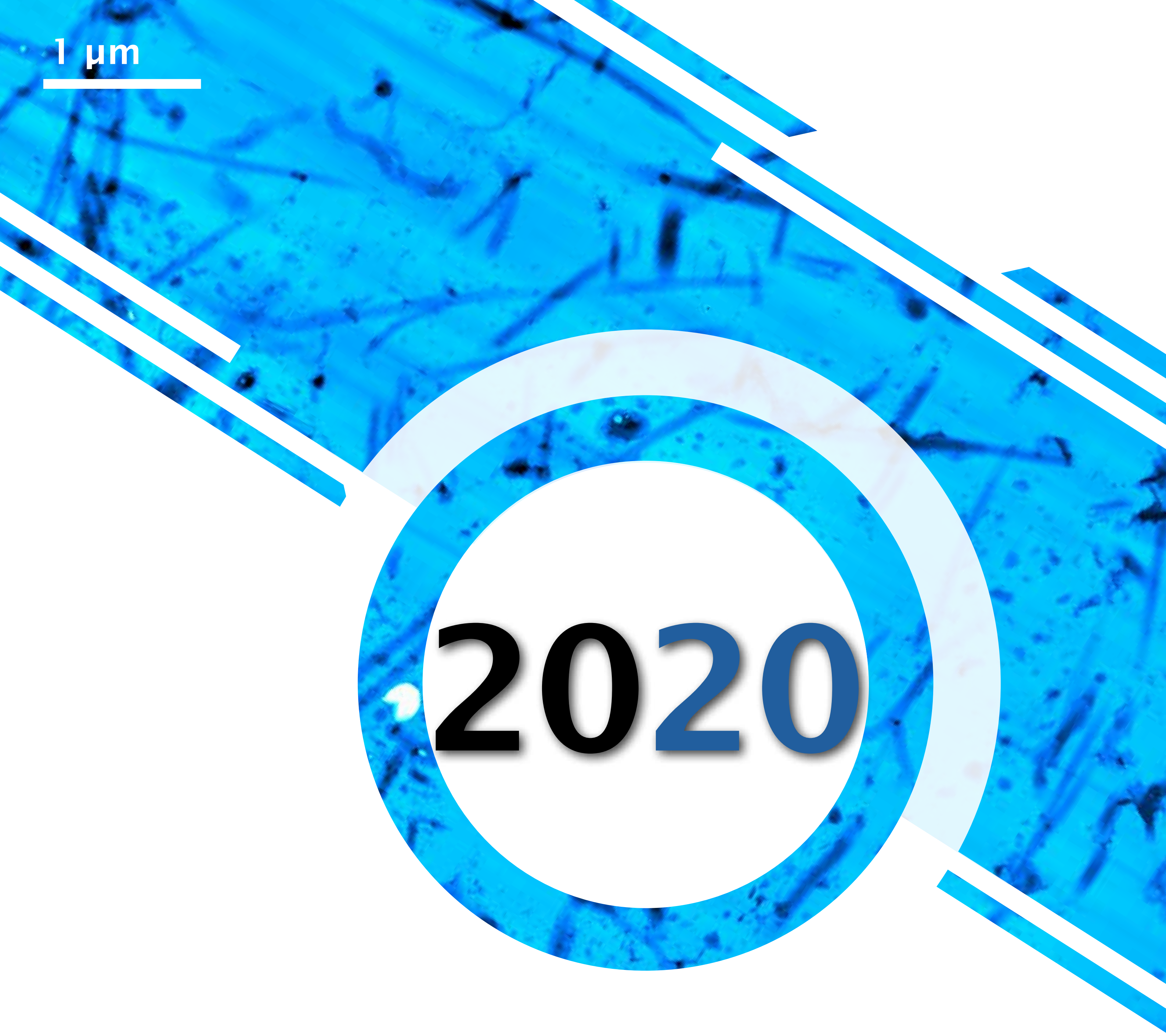
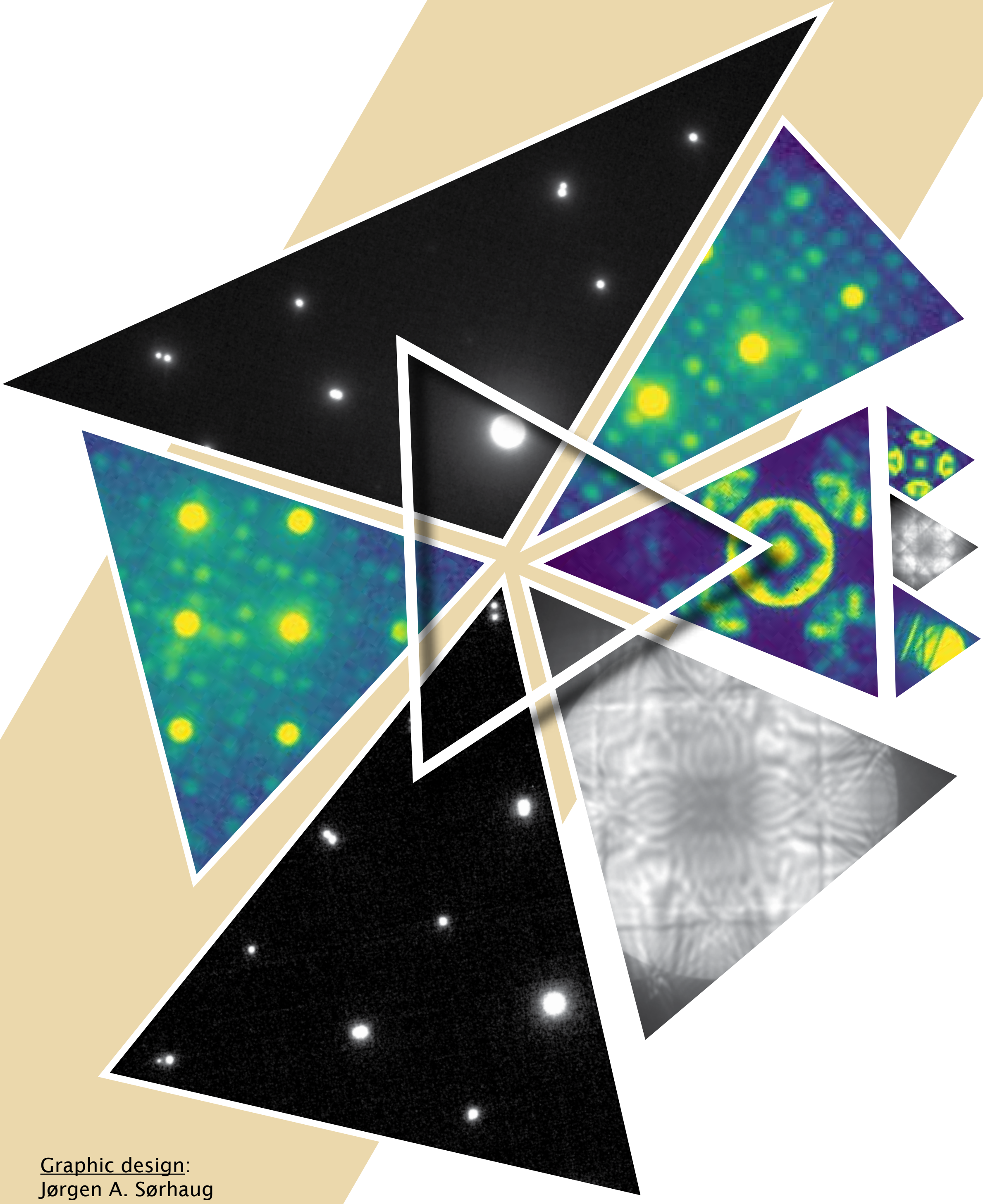


2020

Annual report

TEM GEMINI CENTRE

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



<u>Graphic design</u>:
Jørgen A. Sørhaug
& Gregory Nordahl.

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

Images on front and back: *BF-STEM C-fibres in conductive concrete, by S. Wenner.*

Images above *show different diffraction techniques, and are acquired by I.-E. Nylund, E. Thronsen and C. Hell.*

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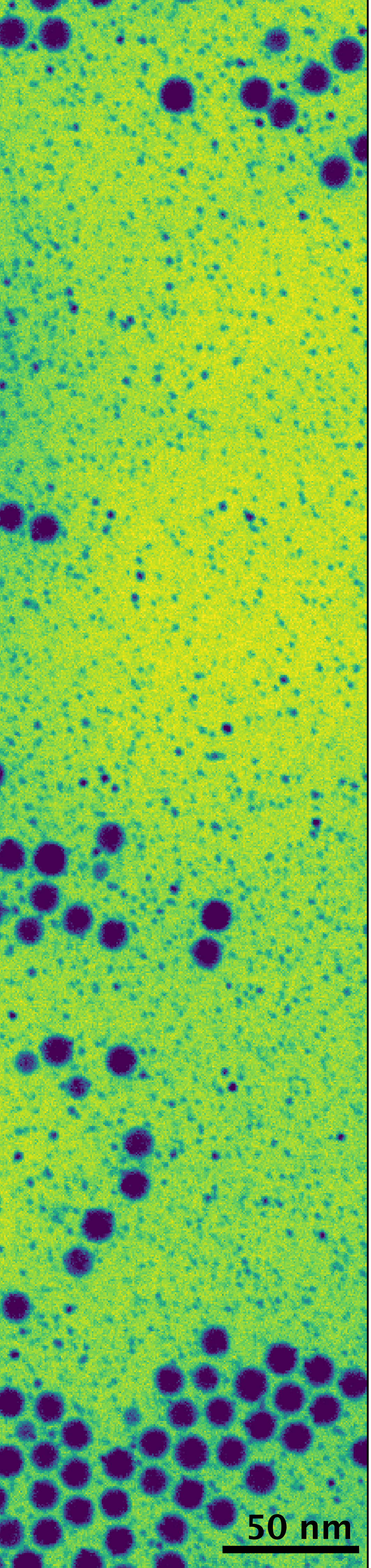


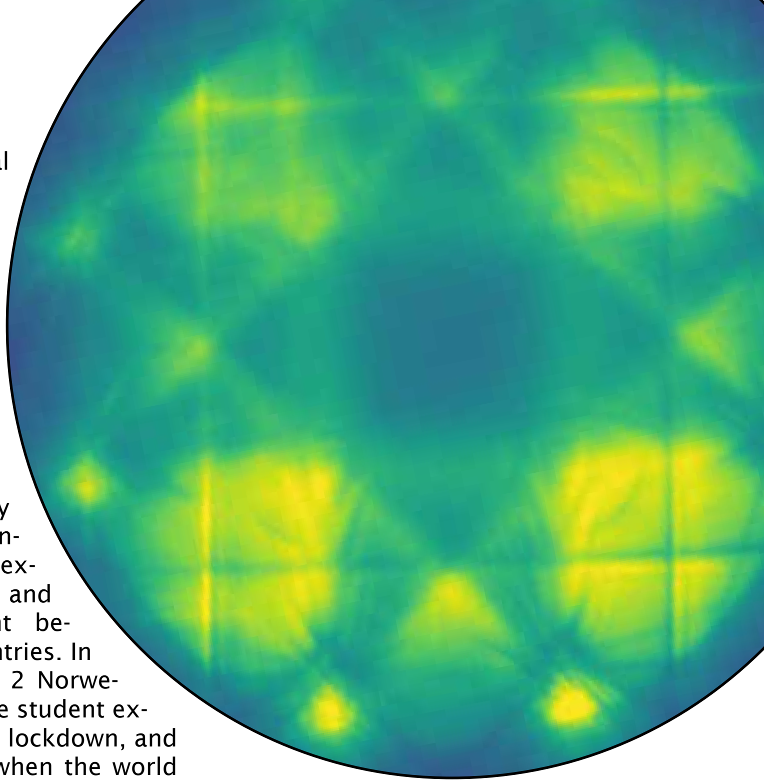
***Image to the left**: HAADF STEM of iron oxide-silver nano-particles, by I.-E. Nylund.*

***Image on next page:** LACBED from Al [001] ZA by C. Hell.*

INTRODUCTION

As for the rest of the world, 2020 has been a strange year for the TEM Gemini Centre - as we were all hit by the Covid-19 pandemic. We have managed to keep the TEM lab open all the time, due to strict infection control and measures to reduce transmission risk. We can see that the structured work of building up good routines, gives return. In spite of the difficulties, we have kept our activities through this year - kept users of the infrastructure and are close to normal output in scientific papers and deliverables on projects. We had a successful installation of a direct camera in spite of pandemic and travel restrictions, thanks to extra effort from QuantumDetectors Nanomegas and the local engineers. We have been working on extending and applying our competence around the NORTEM Trondheim node instruments. 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 a consistent trend of increasingly high level of use and quality of scientific and educational output achieved during the last years.

The total cost model for lab infrastructure introduced by NTNU has been fully implemented in NORTEM for many years. 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.



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, providing access, facilitating, and extending transnational access services. This has been hampered by the pandemic, but we have managed to keep most of the activities in the project. The clustering seminar we planned to organize in October was cancelled. Larger conferences have turned to virtual, and members of the group have participated online.

In November 2020 we submitted a proposal for NORTEM II to the INFRA program in the Research Council. NORTEM II is necessary to keep our instruments on the level we want, but also a big effort and commitment for the whole environment and the Gemini Centre. We will learn the outcome in the summer of 2021. Fingers crossed! :-)

Among the good news this year is that Magnus Nord started as an associate professor in the TEM group in April. He was also granted a researcher project for young talents on TEM studies of magnetic materials in December. Also, Sigmund Andersen got granted a new project, on quasicrystals. In addition, a new SFI in physical metallurgy, [SFI PhysMet](#), was granted, where the TEM Gemini Centre is involved in one research area. The Nordic collaboration project NordTEM-Hub was funded through Nordforsk, headed by Per Persson from Linköping with 7 partners with NTNU as one of them.

The Gemini Centre participates in a broad range of projects, including national, public, industrial and EU funded 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. The two first ones ended in 2020. The INTPART project with Japanese aluminium industry and academia is ongoing (to 2023) exchanging Japanese and Norwegian student between the two countries. In 2020 we managed 2 Norwegian and 1 Japanese student exchanges before the lockdown, and hope to continue when the world is coming back on track.

The Gemini Centre participates in a broad range of projects, including national, public, industrial and EU funded ones.

