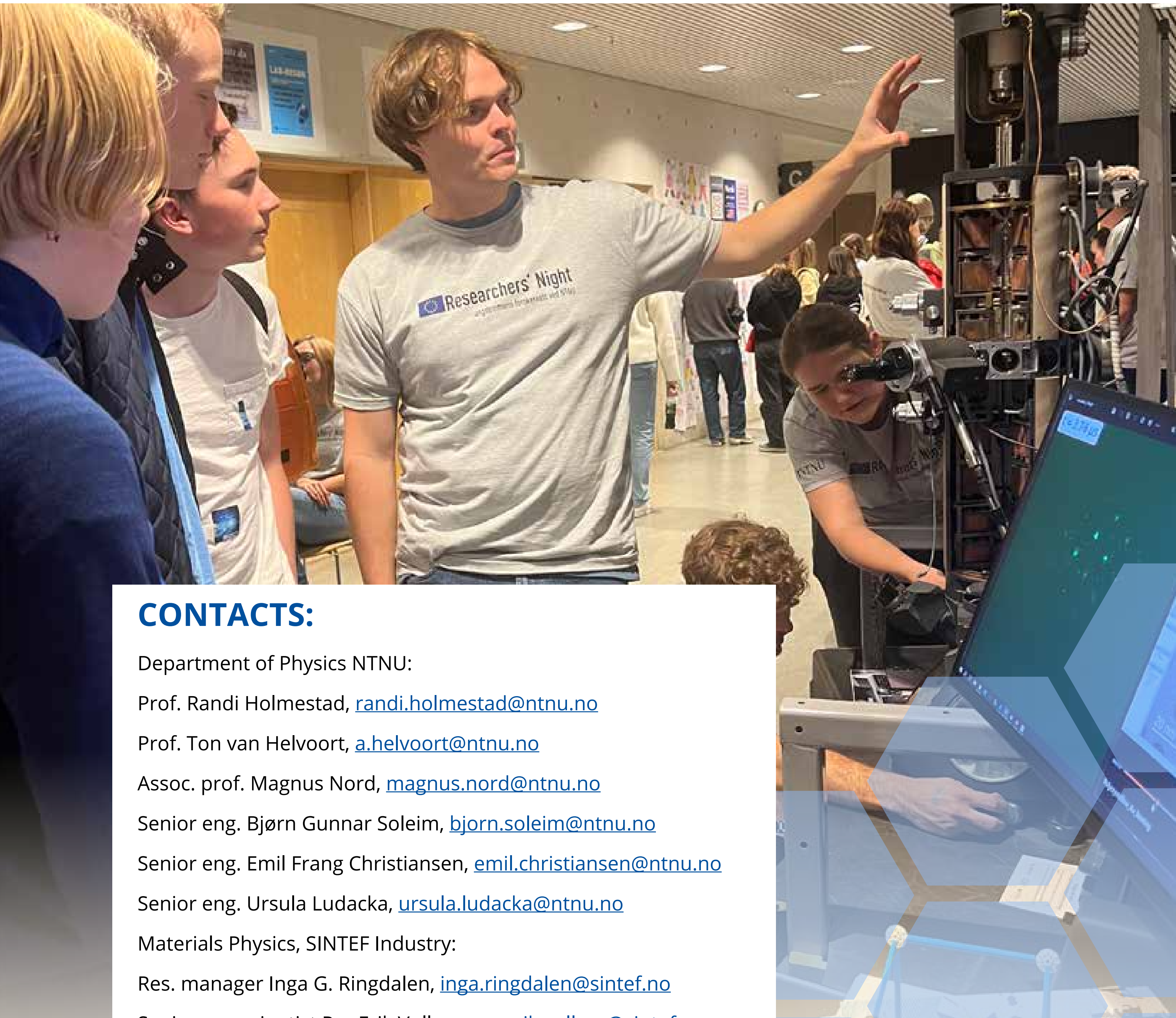
SINTEF

NTNU
Norwegian University of Science and Technology

TEM Gemini Centre



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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 Block 1 - K1. For general enquiries: temlab@phys.ntnu.no

