) Maps of correlation index, (middle) grain boundaries and disorientation colouring and (right) dislocation densities ρ_{GND} (a) before annealing and (e) after the final annealing step. See figure 2 and the text for descriptions of dots and shapes, respectively.

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