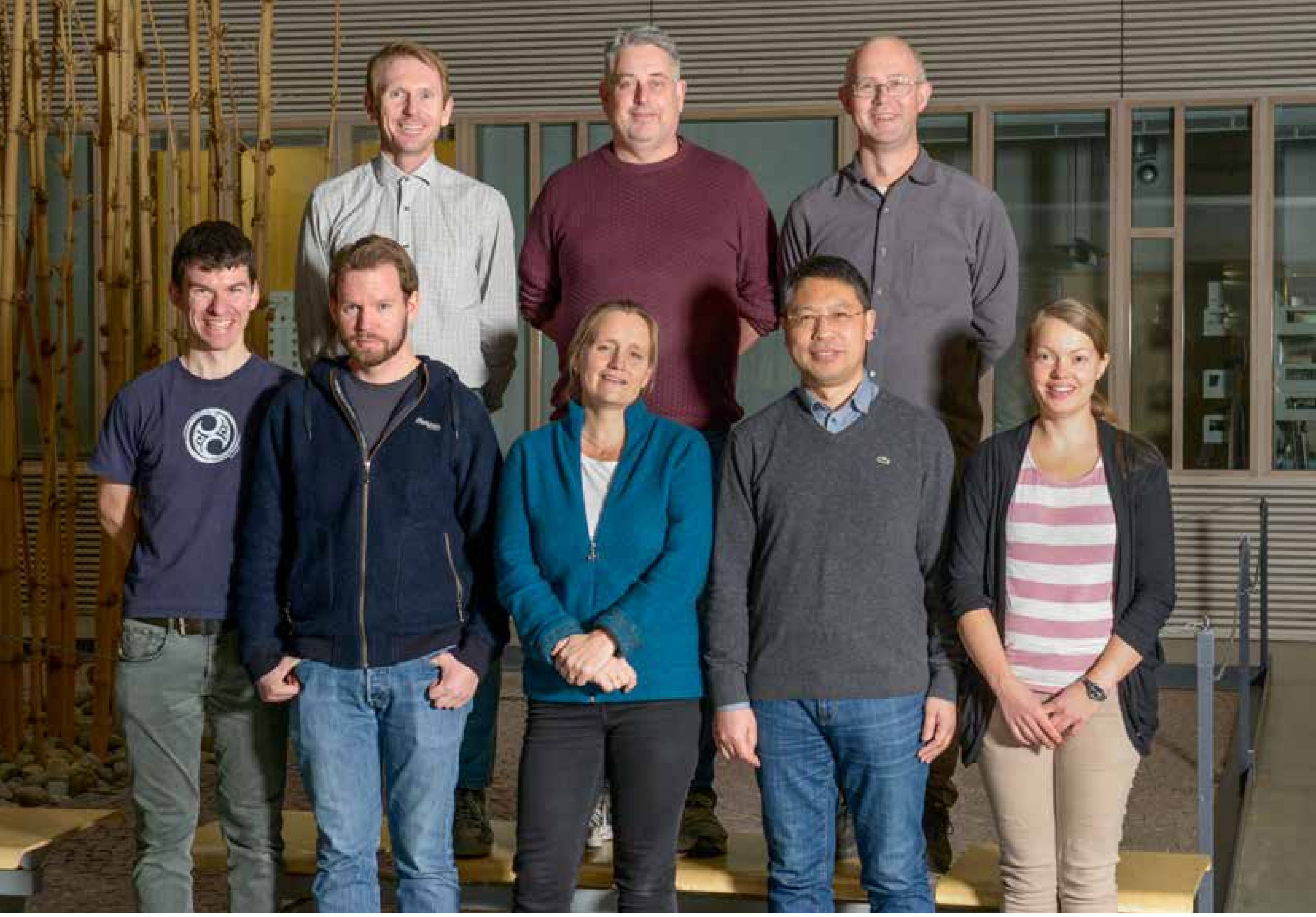
As documented in this report, the Centre had 36 active hands-on users/operators, 18 users through operators and served around ~100 different projects, whose results have contributed to 39 journal publications (plus 10 in press) in 2020. Many of the publications have international co-authors. TEM Gemini Centre publications are found in a broad range of journals and cover a spectrum of topics, showing how generic TEM is. In addition, 5 PhD students and 8 MSc candidates were educated with TEM as a substantial part of their theses in 2020. Due to the pandemic, less NTNU courses used the facility this year.

The annual TEM introduction course has this year been made digital, and several (30-40) people attended. We had group meetings with presentations, from March all via Zoom/Teams, with about 20 participants almost every week.

In September, we managed to install Norway's first direct electron detection (DED) camera, dedicated for electron diffraction on our 2100F instrument after 5 months delay because of the pandemic. Discussions are going on about the Campus project and we are very concerned about 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. Meanwhile, we must deal with and hope for stable conditions in Chemistry building I.

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 2020. For more details, see our home page: ntnu.edu/geminicentre/tem

TEM Gemini Centre management, February 2021.



Tem Gemini Center management group.
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.*

BOARD AND MANAGEMENT OF TEM GEMINI CENTRE

TEM Gemini Centre board:

- Erik Wahlström, Department head, Department of Physics, NTNU
- Ragnar Fagerberg, Research manager, Materials Physics, SINTEF Industry
- Jostein Mårdalen, Department of Materials Science and Engineering (until February), Einar Magne Hjorthol (from August), Department head

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 (until end of March)
- Emil CHristiansen, Physics, NTNU (from April)
- Yanjun Li, Materials Science and Engineering, NTNU
- Per Erik Vullum, Materials Physics, SINTEF Industry
- Ruben Bjørge, Materials Physics, SINTEF Industry

Group picture of people in the TEM Gemini Centre taken January 2020.

PEOPLE IN THE TEM GEMINI CENTRE IN 2020

- Sigmund J. Andersen (Senior research scientist, SINTEF)
- Ragna Bakke (Master student, DP, NTNU)
- Ruben Bjørge (Research scientist, SINTEF)
- Tina Bergh (PhD student, DP, NTNU)
- Dipanwita Chatterjee (Postdoc, DP, NTNU)
- Emil Christiansen (Postdoc, Structural Engineering/ Senior Engineer, DP, PhD student, DP, NTNU)
- Jonas Frafjord (PhD student, DP, NTNU)
- Jesper Friis (Senior research scientist, SINTEF and Assoc. Prof. II, DP, NTNU)
- Sigrid Wanvik Haugen (Master student, DP, NTNU)
- Christoph M. Hell (PhD student, DP, NTNU)
- Ton van Helvoort (Prof., DP, NTNU)
- Randi Holmestad (Prof., DP, NTNU / Leader TEM Gemini Centre)
- Kasper Aas Hunnestad (PhD student, DMSE, NTNU)
- Endre Jacobsen (Master student, DP, NTNU)
- Adrian Lervik (PhD student, DP, NTNU)
- Yanjun Li (Prof., DMSE, NTNU)
- Ursula Ludacka (Postdoc, DMSE)
- Hogne Lysne (PhD student, DP, NTNU)
- Calin Marioara (Senior research scientist, SINTEF)
- Knut Marthinsen (Prof., DMSE, NTNU)
- Aleksander Mosberg (PhD student, DP, NTNU)
- Hanne Mørkeseth (Master student, DP, NTNU)
- Julie Stene Nilsen (PhD student, DP, NTNU)
- Magnus Nord (Assoc. Prof. DP, NTNU)
- Gregory Nordahl (PhD student, DP, NTNU)
- Inger-Emma Nylund (PhD student, DMSE, NTNU)
- Eirik Opheim (Master student, DP, NTNU)
- Ding Peng (Postdoc, DP, NTNU)
- Øystein Rolstad (Master student, DP, NTNU)
- Bjørn Gunnar Soleim (Senior engineer, DP, NTNU)
- Jonas Kristoffer Sunde (PhD student, DP, NTNU)
- Ragnhild Sæterli (Senior engineer, DP, NTNU)
- Jørgen Sørhaug (PhD student, DP)
- Tor Inge Thorsen (Master student, DP, NTNU)
- Elisabeth Thronsen (PhD student, DP, NTNU)
- Hursanay Turgun (Master student, DP, NTNU)
- Haakon Tvedt (Master student, DP, NTNU)
- Per Erik Vullum (Senior research scientist, SINTEF and Assoc. Prof. II, DP, NTNU)
- Sigurd Wenner (Research scientist, SINTEF and Assoc. Prof. II, DP, NTNU)
- Yingda Yu (Senior engineer, DMSE, NTNU)
- Håkon Wiik Ånes (PhD student, DMSE, NTNU)

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 each of the two geographical nodes, Trondheim and Oslo. We have now been running the facility for close to seven years. The support to NORTEM from the Research Council ended in 2016, but the project continued to the end of 2020. In October 2018 we submitted a proposal for the follow-up project NORTEM II, which was not funded. We have again applied in November 2020. The proposal includes upgrading and new instruments in both nodes. In the Trondheim node, we applied for a new state-of-the-art probe corrected Level 1 instrument with modern cutting-edge DEDs, advanced probe-forming systems with more flexibility in illumination, higher voltage (300 kV), improved mechanical and thermal stability and increased automation, focused on structure determination, diffraction and electric/magnetic field imaging.

Key numbers for NORTEM:

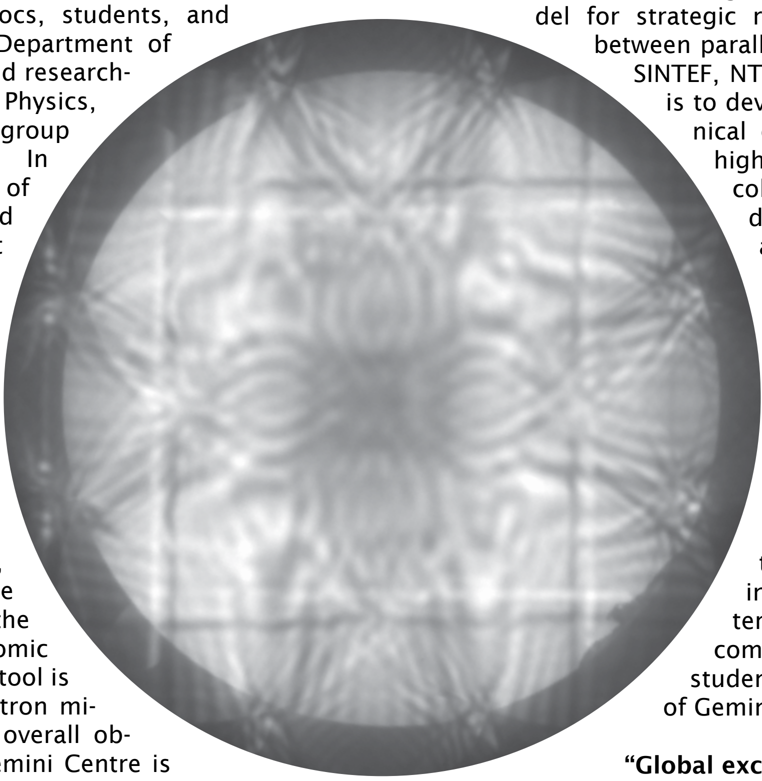
- 3 partners - SINTEF, NTNU and UiO;
- 2 nodes - Trondheim and Oslo;
- 58 MNOK from INFRA in 2011;
- 5 microscopes - 2 top level from 2012/2013;
- 143 projects served in 2019;
- > 40 000 h used since 2012;
- ~ 60 users annualy;
- ~ 60 papers annualy (2/3 international co-authors);
- ~ 15 permanent staff in core research groups;

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. 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 had two senior engineers, Bjørn Gunnar Soleim and Ragnhild Sæterli supporting maintenance, training, competence and techniques. Ragnhild quit her position in March 2020. Since April, Emil Christiansen has worked in a 50% engineer position. We have a high uptime and ca. 7% 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 I.-E. Nylund.

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 nanometer 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.

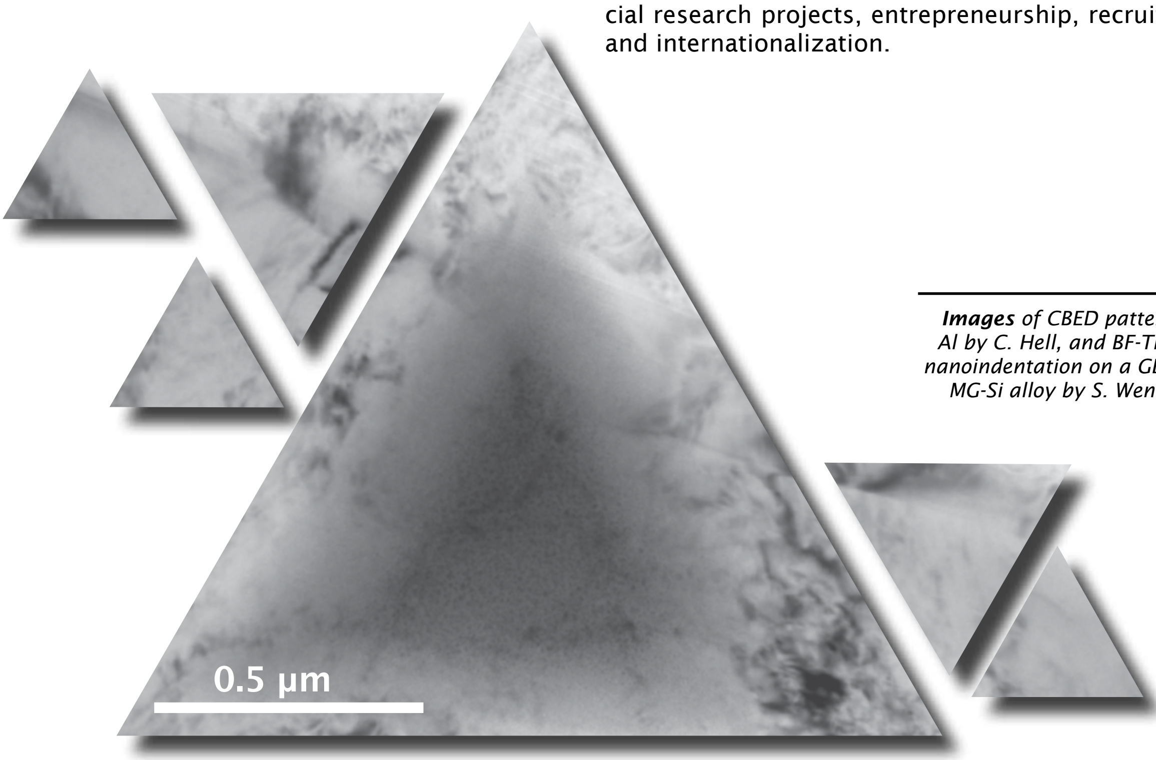


GEMINI CONCEPT

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 internationalization.



***Images** of CBED pattern of Al by C. Hell, and BF-TEM of nanoindentation on a GB in Al-MG-Si alloy by S. Wenner.*

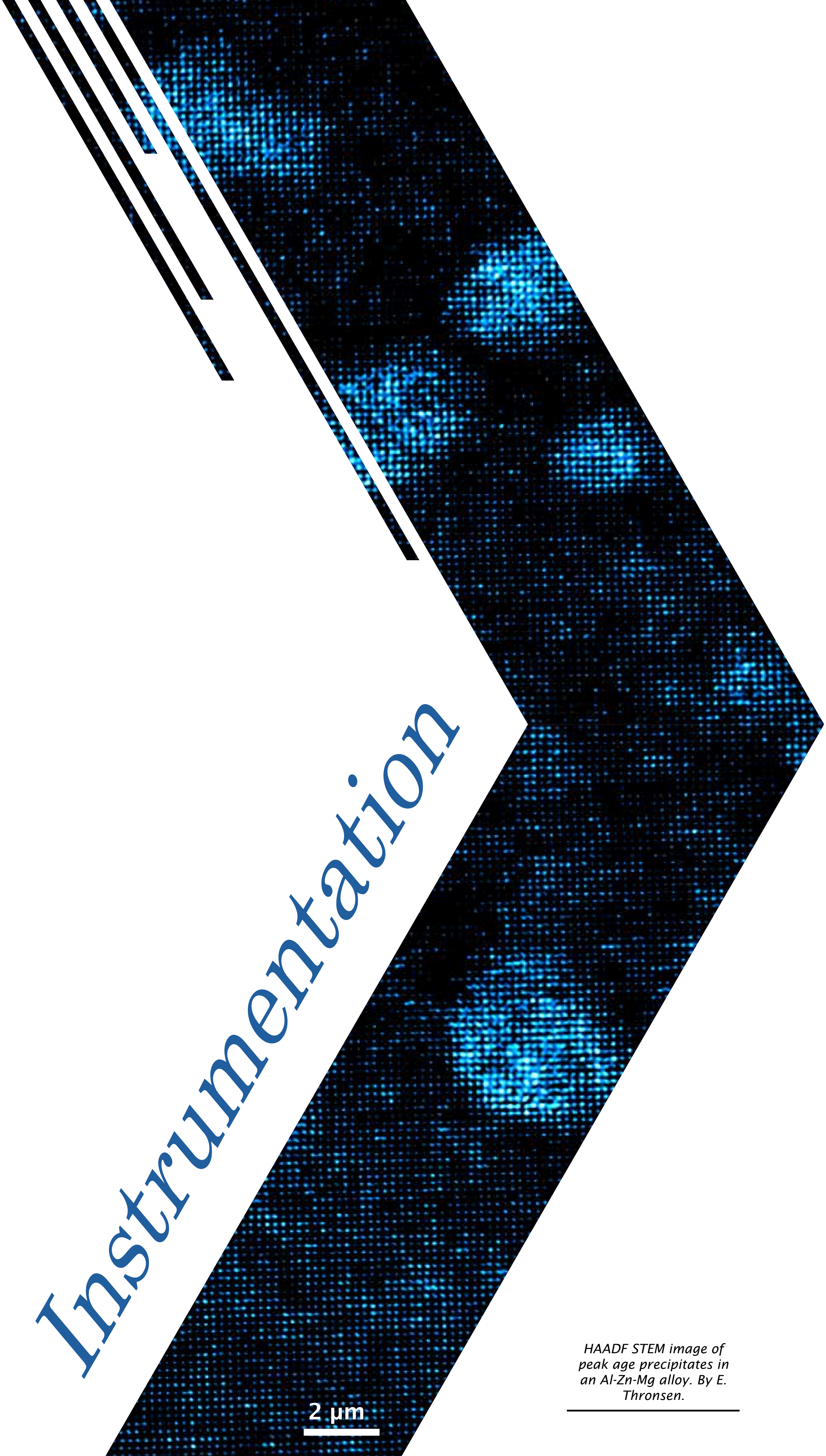
Elisabeth and Emil testing out the new directly detector on the 2100F, Sept. 2020.