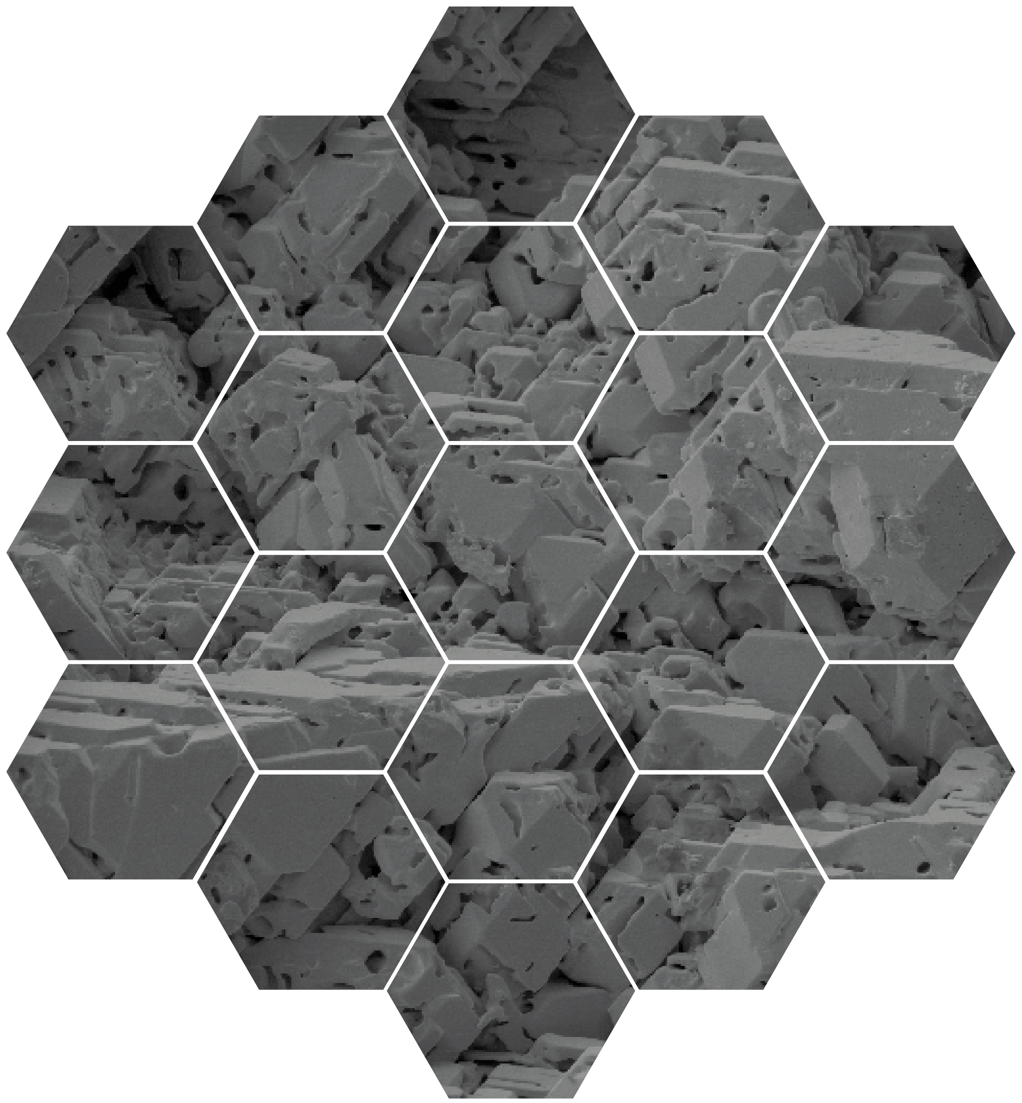
http://www.ntnu.edu/geminicentre/tem

2023

Annual Report

TEM GEMINI CENTRE

DEPARTMENT OF PHYSICS, NTNU
DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING, NTNU
MATERIALS PHYSICS TRONDHEIM, SINTEF INDUSTRY