THANKS TO RAGNHILD SÆTERLI

Unfortunately, the center had to say goodbye to Ragnhild Sæterli. Ragnhild Sæterli did her PhD in the TEM Gemini Centre from 2006 to 2010. After this she was a postdoc in the group up to 2013. She was then hired as an engineer for the NORTEM facility together with Bjørn Gunnar Soleim. In March 2020 she ended her job with us to start a new position in SINTEF Energy. She has done a tremendous job for the TEM facilities in Trondheim, and we want to thank Ragnhild for the big efforts! She has been teaching the TEM introduction course, trained many students in TEM, given a huge number of high school guided tours, organized workshops, kept the instruments in order etc. Ragnhild's position will be continued in 2021, meanwhile Emil Christiansen has been 50% substituting in her position. We want to thank Ragnhild for her great job and good company for 15 years and wish her good luck in her new position!

Head Engineers Dr. R. Sæterli. Photo: O. M. Melgård.

Instrumentation



HAADF STEM image of peak age precipitates in an Al-Zn-Mg alloy. By E. Thronsen.

THE TEM INSTRUMENTS IN TRONDHEIM

The TEM Gemini Centre has three TEMs installed as part of the NORTEM project in 2012/2013 - 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 September 2020)

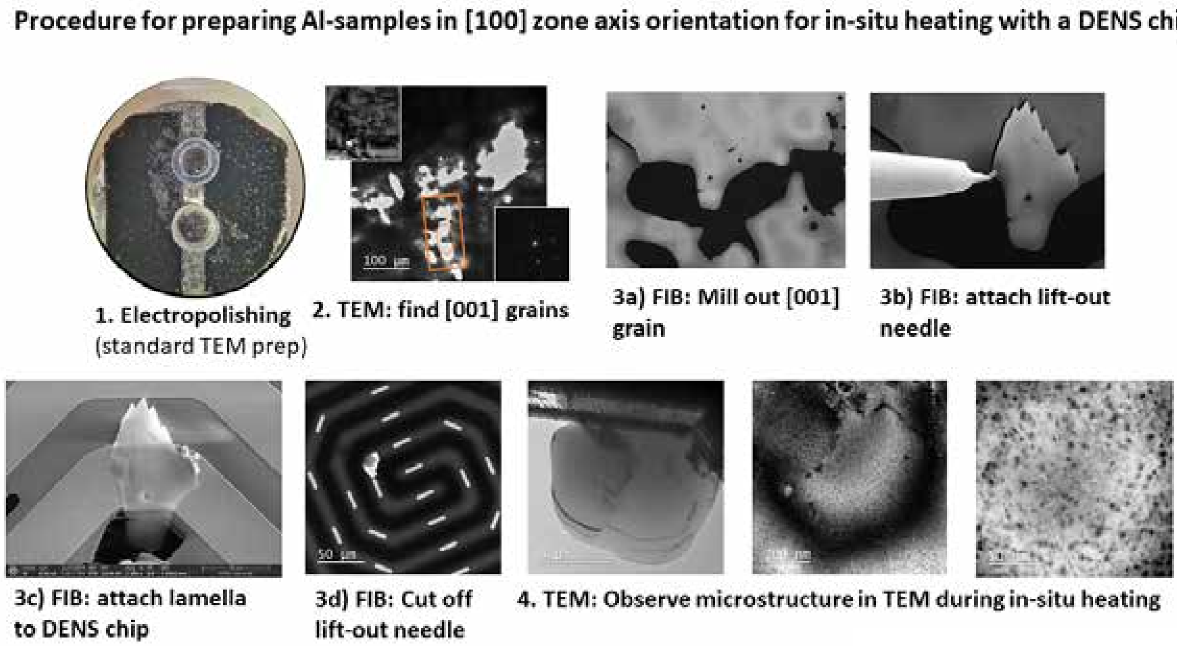
JEOL JEM-2100

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)

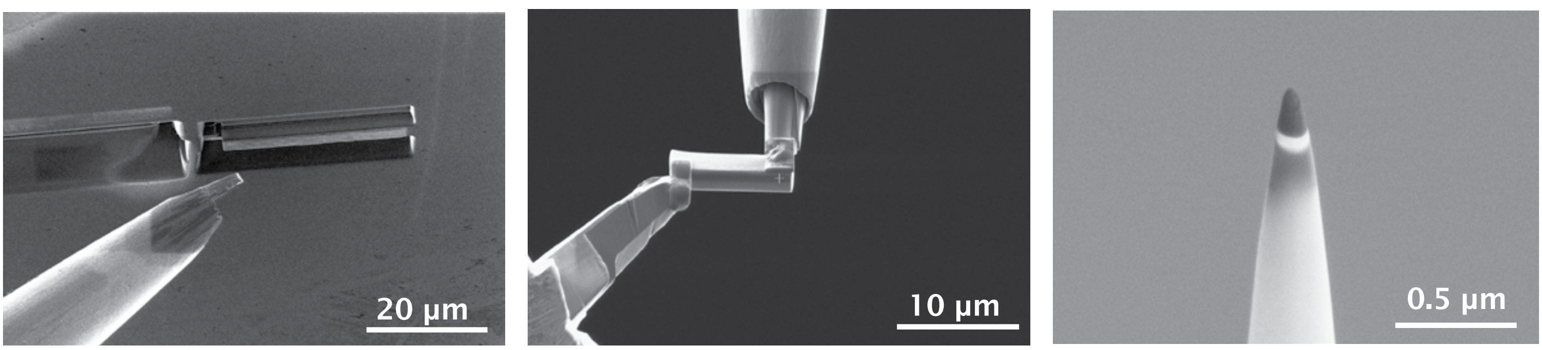
Photos by O. M. Melgård.



Protocol established by T. Bergh & J. K. Sunde for ESTEEM3.

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 one of the two FIB instruments at NTNU NanoLab with lift-out option for site-specific TEM specimen preparation. We also have an inert transfer set-up for FIB-based TEM preparation as 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.



SEM micrographs showing liftout needle relocating an APT lamella from $ErMnO_3$. A finished APT needle of a thin film sample is shown in the last image. By K. A. Hunnestad.

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. The figure on page 11 shows a procedure for preparing TEM lamella of metal materials on the MEMS chip for in-situ TEM studies. We also have a holder tip for TEM characterization of atom probe tomography needles, to enable correlated structural and chemical studies of 100 nm sized volumes.

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 computer room for TEM data analysis has five machines, for postprocessing and simulating TEM results, some of which can be remotely accessed. It also includes more powerful workstations, dedicated to more demanding data processing. In 2020, two new powerful workstations were added to this portfolio to follow up on the increased data load from the new direct electron detector. 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 of the complex TEM data sets created at the facility.

USER STATISTICS IN 2020

The total registered used time for the three instruments in 2020 was 3199 hours, including 177 non-paid hours used for testing, competence development, demonstrations, and guided tours. Of the 3022 paid hours, the use by NTNU corresponds to 71 %, externals (with NTNU operator) 6 %, and SINTEF 23 %. NTNUs use is divided over five departments, where the main use is from Department of Physics (72 % of NTNUs paid hours). 96 different projects used TEM in 2020. The infrastructure had in total 54 users, of which 36 were hands-on operators. 4 of the users were based at SINTEF, 16 were PhD candidates and 13 were master students. During the pandemic, facility was open to users (except ca. 8 weeks not to master students due to campus closure).

Microscope use (hours)	ARM-200F	2100F	2100	Sum
SINTEF	387	190	106	683
NTNU - Physics	376	546	450	1372
NTNU - Other departments	93	242	255	590
NTNU - Visitors from abroad	21	0	15	36
NTNU - Teaching lab	0	95	63	158
External	39	144	0	183
NTNU - Setup/testing/training/ demonstrations	14	153	10	177
Total use	**930**	**1370**	**899**	**3199**

Activities

Research and events

HAADF STEM of disordered Q' precipitate on alpha dispersoid in a Al 6xxx alloy. By D. Chatterjee.

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 2020. 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.

Image *by E. Thronsen.*

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 2020 we have been involved in 2 SFI Centers (plus the new SFI PhysMet which just started!), 3 competence projects (KPN) and one Digitalization project in aluminium research, in addition to the INT-PART 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. Emil Christiansen is hired in the CASA project, as a postdoc in the Structural Engineering department, doing TEM on CASA materials. Since April this has been in 50%, since he has been working as an engineer in the TEM group. Jonas Frafjord 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 behavior in Al alloys. He defended his thesis in December 2020 and is now working as a postdoc in the SumAl project. The 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 characterizes 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). From October 2020, a new postdoc, Ding Peng, started to work on this SFI in the TEM group. Ding will study additive manufactured materials in the TEM.

The project 'In-Sane' - In-situ studies of highly conductive bonded interfaces between aluminium and copper at the nanoscale – started in 2020 and is a Nano2021 project in collaboration with Department of Mechanical and Industrial Engineering. The idea is to perform nanoscale joining in the FIB at nanoscale, in order to understand and develop the HYB (Hybrid Metal Extrusion & Bonding) method. The motivation for In-Sane is to produce dissimilar and highly conductive Cu/Al microjoints with strong and sharp interfaces for battery power packs. PhD student Jørgen Sørhaug is hired on the project from August 2020 and will do advanced TEM on the joints. In addition, one PhD student (Ambra Celotto) is hired at Department of Mechanical and Industrial Engineering.

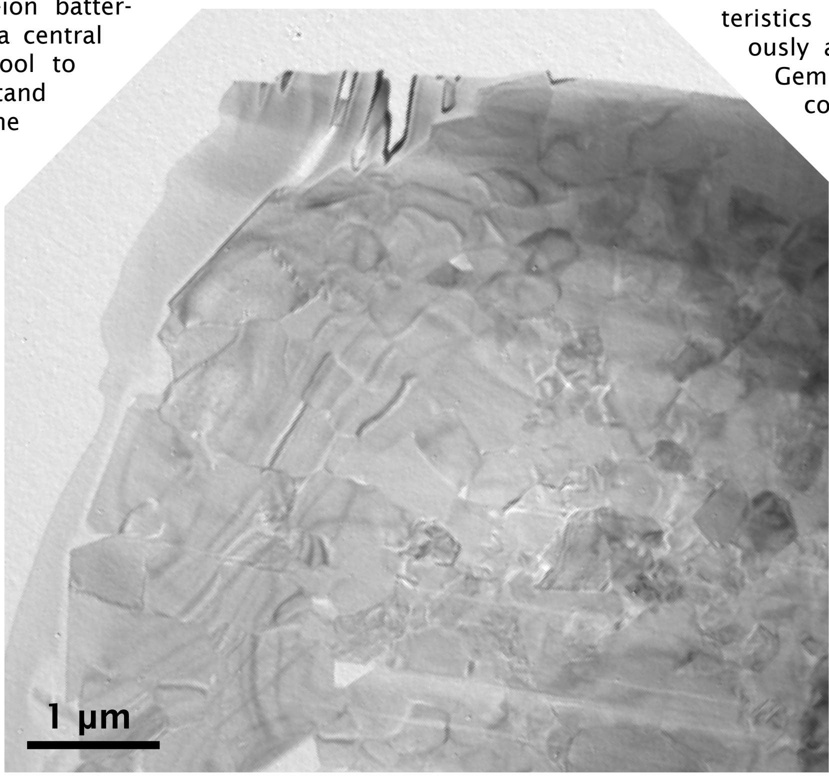
Two competence projects involving NTNU and SINTEF on aluminium research ended in 2020. The first one is the project 'Fundamentals of intergranular corrosion in aluminium alloys' (FICAL) where Randi Holmestad is the project leader. FICAL was a 5 years project that had the objective of establishing new fundamental understanding of the mechanisms of intergranular corrosion (IGC) in aluminium. Industrial funding was provided by a consortium of four aluminium companies; Hydro Aluminium, Benteler, Gränges and Steertec, representing the entire aluminium value chain. The mechanisms of IGC were studied at the nm-scale utilizing advanced laboratory infrastructure, especially TEM, plus modelling. Adrian Lervik worked as a PhD student in the FICAL project focusing on quantitative understanding of nanoscale structure and chemistry related to IGC. One concrete case studied was stress corrosion cracking in 7xxx alloys from Benteler Automotive. Adrian finished his PhD in September 2020 and has now started a new job in FFI at Kjeller. The second competence project with aluminium industry ending in 2020 was the project 'Aluminium alloys with mechanical properties and electrical conductivity at elevated temperatures' (AMPERE) project, with Knut Marthinsen as project leader. Here, Al alloys were studied for several combined properties at elevated temperatures. Hydro Aluminium, Gränges, Nexans and Neuman were partners. The project aimed at providing new advances in experimental technologies, experimental databases and developing modelling tools for combinations of aluminium properties. Jonas K. Sunde was a PhD student on this project and studied 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. Jonas defended his PhD thesis in August 2020 and is now working with NORCE in Stavanger. A 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. Christoph Hell was hired from January 2020 to do advanced TEM in this project. SINTEF has a big part of this project, doing TEM, in addition to APT and modelling. 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. As seen from the publication lists of the TEM Gemini Centre, we have the last years had many 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 ACMM in Canberra and ICAA (supposed to be in Grenoble, but went online) , which shows that our work on aluminium is internationally recognized.

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. 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. 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. Julie had her defense in February 2020. 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 (SiBanode)" 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, "HAST", is led by the company CENATE, a spin-off company to Dynatec. This project also 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 the 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 is here 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. TEM is also used in several 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 behavior 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 behavior 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



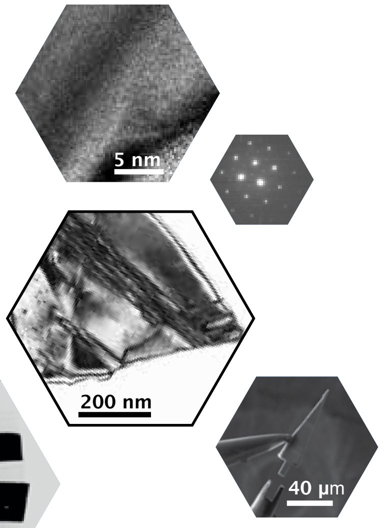
LiMnNiO particle coated with Al_2O_3. By S. Wenner.

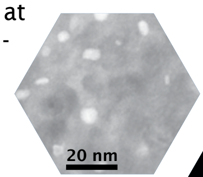
(i.e. nanowires, carbon nanotubes), thin films and 2D-materials such as graphene. These activities are part of NANO@NTNU. NTNU NanoLab is our direct neighbour and many of the TEM operators also use equipment in the cleanroom. Especially the FIBs are important for the TEM Gemini Centre. We developed together 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. Aleksander Mosberg was funded by NTNU's "Enabling technologies: Nanotechnology" and based in the TEM Gemini Centre. Mosberg's PhD focused on using FIB for nanostructuring. TEM is used to understand how the ion beam alters the materials. He used TEM, as element in correlated studies, to study the made structures in detail. Aleksander defended his thesis in October 2020 and is now working as a postdoc and FIB expert at SuperSTEM in Daresbury, UK.

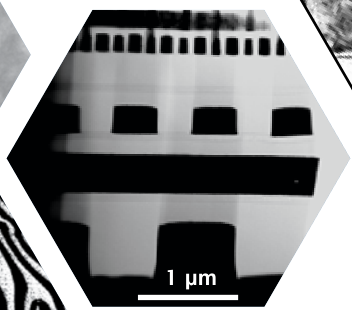
Many of our master students follow the Nanotechnology study program, a further demonstration that TEM is an essential tool for practical nanotechnology. These student projects include both practical as well as theoretical/computational focus. Several core TEM Gemini people are actively involved in nanotechnology 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 2020. 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.

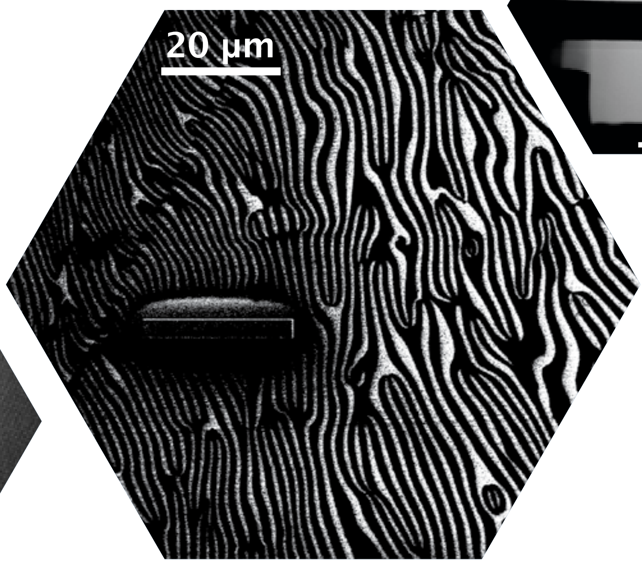
MAGNETIC MATERIALS: IMAGING ELECTRO-MAGNETIC FIELDS

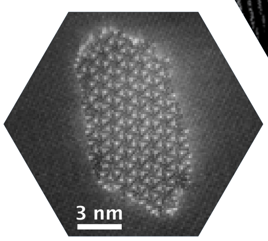
While there has been much work done studying magnetic materials at the TEM Gemini Centre, there has been very little work on directly imaging the magnetic fields themselves. Thanks to recent advances in fast pixelated direct electron detectors this has become easier, making techniques such as scanning TEM-differential phase contrast (STEM-DPC) much more accessible. One of the focuses of newly hired associate professor Magnus Nord is to improve the STEM-DPC technique, utilizing the recently installed MerlinEM fast pixelated direct electron detector, together with PhD student Gregory Nordahl. This activity will increase in 2021, with Magnus Nord being granted a Young Research Talents "In-situ correlated nanoscale imaging of magnetic fields in functional materials" (InCoMa) project from the Research Council of Norway.











***Images of** dispersoid in Al 6xxx alloy by D. Chatterjee; Ferroelectric domains of $ErMnO_3$ by K. A. Hunnestad; Al-Zn-Mg alloy, by E. Thronsen; STEM image of computer CPU samples by E. Christiansen; Mica from 60 km south of Selbu (and corresponding DP), by T. von Helvoort; Al needle on W Omniprobe, by A. Celotto.*

HyperSpy
multi-dimensional data analysis

As the STEM-DPC works almost identically on both magnetic and electric fields, this new capability will make it possible to image electric fields in ferroelectric materials and potentially electric devices such as solar cells.

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 SFI Innovation for a Competitive and Sustainable process Industry (iCSI), headed by professor Hilde Johnsen Venvik, will in 2021 hire Tina Bergh as a postdoc to 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 data” processing, machine learning, open source development and increasingly automated data handling are some of the current “hot topics”. In 2020 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. In 2019 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 2020 we invested in two powerful local workstations for all TEM users to be able to analyze the large data volumes produced. In September 2020 Norway’s first direct detection TEM detector (DED) was mounted. As this detector can easily generate giga- or terabyte size data during one TEM session, file conversion, data transfer and storage have quickly become a bottleneck. 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. This is also in line with the open science policy pushed by EU and other authorities. 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. After Magnus Nord started in the group in 2020 these topics will be further developed and used. An example of this is the merger of pixStem (pixstem.org) with pyXem in 2020. This has created a more robust software project for working on scanning electron diffraction data and has enabled “lazy” versions of several processing categories. The “lazy” processing allows for very big datasets to be processed without being loaded into memory at the same time, making it possible to analyze the very large datasets generated by the DED detector on desktop or laptop computers. 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

The TEM Gemini Centre has contributed to many high school visits and Researchers Night. However, this year, because of the pandemic, there has been very little activity on this arena. We hope to contribute more again in the years to come!

ACTIVE PROJECTS IN 2020

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 about 100 different projects using the facilities in 2020.

Project type	Project title	Involved with TEM	Duration
SFI	SFI PhysMet (Centre for Sustainable and Competitive Metallurgical and Manufacturing Industry)	~3 PhDs NTNU, SINTEF	2020-2028
Partners: NTNU, SINTEF, Statens vegvesen, Forsvarsbygg, Hydro, Elkem, Neuman Aluminium, Equinor, Benteler, ThermoCalc Software.			
SFI	SFI CASA Centre for Advanced Structural Analysis	1-2 PhDs NTNU, SINTEF	2015-23
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, Renault			
SFI	SFI Manufacturing	1 PhD NTNU, 1 postdoc NTNU, SINTEF	2015-23
Partners: SINTEF, NTNU, Benteler, Brødrene AA, Ekornes, GKN Aerospace, Hexagon composites, Kongsberg Automotive, Nammo, Raufoss Neuman, Plastal, Plasto, Rolls Royce, Teeness, Hybond, Hydro			
SFI	SFI iCSI - Industrial Catalysis Science and Innovation for a Competitive and Sustainable process Industry.	SINTEF	2015-23
Partners: Yara Norge, K.A. Rasmussen, Dynea INOVYN Norge, Haldor Topsøe AS			
FME	SuSolTech – The Research Center for Sustainable Solar Cell Technology	1 PhD, NTNU, SINTEF, UiO	2017-25
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	2015-23
Partners: 7 research institutions including both SINTEF and NTNU, 7 public bodies, 26 industrial partners			
KPN/BIA	AMPERE - Aluminium alloys with mechanical properties and electrical conductivity at elevated temperatures	1-2 PhDs NTNU, SINTEF	2015-20
Partners: NTNU, SINTEF, Hydro, Nexans, Raufoss Neuman, SAPA, Gränges			
KPN/BIA	FICAL - Fundamentals of Intergranular Corrosion in Aluminum Alloys	1 PhD NTNU, SINTEF	2015-20
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, 1 Postdoc, SINTEF	2019-24
Partners: NTNU, SINTEF, Hydro, Benteler, Neuman			
Nano2021	In-Sane - In-situ studies of highly conductive bonded interfaces between aluminium and copper at the nanoscale	2 PhD NTNU, SINTEF	2020-24
FRINATEK	Oxide Intermediate Band Photovoltaics (Ox-IB)	1 PhD NTNU, SINTEF	2015-20
IPN/ENERGIX	Nanowire UV LEDs on graphene	SINTEF	2019-21
Partners: CrayoNano, SINTEF, NTNU			
IPN/ENERGIX	Silicon-based anodes towards market penetration – SiBanode	SINTEF	2019-21
Partners: Elkem Technology AS, IFE, SINTEF			
Nano2021	GRANASOL - Low Cost, Ultra-High Efficiency Graphene Nanowire Solar Cells	1 PhD 2014-2019	2014-19
Partners: NTNU, Sejong University, Aalto University, CRAYONANO AS			
NTNU Digital transformation	Rational Alloy Design – ALLDESIGN	1 PhD, NTNU	2018-22
Partners: 4 departments at NTNU; Physics, Materials Science and Engineering, Mechanical Engineering, Mechanical and Industrial Engineering			
FRIPRO	FractAl- Microstructure-based modelling of ductile fracture in aluminium alloys	SINTEF	2017-22
INTPART	Norwegian-Japanese Aluminium alloy Research and Education Collaboration (NJALC) – II.	NTNU, SINTEF, Travel, exchange students	2019-2023
Partners: NTNU, SINTEF, Hydro, University of Toyama, Tokyo Institute of Technology			
EU	ESTEEM3 - https://www.esteem3.eu/	NTNU	2019-24
EU	eForFuel	SINTEF	2018-21
IPN/PETROMAKS2	AMRREX	SINTEF	2018-20
IPN/BIA	HIPTEC	SINTEF	2018-20
NFR/Nano2021	ENCASE	SINTEF	2018-20
NFR/FRIPRO	HEATER	SINTEF	2018-20
NFR/ENERGIX	ANSWER	SINTEF	2018-20
SINTEF/SEP	Conductive cement transducer materials for detecting well plug failures (FARAWELL)	SINTEF	2019-20
IPN	Novel Failure Monitoring System for Marine Applications by including Acoustic Emission (AEMON)	SINTEF	2019-22
EU/H2020	SAFE-N-MEDTECH	SINTEF	2019-23

INTERNATIONAL COLLABORATION

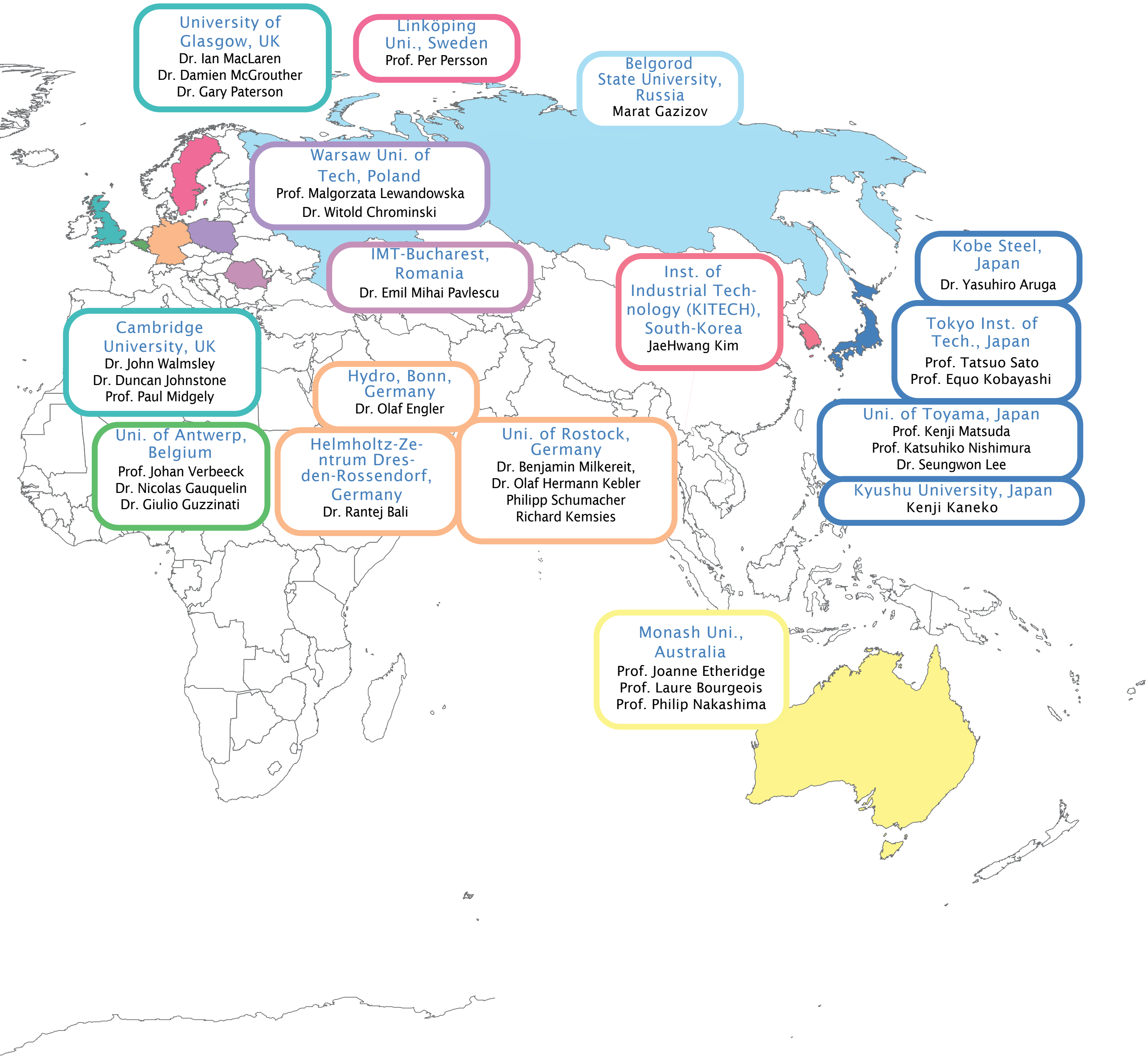
As can be seen from the map and the publication list, the TEM Gemini Centre has productive relations to many research institutions and researchers across the world. Some are long term collaborators; others are new initiatives. However, because of the corona pandemic, there have been very few visits in 2020. Within ESTEEM3 we have in 2020 been granted several transnational (TA) projects and collaborated with renowned researchers across Europe. The map illustrates the direct scientific collaborations. In addition to the ESTEEM3 project, the INTPART project ensures international collaboration, in this case with Japan. We thank all our international collaborators for the productive and stimulating digital interaction and hope we can be able to continue the cooperation and meet physically in the coming years!

NORDIC HUB (NORDTEMHUB)

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) were in. The proposal was funded in the spring of 2020, and is slowly now starting to act, but so far it has been strongly affected by COVID-19. The support to NordTEMhub is mainly for exchange, networking and workshops, which are difficult to do during a pandemic, but we start with some online workshops, and will hopefully be able to utilize the Nordic network more in the years to come.

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 2020 we have had several ongoing projects, initiated from Germany, UK, Spain and Netherlands. 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.



INTERNATIONAL VISITS IN 2020*

- Randi Holmestad, University of Illinois, Urbana-Champaign, US, 6-13. Febr.
- Endre Jacobsen, UACJ &Tokyo Tech, Japan, 28. Febr.- 13. April
- Sivert J.V. Dagenborg, Toyo Aluminium & Tokyo Tech, 17. Jan.- 13.April
- Daehan Kim (Tokyo Institute of Technology, Japan) 1. Jan.-15. March

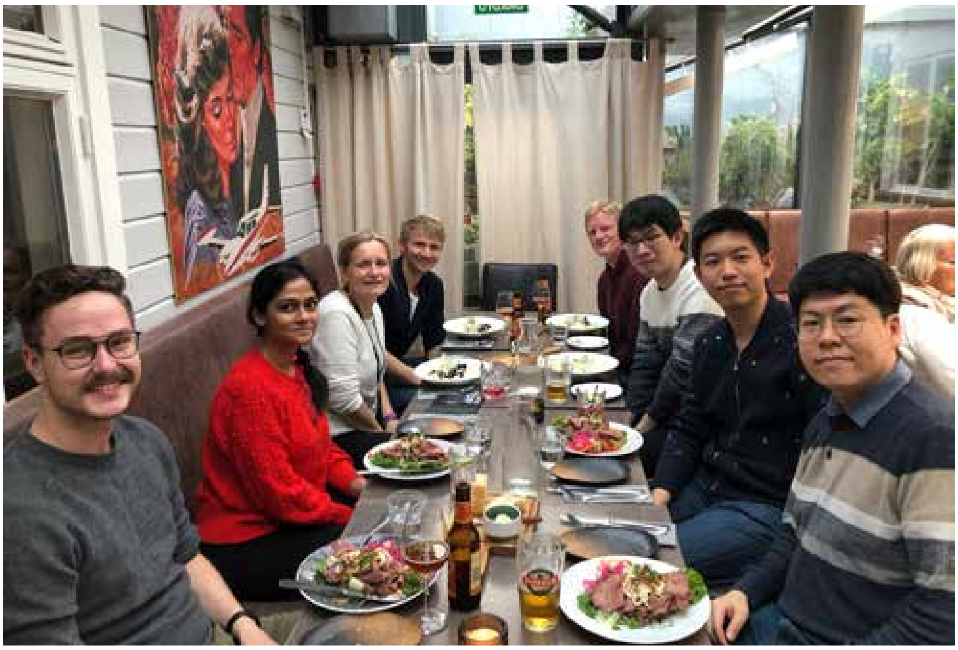
*Due to the pandemic we had a low number of international visits in 2020, but we have managed to maintain most of our international network, as seen from the map which shows international collaborators - we hope for more travel and exchange in the years to come!

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 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 prioritized activity. During 2020 there has been a low activity in the INTPART project due to the pandemic. We had one visitor from Tokyo Tech to NTNU (Daehan Kim), who continued his stay from 2019, and we had two Norwegian Master students on internships in Japanese aluminium industry, to UACJ and Toyo Aluminium, respectively before the lockdown in March. The planned trip of 12 people from Trondheim to Japan in May was unfortunately cancelled. In October, we participated in the CAMRIC conference in Toyama by Zoom. Knut and Randi also gave lecture online for Japanese students. The project has been extended for 6 months but will probably be for longer. We hope for physical meetings in 2021!

From the digital CAMRIC6 conference organized from Toyama, October 2020. Dr. Calin Marioara (SINTEF), Prof. Knut Marthinsen (NTNU), Dr. Takeshi Saito (Hydro) and Prof. Randi Holmestad (NTNU) can be seen among the participants.