All pictures and figures in the report are taken/made by people in the TEM Gemini - trykket på NTNU Grafisk Senter

Introduction

NORTEM

2023 has (again 😊!) been a good year for the TEM Gemini Centre. We see that structural and strategic efforts in the Centre pay off and enable users to perform quality work efficiently and fast. Together, NTNU and SINTEF collaborate through the Gemini Centre to create a safe, robust, and efficient research atmosphere. Many new users have been trained on the microscopes, the number of research papers is still high, and we have this year signed the contract for a new TEM which will be installed in 2025. The NORTEM II proposal for new microscopes in Oslo and in Trondheim was granted by the Research Council of Norway (RCN) through the INFRA program in December 2021, we had a lengthy competitive dialog with three companies, many meetings and several demos and tests. Two state-of-the-art instruments will be installed in 2025 (Trondheim-node) /2026 (Oslo-node). This re-investment is not only strategically important for research in Norway, but also a testimony to the competence of the host institutions and their ability to run advanced infrastructures. The TEM Gemini Centre collaboration has been crucial in this. The grant was a result of tremendous effort from the NORTEM consortium (UiO, NTNU, and SINTEF) and key members in the TEM Gemini Centre. The ever-continuing quest for excellence is also supported by the Centre through continued development of competence and science related to the NORTEM infrastructure in Trondheim.

In the Trondheim node, the three NORTEM instruments, financed by the RCN and the partners in 2011, have been using the total cost model for several years. This is crucial for a systematic management of the infrastructure. Managing this important infrastructure is always a team effort. With With high use of the instruments (but with high variations over the years) and the resulting high scientific throughput, we recognize the importance of strategic collaboration. While NTNU owns and runs the TEM infrastructure in Trondheim, SINTEF is an important scientific user and collaborator, providing stability and hands-on competence. Through this relationship, advanced TEM research is made available to both national and international industry and partners. We also see that continuous reinvestments are crucial.

2023 was the last year of the ESTEEM3 EU Horizon 2020 network project where NTNU has been a partner together with several other world-leading TEM laboratories. This has increased our visibility in Europe and led to new possibilities. We are now partners in two new EU projects, IMPRESS and RIANA. The Gemini Centre participates in a broad range of projects, including industrial and EU funded ones. Several long-term SFI projects also ended in 2023 – Centre of advanced structural studies (CASA), Sustainable innovations for automated manufacturing of multi-material products (SFI-Manufacturing) and Industrial catalysis science and innovation (iCSI) where both SINTEF and NTNU had TEM activities. We are currently active in one SFI - Centre for sustainable and competitive metallurgical and manufacturing industry (SFI PhysMet). Furthermore, the TEM Gemini Centre is central in the SumAl KPN project on aluminium with Norwegian aluminium industry. We have also started new research activity on magnetic materials. Also, the INTPART project with Japanese aluminium industry and academia ended in 2023, with a trip to Japan in April several from the TEM Gemini Centre participated. In May the Centre organized a seminar on clustering in age-hardenable aluminium alloys through the SumAl project with 46 participants at Bårdshaug in Orkanger.

As documented in this report, the Centre had 38 active hands-on users/operators, 15 users through operators and served 81 different projects, whose results have contributed to 48 journal publications (plus 8 in press/preprints) in 2023. 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, 2 PhD students were co-supervised, and 10 MSc candidates were educated with TEM as a substantial part of their theses in 2023. The TEM lab activity in scheduled courses is high, and 4courses with ~ 200 students used TEM in lab exercises last year. The annual TEM introduction course was for the first time after the pandemic given physically in September with 15 participants. The TEM Gemini Centre organizes weekly group meetings with presentations where often more than 30 group members participate. In relation to the NORTEM II investments and stability of the existing instruments, we are concerned about the Campus project at Gløshaugen. The complexity of the situation requires constant and diligent work from the Centre and is of great concern.