The three visiting students from Tokyo Institute of Technology, September 2019. From Left: Postdoc Emil Christiansen (NTNU), Postdoc Dipanwita Chatterjee (NTNU), Prof. Randi Holmestad (NTNU), Dr. Jonas K. Sunde (NTNU), Dr. Sigurd Wenner (SINTEF), Yamato Takeuchi (Tokyo Tech), Daehan Kim (Tokyo Tech), and Xuanling Chen (Tokyo Tech).



PHD DEFENSES

IN THE TEM GEMINI CENTRE 2020

JULIE STENE NILSEN, FEBRUARY 27.

The aim of Julie Stene Nilsen's PhD thesis was to improve structural and compositional characterization of nanowires. By this, the origin of their optical and electronic properties can be better understood, and the synthesis of nanowires and nanowire-based devices further developed. Several different material systems, mostly based on ternary III-V nanowires, were studied. This include GaAs/AlGaAs core-shell nanowires, GaAsSb superlattice structures, InGaN quantum wells in GaN nanowires and Pd/Ge/Au contacts to n-GaAs nanowires. More accurately determination of the composition for different components in the material systems was achieved using scanning transmission electron microscopy. To achieve this, non-standard analysis methods for X-ray energy dispersive spectroscopy were applied and new workflows were developed. In addition, structural characterization, including scanning precession electron diffraction, was used to complement and confirm the compositional analysis and structural details. Julies supervisors were Ton van Helvoort (main) and Bjørn-Ove Fimland.

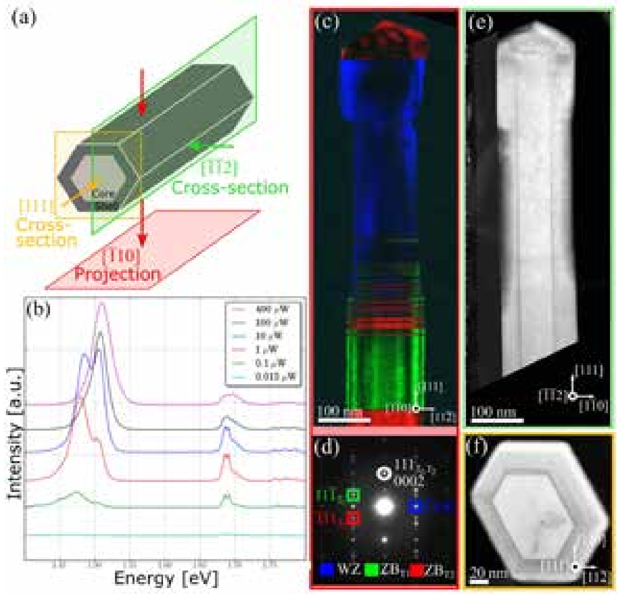


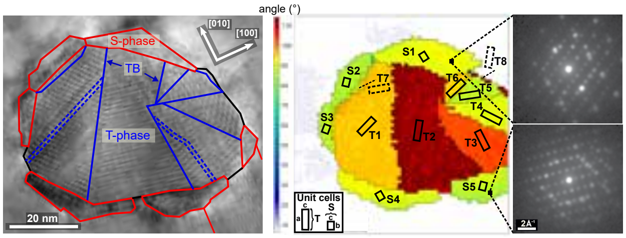
Image on the top righ: *Julie during her defense, opponents Prof. A. Sanchez (University of Warwick, UK) and Dr. K. MacArthur (P. Grünberg Institute, Germany) and Assoc., Prof. C. Bråune (Committee administrator, NTNU);*
The Images to the right: *Correlated PL-TEM study of a randomly grown GaAs/AlGaAs core-shell nanowire. (a) Color-coded schematic of the dierent viewing directions of the nanowire in TEM. (b) Power-dependent PL spectra at 12 K. (c) RGB-colored, stacked DFs of the three dierent phases, as color-coded in the diraction pattern (d). (e)-(f) HAADF-STEM of the two FIB cross-sections in the 112 and 111-directions, respectively.*

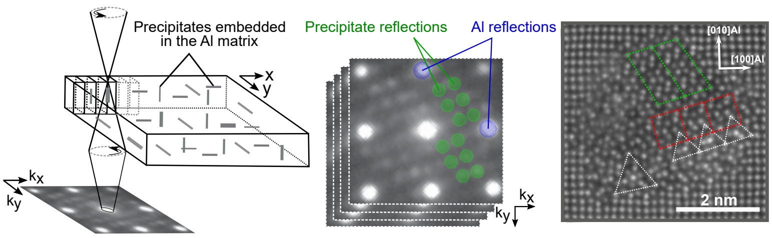
JONAS KRISTOFFER SUNDE, AUGUST 21.

The title of Jonas K. Sundes PhD thesis is "The Effect of Elevated Temperatures on Precipitation in Aluminium Alloys – An Advanced Transmission Electron Microscopy Study". He did a great job in developing the scanning precession electron diffraction (SPED) technique. In particular, he showed how SPED combined with advanced data analysis can be an important tool in future alloy design, by obtaining improved precipitate statistics from larger regions of the sample in a more objective manner than by using conventional approaches. Furthermore, it was found that low additions of Cu (0.01 atomic %), being a common alloying element in these alloys, may change the Al-Mg-Si system precipitation, particularly after long temperature exposure. This has important implications for alloy recycling, where inclusions of trace elements are

practically unavoidable. In addition, direct observations of precipitate transformations during thermal ageing have been achieved using in situ heating TEM. In the study of an Al-Mg-Si-Cu alloy, it was shown that the fragmented L phase exhibits a significantly improved thermal stability as compared to the main age-hardening precipitate phase β. Jonas was funded from the AMPERE project and started as a scientist in NORCE in Stavanger after moving from Trondheim. Supervisors for Jonas were Randi Holmestad (main) and Calin D. Marioara.

Image on bottom previous page: Dr. J. K. Sunde (NTNU). *Image above*: J. K. Sunde, R. Holmestad (NTNU), opponents Prof. H. Fraser (Ohio State University, USA) and Dr. D. Alexander (EPFL, Switzerland), and Prof. J. He (Committee administrator, NTNU).



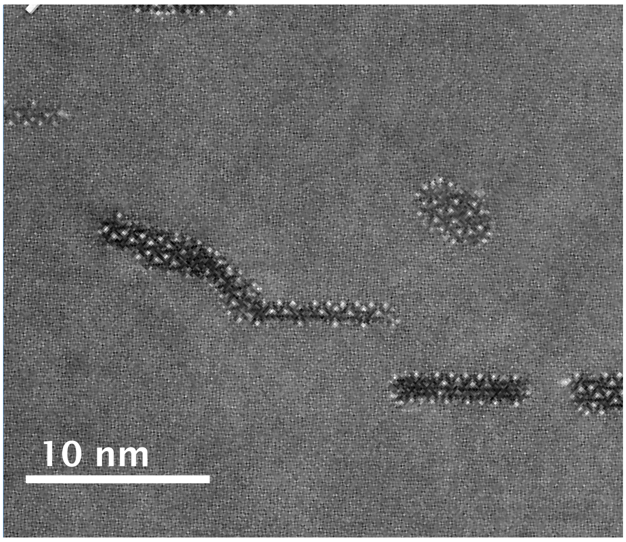


Left top figure: *A HRTEM image of a T-/S-phase aggregate next to orientation mapped SPED data of the same aggregate structure;*
Left figure*: Schematic of the SPED scan data recording and a HAADF STEM lattice image of a hybrid precipitate cross-section.*

ADRIAN LERVIK, SEPTEMBER 25.

Adrian Lervik worked on the FICAL project where the main goal has been to get a more fundamental understanding of corrosion effects in aluminium alloys, in particular intergranular corrosion. To understand the corrosion properties better requires detailed studies of the microstructure at the nanoscale. The main findings involve new understandings concerning the precipitation behavior in both 7xxx and 6xxx alloys. It is shown that corrosion can be reduced by altering the processing parameters, and that the fundamental reason for this is closely related to the precipitates. Methodologies for improved investigation of grain boundaries have been presented and are applicable to other material systems. In addition, a nearly 100-year-old riddle concerning the atomic structure of clusters forming in room temperature has been solved. The combined findings will be important in producing Al alloys with excellent mechanical properties and corrosion resistance. Adrian started in Norwegian Defence Research Establishment (FFI) as a research scientist. Adrians supervisors were Randi Holmestad (main) and John Walmsley.

Left to right*: R. Holmestad (NTNU), opponent V. F. Hansen (Univ. of Stavanger, Norway), Dr. Adrian Lervik (NTNU) and Prof. A. Erbe (Committee administrator, NTNU).* ***On the screen****: opponent Prof. B.D. Zander (RWTH Aachen University, Germany), and C. Marioara (NTNU).*



Left to right: *HAADF STEM image of precipitates formed on dislocations in an Al-Mg-Si-Cu alloy, and A. Lervik during his defense.*

ALEKSANDER BUSETH MOSBERG, OCTOBER 2.

Aleksander Buseth Mosberg's thesis represents the application and further developments of focused ion beam (FIB) to two promising functional material systems for next-generation devices and demonstrates some of the large untapped potential that can be accessed. For III-V semiconductor nanowires, the FIB has been used to pattern the growth mask defining nanowire position-controlled growth, shortening and simplifying the mask fabrication process. Techniques such as computer vision-based image analysis for scanning electron microscopy and efficient in-situ electrical probing have been developed. Ferroelectric domain walls in hexagonal manganites are sub-nanometer features that can exhibit varying degrees of conductivity, making them interesting as future circuit elements for nanoelectronics. In this material local field-induced conductivity enhancement has been characterized in 3D through use of FIB specimen preparation. Furthermore, a workflow has been developed to prepare arbitrary domain wall specimens with sufficient surface quality for advanced scanning probe microscopy characterization while preserving the original domain structure. Aleksander's work demonstrates the potential of FIB and its role in advanced materials characterization. Supervisors for Aleksander were Ton van Helvoort (main) and Per Erik Vullum.

Dr. A. B. Mosberg (in the middle), Antonius T. J. van Helvoort (NTNU, to the left) and Prof. J. Glaum (Committee administrator, NTNU). ***On the screen:*** *opponents Dr. N. Bassim (McMaster University, Canada) and Dr. D.Cooper (CEA/LETI, Grenoble, France).*

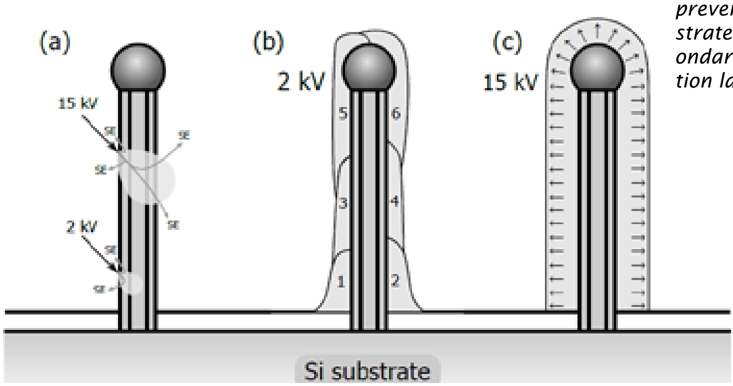
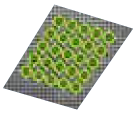


Figure*: Protection deposition for nanowires. (a) Schematic of relative interaction volumes for 15 kV and 2 kV electron beams. (b) Schematic of common deposition scheme, with the nanowire protected by building up multiple to prevent bending when depositing at 2 kV. (c) Alternative strategy, depositing at 15 kV to more uniformly emit secondary electrons, resulting in a radially uniform protection layer and less strain-induced bending.*

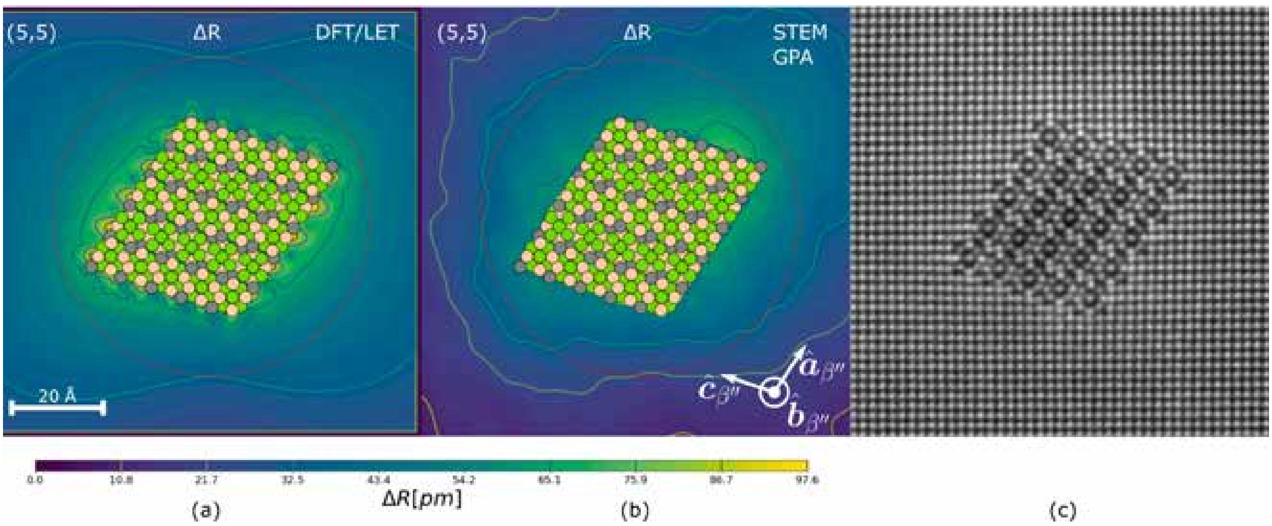
JONAS FRAFJORD, DECEMBER 18.



Jonas Frafjord wrote a thesis on "Atomistic Scale Modelling of Defects in Aluminium Alloys" and was working in SFI CASA. In his work, the focus has been using density functional theory (DFT) on the various defects that help to control the properties of aluminum, especially aimed at strength. The understanding of the movements of dislocations, solute atoms and the interaction of these defects help to bring the industry closer to the goal of digitally constructing tailor-made aluminum alloys, adapted to specific purposes. Impurities are very important for the properties and are fundamental for achieving mechanical strength in heat-treated alloys. Also, a study on strain in the aluminium lattice surrounding precipitates has been done as a part of his thesis. Jonas continues as a post-doc in the SumAl project. Supervisors for Jonas were Randi Holmestad, Jesper Friis and Inga G. Ringdalen.

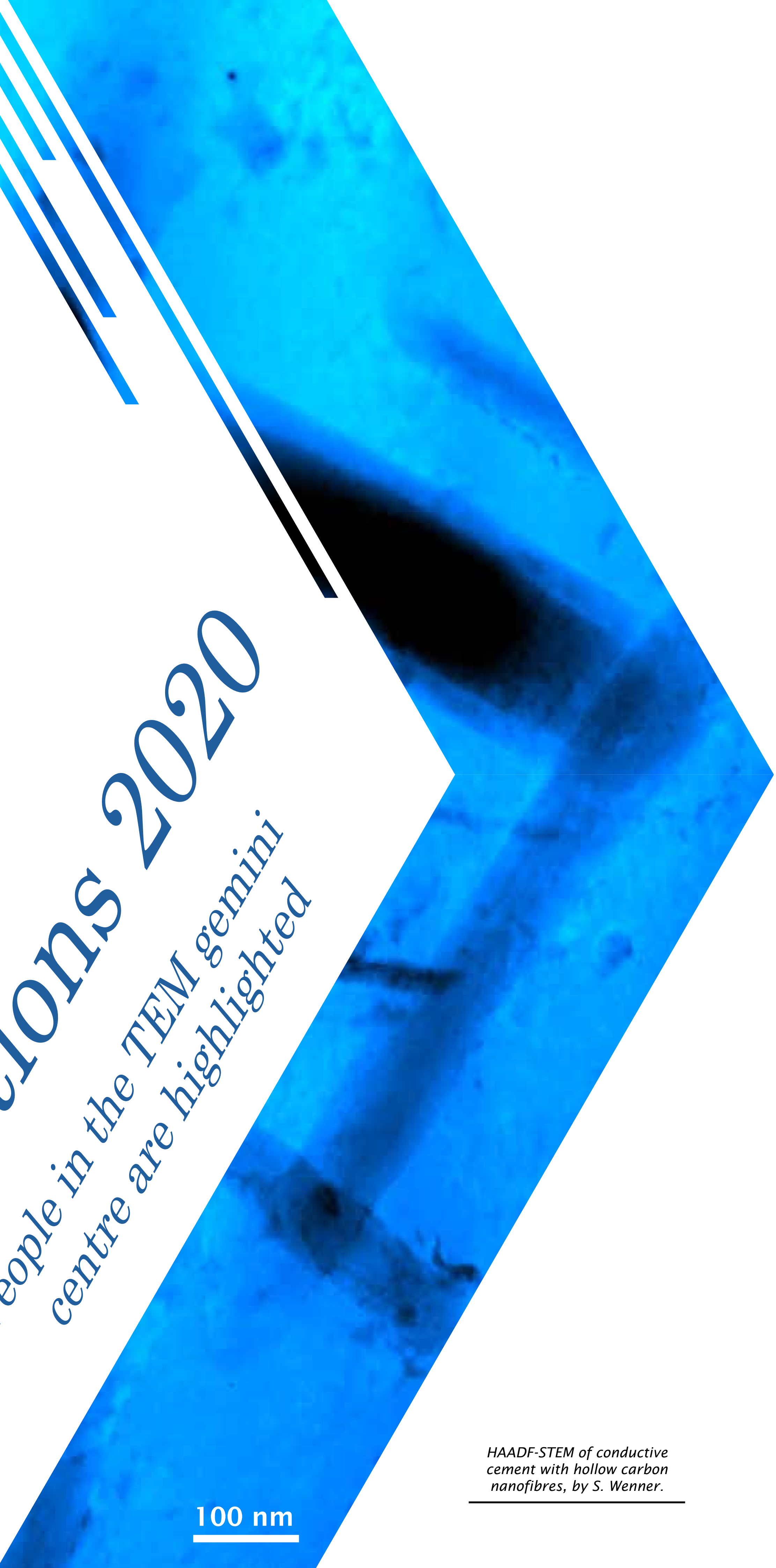
Figure on top: *Atomic positions added onto a STEM image (by S. Wenner) to illustrate the movement of the atoms within each eye. Insertion shows the atomic positions of a β'' eye based on the aluminium lattice vectors;*
Image above: *Jonas Frafjord (NTNU);*
Image to the right: *J. Frafjord and opponents Prof. Erik Bitzek (Friedrich-Alexander-Universität, Germany) and Assoc. Prof. Lucile Dezerald (Université de Lorraine, France) during the defense.*

Displacement field of the aluminium matrix outside the embedded β''. Displacement is calculated from the change in atomic positions after DFT relaxation, GPA analysis is used to find the displacement field in the STEM image.



Publications 2020

People in the TEM gemini centre are highlighted



HAADF-STEM of conductive cement with hollow carbon nanofibres, by S. Wenner.

TEM GEMINI CENTRE PUBLICATIONS 2020

*Published online, in journals for 2021.
** Not including TEM, but with people from TEM Gemini Centre

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When the weather allows, skiing is a decent way to wrap up a day of work :)
(left to right) T. Bergh, E. Christiansen, C. Hell, E. Thronsen and J. Frafjord.

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CONFERENCE CONTRIBUTIONS (A SELECTION)

Chen, X; **Mørtsell**, Eva Anne; **Sunde**, Jonas Kristoffer; **Marioara**, Calin Daniel; **Holmestad**, Randi; Kobayashi, Equo. Effect of clustering and dislocation loop formation during cyclic deformation on mechanical properties of 6082 extruded aluminium alloy. Annual meeting in Japanese Institute of Light Metals; 2020-05-05 - 2920-05-07

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Holmestad, Randi, Crash course in TEM -(with examples from microstructure and precipitates in Al alloys). Global Engineering Lectures, Toyama University; 2020-10-06 - 2020-10-06

Holmestad, Randi, SPED and HAADF- STEM used to aid aluminium alloy developments. 26th Australian Conference on Microscopy and Microanalysis (ACMM); 2020-02-16 - 2020-02-20

Holmestad, Randi, TEM used to study microstructure and precipitates for understanding 6xxx alloys. Global Engineering Lectures; 2020-10-06 - 2020-10-06

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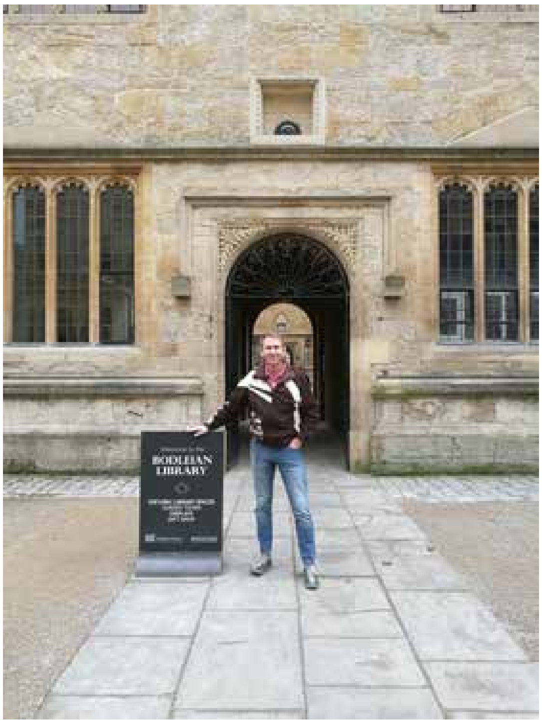

P. E. Vullum sightseeing in Oxford during microscope downtime.

structure of solute clusters in Al-Zn-Mg alloys. Nasjonal konferanse for materialteknologi; 2020-12-02 - 2020-12-03

Tvedt, Haakon; **Marioara**, Calin Daniel; **Andersen**, Sigmund Jarle; **Holmestad**, Randi; **Friis**, Jesper. AutomAl 6000: Automatic structural labeling of STEM images of hybrid precipitates in 6xxx alloys. 17^{th} International conference of aluminium alloys (ICAA17); 2020-10-26 - 2020-10-29

Sunde, Jonas Kristoffer; **Wenner**, Sigurd; **Holmestad**, Randi. Tracking the growth and transformation of Al-Mg-Si-Cu precipitates by in-situ heating TEM. 6^{th} Forum of Center for Advanced Materials Research and International Collaboration (CAMRIC6); 2020-09-28 - 2020-10-08

Thronsen, Elisabeth; **Marioara**, Calin Daniel; **Andersen**, Sigmund Jarle; **Friis**, Jesper; Matsuda, Kenji; **Holmestad**, Randi. Precipitates in a heavy pre-deformed Al-1.3Cu-1.0Mg-0.4Si wt.% alloy. 17^{th} International Conference on Aluminium Alloys 2020 (ICAA17); 2020-10-26 - 2020-12-29

Ånes, Håkon Wiik; **van Helvoort**, Antonius T. J.; **Marthinsen**, Knut. Orientation dependent precipitation in an AlMn alloy. Nasjonal konferanse for Materialteknologi; 2020-12-02 - 2020-12-03

POPULAR SCIENCE

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MASTER'S THESES

- Ragna Bakke, Transmission electron microscopy based characterization of CdTe–HgTe core–shell semiconductor nanowires (Supervisor Sigurd Wenner)
- Sigrid Wanvik Haugen, Structural and Optical Characterization of AlGaN Nanostructures for UV-LEDs by Correlated Electron Microscopy (Supervisor Ton van Helvoort)
- Endre Jacobsen, Scanning Precession Electron Diffraction Template Matching for Automated Phase Mapping of Precipitates in 6xxx Aluminium Alloys (Supervisor Ton van Helvoort)
- Hanne Mørkeseth, The effect of Fe and Mn on precipitation in an Al-Cu-Mg-Si alloy (Supervisor Randi Holmestad)
- Eirik Opheim, Evaluating template matching for orientation analysis based on electron diffraction (Supervisor Ton van Helvoort)
- Øystein Rolstad, Orientation effects on energy dispersive spectroscopy in TEM (Supervisor Ton van Helvoort)
- Tor Inge Thorsen, Heterostructured GaAs/GaAsSb nanowires characterized by scanning precession electron microscopy (Supervisor Ton van Helvoort)
- Hursanay Turgun, Electron Microscopy Characterization of Aluminium-Copper-Titanium-Steel Joint made using the Hybrid Metal Extrusion & Bonding Method (Supervisor Per Erik Vullum)
- Haakon Tvedt, AutomAl 6000: Semi-automatic structural labeling of HAADF-STEM images of precipitates in Al-Mg-Si-(Cu) alloys (Supervisor Randi Holmestad)

PROJECT THESES

- Sivert J.V. Dagenborg, The effects of precursors, doping, and undercoat on the coating and phases of TiO_2 on Al-flakes (Supervisor Randi Holmestad)
- Oskar Ryggetangen, Characterization of $K_3Nb_3B_2O_{12}$ by transmission electron microscopy (Supervisor Ton van Helvoort)
- Idun Bækken, Correlated SEM – CL – TEM of AlGaN nanowire based LEDs (Supervisor Ton van Helvoort)
- **Knut Håvard Raen, Correlated SEM-electrical characterization of GaN/AlGaN nanowire based LEDs (Supervisor Ton Van Helvoort)
- Ingeborg Nævra Prestholdt, Semiconductor nanowire characterization based on scanning electron diffraction (Supervisor Ton van Helvoort)

The Nature Materials publication on conductivity control in functional oxides described on page 36 shows an excellent example of multidisciplinary research collaboration across different departments at NTNU, where TEM has had a central role and combined with other techniques and simulations. On the picture we see two of the group leaders at the ARM200. Jan Torgersen (Department of Mechanical and Industrial Engineering) at the left, and Dennis Meier (Department of Materials Science and Engineering) have a starting and consolidator ERC grant respectively and ongoing research projects that involve TEM. (Photo: Geir Mogen)
(see https://gemini.no/2020/09/stort-fremskritt-for-fremtidens-elektroniske-dingser/)

Selected scientific papers

HAADF STEM of Q' on alpha dispersoid in Al 6xxx alloy. By D. Chatterjee.

nature materials

Conductivity control via minimally invasive anti-Frenkel defects in a functional oxide

Donald M. Evans[1,10], Theodor S. Holstad[1,10], Aleksander B. Mosberg[2,10], Didrik R. Småbråten[1,10], Per Erik Vullum[3], Anup L. Dadlani[4], Konstantin Shapovalov[5], Zewu Yan[6,7], Edith Bourret[7], David Gao[2,8], Jaakko Akola[2,9], Jan Torgersen[4], Antonius T. J. van Helvoort[2], Sverre M. Selbach[1] and Dennis Meier[1]

Utilizing quantum effects in complex oxides, such as magnetism, multiferroicity and superconductivity, requires atomic-level control of the material's structure and composition. In contrast, the continuous conductivity changes that enable artificial oxide-based synapses and multiconfigurational devices are driven by redox reactions and domain reconfigurations, which entail long-range ionic migration and changes in stoichiometry or structure. Although both concepts hold great technological potential, combined applications seem difficult due to the mutually exclusive requirements. Here we demonstrate a route to overcome this limitation by controlling the conductivity in the functional oxide hexagonal Er(Mn,Ti)O3 by using conductive atomic force microscopy to generate electric-field induced anti-Frenkel defects, that is, charge-neutral interstitial–vacancy pairs. These defects are generated with nanoscale spatial precision to locally enhance the electronic hopping conductivity by orders of magnitude without disturbing the ferroelectric order. We explain the non-volatile effects using density functional theory and discuss its universality, suggesting an alternative dimension to functional oxides and the development of multifunctional devices for next-generation nanotechnology.

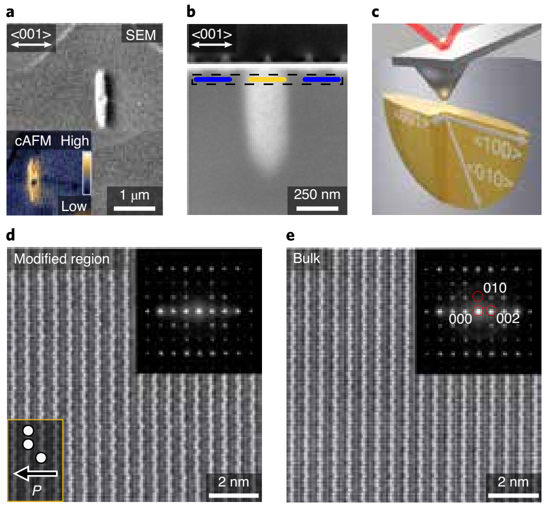


Fig. 2 | Morphology and structure of electric-field-induced conducting features. a, Top-down SEM and cAFM (inset, Uread = +45 V) images of an elliptically shaped conducting region (bright) generated by applying Uwrite = −60 V for 5 s with the probe tip kept stationary. The white arrow indicates the ferroelectric axis (P ∥ <001>). **b**, SEM image of a FIB cross-section from **a** that reveals how the conducting feature protrudes into the bulk of h-Er(Mn,Ti)O3 and shows where, in the final transmission electron microscopy (TEM) lamella, the EELS line scan for Fig. 3 was taken (blue and yellow represent the bulk and electrically modified region, respectively). **c**, 3D sketch representing the cAFM-induced conducting feature and the shape dependence on the crystallographic axes based on the cross-section in **b** and cross-sections of equivalent dots taken in perpendicular directions. **d**, HAADF-STEM image from the conductive region seen in **b**, viewed along the P63cm [100 I] zone axis. The brighter Er atomic positions show the characteristic up–up–down displacement as discussed in Holtz et al.29 and the arrow in the bottom inset shows how this links to the ferroelectric polarization P. Top inset: corresponding selected area electron diffraction pattern. **e**, HAADF-STEM image taken in the unmodified bulk region viewed as for **d**. Inset: corresponding selected area electron diffraction pattern. The analysis of the crystal lattice in **d**, **e** and Supplementary Fig. 10 reveals no measurable differences, which reflects that the atomic-scale structure and, hence, the ferroelectric polarization, are unaffected by the electric-field-induced change in conductivity.

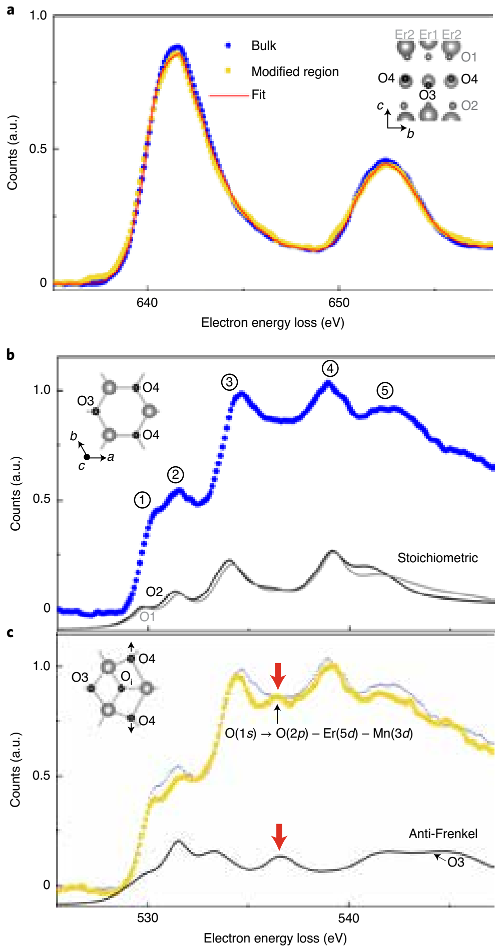


Fig. 3 | Comparison of the electronic structure in as-grown and electrically modified regions. a, Blue and yellow data points represent EELS spectra of the Mn $L_{2,3}$-edge in h-Er(Mn,Ti)O_3 taken in the bulk and in the modified conducting region, respectively (ratio of lamella thickness (t) and mean free path (λ), t/λ = 0.40 ± 0.02). The red line is a fit to the Mn L2,3-edge in the conducting region based on a linear combination of spectra that correspond to Mn^{2+}, Mn^{3+} and Mn^{4+} valence states with 3.75% Mn^{2+} and 3.75% Mn^{4+}, that is, approximately one anti-Frenkel defect in every nine unit cells. Inset: symmetry-inequivalent Er and O positions. **b**, Data points present the O K-edge in the bulk. Grey and black lines are calculated spectra for apical oxygen (O1 and O2), respectively, in a stoichiometric crystal. **c**, Yellow points present the O K-edge in the region with enhanced conductivity (the bulk spectrum (blue) is shown for reference). The black line is the calculated spectrum for planar oxygen (O3) in the presence of anti-Frenkel defects, as sketched in the inset. Transitions are labelled according to the projected DOS (Supplementary Fig. 13b). Red arrows indicate a peak at about 537 eV, which is characteristic for contributions from planar oxygen. All the EELS spectra were taken on the same single line scan with an even sample thickness and are spatially averaged over the regions indicated by the blue and yellow lines in Fig. 2b. a.u., arbitrary units.

Evans, D. M., Holstad, T. S., Mosberg, A. B., Småbråten, D. R., Vullum, P. E., Dadlani, A. L., Shapovalov, K., Yan, Z., Bourret, E., Gao, D., Akola, J., Torgersen, J., van Helvoort, A. T. J., Selbach S. M. & Meier, D. Conductivity control via minimally invasive anti-Frenkel defects in a functional oxide. *Nature Materials.* **19, 1195** (2020). https://doi.org/10.1038/s41563-020-0765-x

The effect of low Cu additions on precipitate crystal structures in overaged Al-Mg-Si(-Cu) alloys

Jonas K. Sunde[a,*], Calin D. Marioara[b], Randi Holmestad[a]

A B S T R A C T

This study concerns the effect of low Cu additions (≲0.1 at.%) on the precipitate crystal structure evolution in three Al-Mg-Si(-Cu) alloys during overageing. The evolution was assessed through a combination of atomic resolution scanning transmission electron microscopy, scanning electron diffraction, and differential scanning calorimetry. It was found that relatively small changes in the Cu level and the Si:Mg ratio had significant effects on the resulting distribution of precipitate phases, their structural evolution, and their thermal stability. Two Si-rich alloys formed hybrid *β′* phase and Q′ phase precipitates on overageing. A third Mg-rich alloy primarily formed L phase precipitates, which exhibited superior thermal stability. Three distinct Cu-containing sub-units that form the basis for all Al-Mg-Si-Cu precipitate phases were identified: the three-fold symmetric Q′/C and the β_{Cu}' sub-units, in addition to a newly discovered C sub-unit. The formation of each sub-unit was discussed, and the atomic structures and connections to other precipitate phases in the Al-Mg-Si(-Cu) system were elaborated. The work presented provides new insights into the complex precipitation of Cu-added Al-Mg-Si alloys, with implications for material properties. The results obtained will be of importance in future alloy and process development, and are thought to be of high value in modelling work on the quaternary Al-Mg-Si-Cu system.

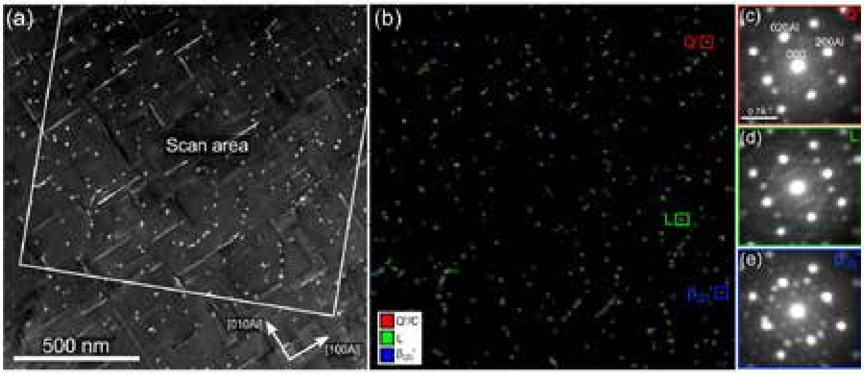


Fig. 5. (a) Dark-field TEM image of alloy M in the 1 month ageing condition. SPED scan area is indicated. The image was acquired near the $[001]_{Al}$ zone axis. (b) Phase map constructed from analysed SPED data showing 437 precipitate cross-sections. Three groups of phases are mapped using RGB colouring: Q′/C (red), L (green), and $\beta_{(2)}'$ (blue). (c–e) Individual pixel PED patterns (raw) from indicated precipitates in (b). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

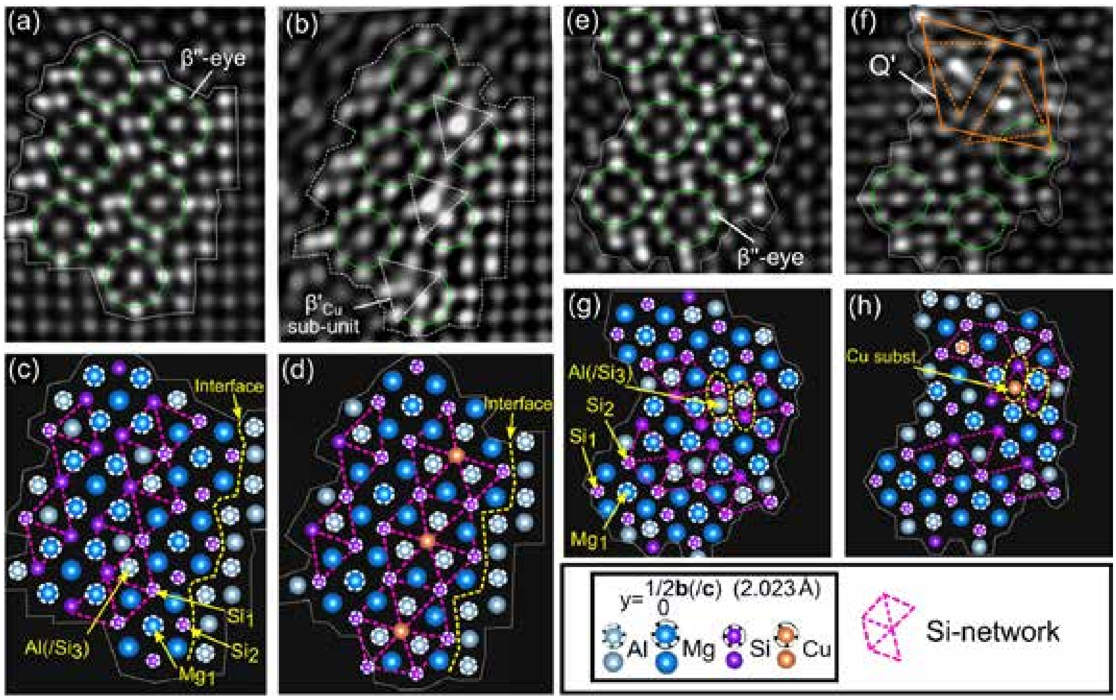


Fig. 8. (a–d) Side-by-side lattice images and atomic overlays of a pure β″ and a hybrid precipitate containing β″ and β_{Cu}' sub-units. (e–h) Side-by-side lattice images and atomic overlays of a pure β″ and a hybrid precipitate containing β″-eyes and a Q′ unit cell. Atomic overlay of the lattice images have been constructed according to the rules of Andersen et al. [17].

Sunde, J. K., Marioara, C. D. & Holmestad, R. The effect of low Cu additions on precipitate crystal structures in overaged Al-Mg-Si(-Cu) alloys. *Materials Characterization*. **160** 110087 (2020). https://doi.org/10.1016/j.matchar.2019.110087

Grain boundary structures and their correlation with intergranular corrosion in an extruded Al-Mg-Si-Cu alloy

A. Lervik [a,*], S. Wenner [b,a], O. Lunder [b], C.D. Marioara [b], R. Holmestad [a]

A B S T R A C T

A detailed analysis of grain boundaries in a highly textured Al-Mg-Si-Cu alloy is presented in this work. Electron backscatter diffraction demonstrates the presence of three main categories of grain boundaries in addition to sub-grain boundaries. These grain boundaries have been systematically analysed using high–resolution scanning transmission electron microscopy. Intergranular corrosion (IGC) susceptibility was statistically correlated with the same defined grain boundaries. A high density of metastable Q′-phase grain boundary particles correlates with a reduction in Cu segregation at grain boundaries and increased IGC resistance. Results herein are relevant in further understanding grain boundary structures in Al-Mg-Si-Cu alloys and their susceptibility to IGC, and can be implemented into modelling frameworks.

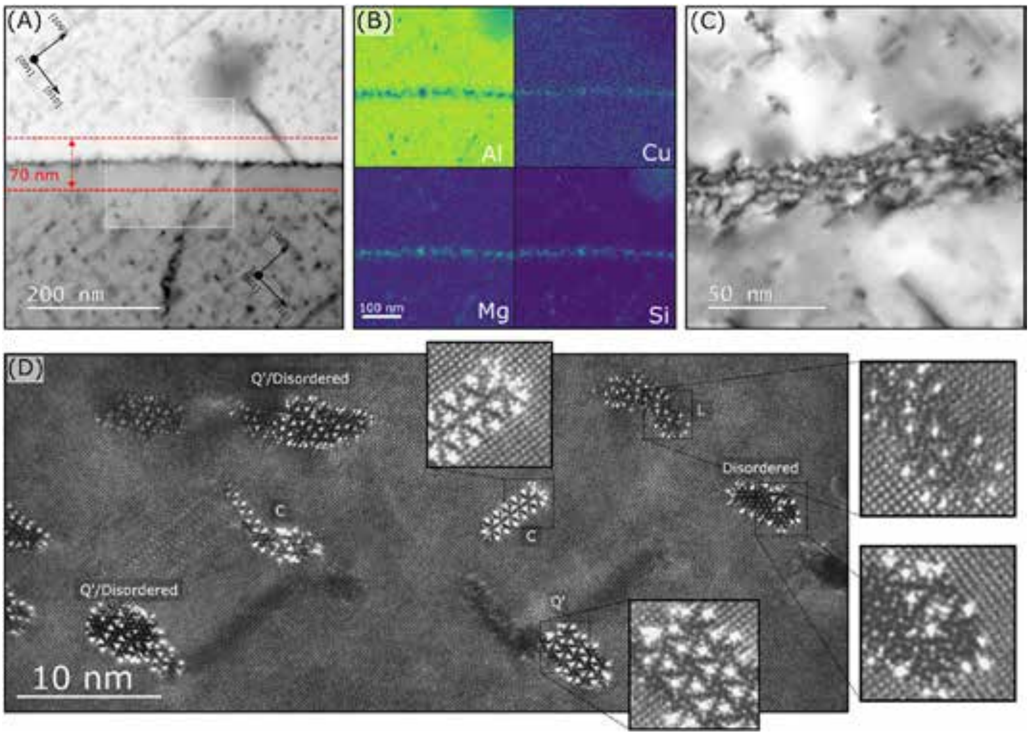


Fig. 5. (A) BF-STEM image of a sub-grain boundary in a [001]Al oriented grain distinguished by an in-plane rotation of <0.5°. (B) Shows the EDS results from the indicated region in (A). (C) BF-TEM image demonstrating that dislocations are associated with the grain boundary. (D) High–resolution HAADF-STEM image of the particles present on the sub-grain boundary.

wo regions on each side of the interface. The schematic cubes indicate the approximate orientation of the grains. (B) Shows the same region, b espect to the electron beam. EDS maps of Al, Cu, Mg and Si are shown in both projections.

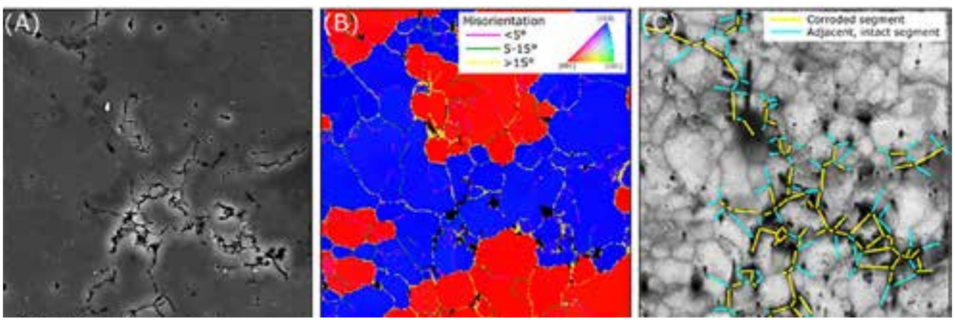
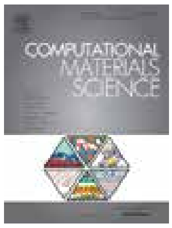


Fig. 7. Methodology for statistically analysing IGC using EBSD. (A) Shows a secondary electron SEM image after 2 h exposure to the IGC test solution. (B) Inverse pole figure map with grain boundaries separated by misorientation angle. (C) EBSD image quality map, overlaid with corroded (yellow) and uncorroded (cyan) grain boundary segments. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Lervik, A., Wenner, S, Lunder, O., Marioara, C. D. & Holmestad, R. *Materials Characterization.* **170** 110695 (2020). https://doi.org/10.1016/j.matchar.2020.110695

Fully resolved strain field of the β'' precipitate calculated by density functional theory

Jonas Frafjord [a,b,*], Stéphane Dumoulin [a,c], Sigurd Wenner [c], Inga G. Ringdalen [a,c], Randi Holmestad [a,b], Jesper Friis [a,c]



A B S T R A C T

The β'' precipitate is the main hardening phase in age hardenable Al-Mg-Si alloys, and it is therefore of major scientific and industrial importance. A full model of the β'' precipitate cross-section embedded in an aluminium host lattice is created for a range of precipitate sizes, and relaxed by first principle calculations. The influence of periodic images is avoided by applying a cluster based model with fixed boundary conditions, where the surface is corrected by a displacement field calculated by linear elasticity theory. The calculated misfit values between the precipitate and the host lattice vectors are consistent with experimental scanning transmission electron microscopy results. The misfit area increases proportionally with the cross sectional area, suggesting that the lattice parameters of β'' do not change as the size increases. Both the displacement field and the strain field are in agreement with experimental results. The strain field calculated by density functional theory shows a local zone close to the precipitate where the chemical contribution to the strain field is dominant. The strong correspondence between the experimental and the modelling results supports the methodology to be used in general to study other phases.

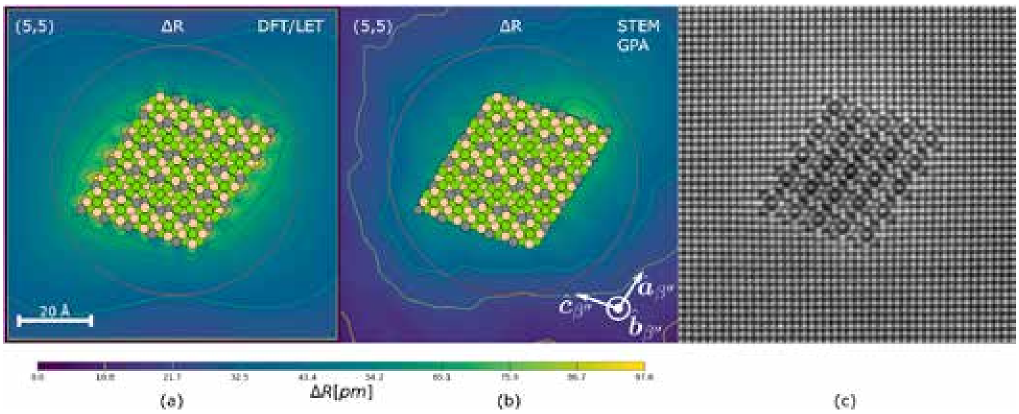


Fig. 5. The radial displacement field, ΔR, for a 5 × 5 β″ precipitate. (a) was calculated from atoms relaxed by DFT, inside the red circle, and atoms relaxed by LET, outside the red circle. (b) is the GPA result of the STEM image in (c). The blue bar is the scale bar, common for all subfigures. The coloured dots represent the precipitate, where the beige, lime and grey are Si, Mg and Al atoms, respectively.

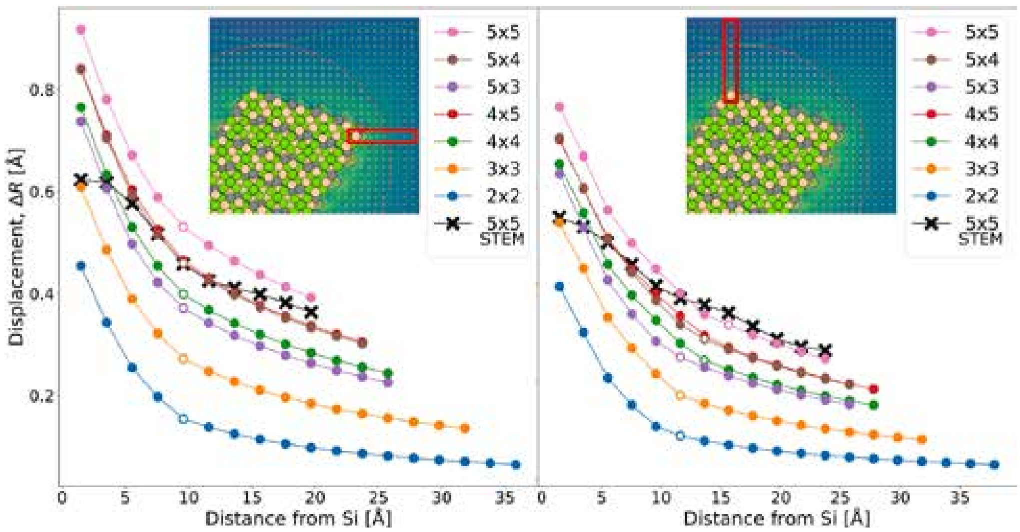


Fig. 6. Linescans of the displacement field for different sized β″ precipitates. The size description follows $N_a \times N_c$, representing the number of β″ eyes. The linescans are taken along the atoms marked by the red box. The coloured dots in the inset figure represent the precipitate, where the beige, lime and grey are Si, Mg and Al atoms, respectively. The open dots in the graph are the first atom outside the red circle, separating the DFT relaxed region, inside, and the LET relaxed region, outside. The black line with cross markers is GPA results from the STEM image shown in Fig. 5(c).

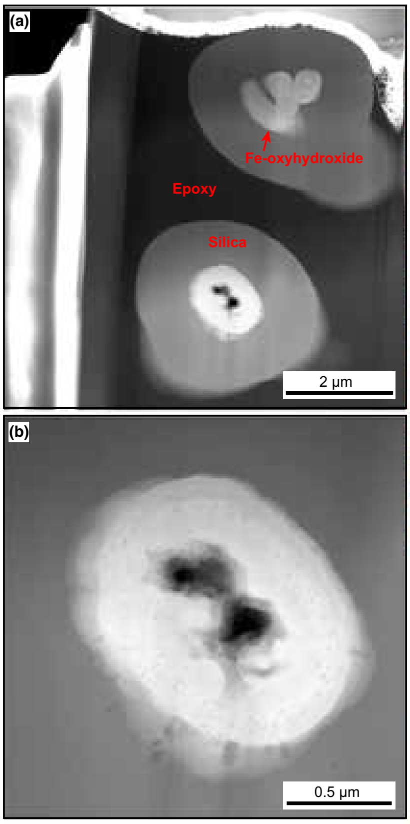
Frafjord, J., Dumoulin, S., Wenner, S., Ringdalen, I. G., Holmestad, R. & Friis, J. Fully resolved strain field of the precipitate calculated by density functional theory. *Computational Materials Science.* **187** 110054 (2021). https://doi.org/10.1016/j.commatsci.2020.110054 geobiology WILEY

On the biogenicity of Fe-oxyhydroxide filaments in silicified low-temperature hydrothermal deposits: Implications for the identification of Fe-oxidizing bacteria in the rock record

Karen C. Johannessen[1] | Nicola McLoughlin[2] | Per Erik Vullum[3,4] | Ingunn H. Thorseth[1]

Abstract

Microaerophilic Fe(II)-oxidizing bacteria produce biomineralized twisted and branched stalks, which are promising biosignatures of microbial Fe oxidation in ancient jaspers and iron formations. Extracellular Fe stalks retain their morphological characteristics under experimentally elevated temperatures, but the extent to which natural post-depositional processes affect fossil integrity remains to be resolved. We examined siliceous Fe deposits from laminated mounds and chimney structures from an extinct part of the Jan Mayen Vent Fields on the Arctic Mid-Ocean Ridge. Our aims were to determine how early seafloor diagenesis affects morphological and chemical signatures of Fe-oxyhydroxide biomineralization and how extracellular stalks differ from abiogenic features. Optical and scanning electron microscopy in combination with focused ion beam-transmission electron microscopy (FIB-TEM) was used to study the filamentous textures and cross sections of individual stalks. Our results revealed directional, dendritic, and radial arrangements of biogenic twisted stalks and randomly organized networks of hollow tubes. Stalks were encrusted by concentric Fe-oxyhydroxide laminae and silica casings. Element maps produced by energy dispersive X-ray spectroscopy (EDS) in TEM showed variations in the content of Si, P, and S within filaments, demonstrating that successive hydrothermal fluid pulses mediate early diagenetic alteration and modify the chemical composition and surface features of stalks through Fe-oxyhydroxide mineralization. The carbon content of the stalks was generally indistinguishable from background levels, suggesting that organic compounds were either scarce initially or lost due to percolating hydrothermal fluids. Dendrites and thicker abiotic filaments from a nearby chimney were composed of nanometer-sized microcrystalline iron particles and silica and showed Fe growth bands indicative of inorganic precipitation. Our study suggests that the identification of fossil stalks and sheaths of Fe-oxidizing bacteria in hydrothermal paleoenvironments may not rely on the detection of organic carbon and demonstrates that abiogenic



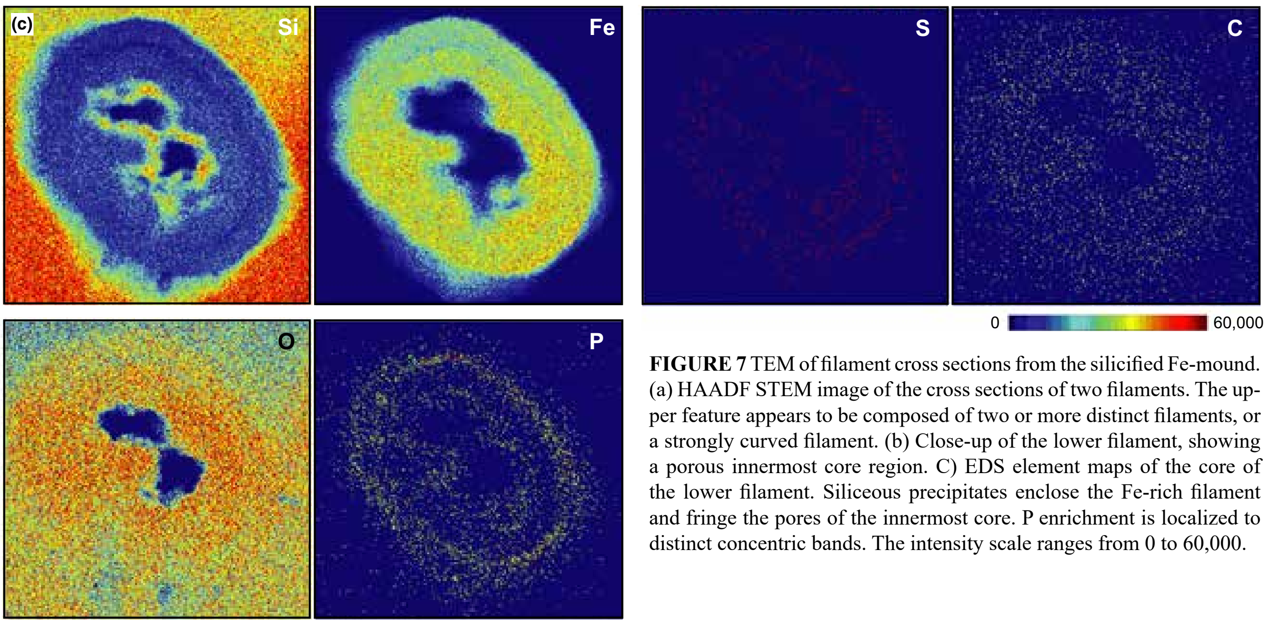
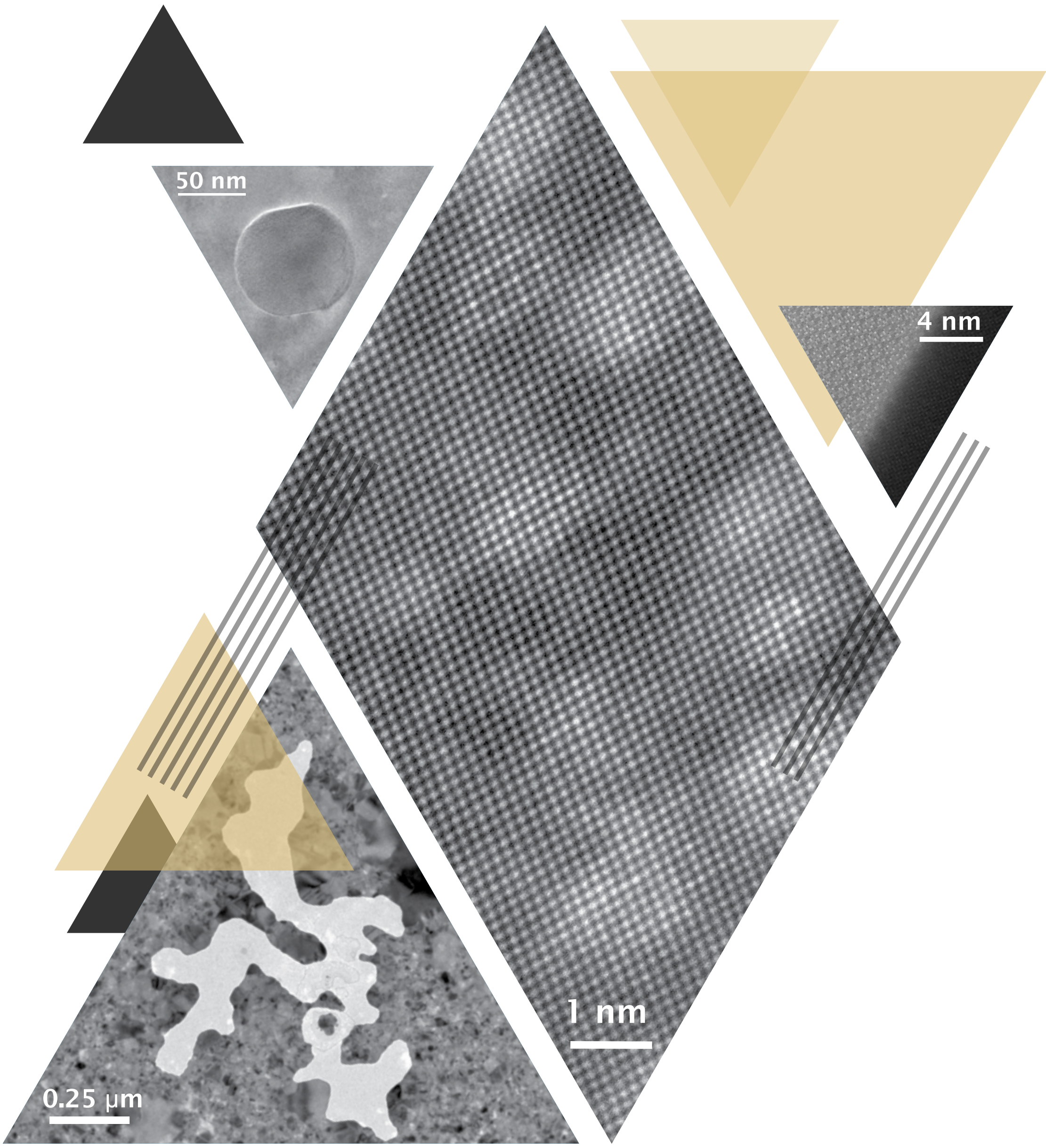
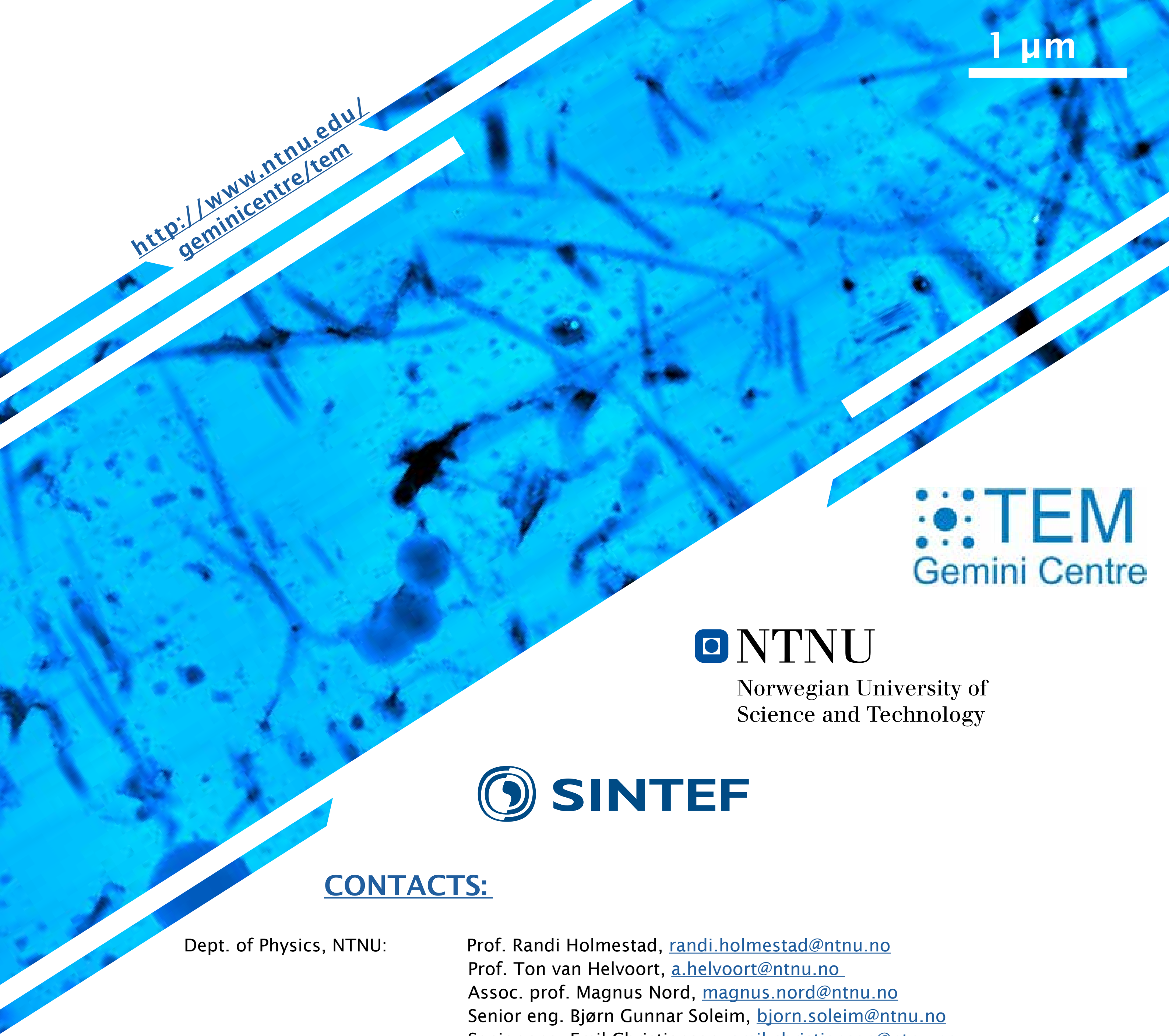


FIGURE 7 TEM of filament cross sections from the silicified Fe-mound. (a) HAADF STEM image of the cross sections of two filaments. The upper feature appears to be composed of two or more distinct filaments, or a strongly curved filament. (b) Close-up of the lower filament, showing a porous innermost core region. C) EDS element maps of the core of the lower filament. Siliceous precipitates enclose the Fe-rich filament and fringe the pores of the innermost core. P enrichment is localized to distinct concentric bands. The intensity scale ranges from 0 to 60,000.

Johannessen, K. C., McLoughlin, N., Vullum, P. E. & Thorseth, I. H. On the biogenicity of Fe-oxyhydroxide filaments in silicified low-temperature hydrothermal deposits: Implications for the identification of Fe-oxidizing bacteria in the rock record. *Geobiology*. **18** 21 (2019) https://doi.org/10.1111/gbi.12363



Images taken by C. Hell, E. Thronsen and D. Chatterjee showing dispersoids, voids and clusters in Al matrix.



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You can find us in the Natural Science Building (Realfagbygget) in the 4th floor – D4 and B4 corridors.

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

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