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 2023. For more details, see our home page.

TEM Gemini Centre management, February 2024.

BOARD AND MANAGEMENT OF TEM GEMINI CENTRE

TEM Gemini Centre board:

- Erik Wahlström, Department head, Department of Physics, NTNU
- Inga G. Ringdalen, Research manager, Materials Physics Trondheim, SINTEF Industry
- Ida Westermann, Department head, Department of Materials Science and Engineering

Centre management:

- Randi Holmestad, Physics, NTNU, Leader
- Inga G. Ringdalen, Materials Physics, SINTEF Industry
- Ton van Helvoort, Physics, NTNU
- Bjørn Gunnar Soleim, Physics, NTNU
- Emil Frang Christiansen, Physics, NTNU
- Ursula Ludacka, Physics, NTNU
- Per Erik Vullum, Materials Physics, SINTEF Industry
- Ruben Bjørge, Materials Physics, SINTEF Industry

PEOPLE IN THE TEM GEMINI CENTRE IN 2023

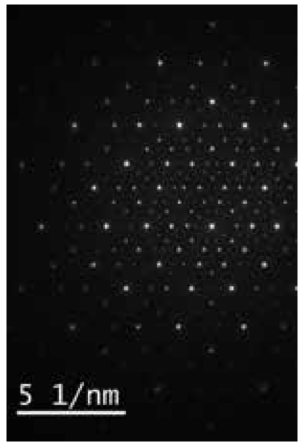
- Sigmund J. Andersen (Senior research scientist, SINTEF)
- Kaja Eggen Aune (Master student, DP, NTNU)
- Tina Bergh (Postdoc, Department of Chemical Engineering, NTNU)
- Ruben Bjørge (Research scientist, SINTEF and Assoc. Prof. II, DP, NTNU)
- Susanne Boucher (Master student, DP, NTNU)
- Dipanwita Chatterjee (Postdoc, DP, NTNU)
- Emil Frang Christiansen (Senior Engineer, DP, NTNU)
- Sivert Johan Vartdal Dagenborg (PhD student, DP, NTNU)
- Thea Marie Dale (Master student, DP, NTNU)
- Viljar Johan Femoen (Master student, DP, NTNU)
- Jonas Frafjord (Postdoc, DP, NTNU)
- Jesper Friis (Senior research scientist, SINTEF)
- Kristin Frøystein (Master student, DP, NTNU)
- Håvard Holm Fyhn (Master student, DP, NTNU)
- Erik Gaupseth (Master student, DP, NTNU)
- Evy Gjedrem (Master student, DP, NTNU)
- Espen J. Gregory (Master student, DP, NTNU)
- Olav Hellebust Haaland (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)
- Sindre Vie Jørgensen (Master student, DP, NTNU
- Supreet Kaur (Master student, DP, NTNU)
- Inga Dahlen Konow (Master student, DP, NTNU)
- Håkon Longva Korsvold (Master student, DP/ PhD student DMSE, NTNU)
- Petter Lervik (Summer student, DP, NTNU)
- Marthe Linnerud PhD student, DP, NTNU)
- Ursula Ludacka (Postdoc, DMSE / Senior Engineer, DP, NTNU)
- Hogne Lysne (PhD student, DP, NTNU)
- Calin D. Marioara (Senior research scientist, SINTEF)
- Anders Christian Mathisen (Master student, DP, NTNU)
- Brynjar Mæhlum (Master student, DP, NTNU)
- Magnus Nord (Assoc. Prof. DP, NTNU)
- Gregory Nordahl (PhD student, DP, NTNU)
- Inger-Emma Nylund (PhD student /Postdoc, DMSE, NTNU)
- Oskar Ryggetangen (PhD student, DP, NTNU)
- Martin Seiter (Master student, DP, NTNU)
- Simen Skurdal (Master student, DMSE, NTNU)
- Bjørn Gunnar Soleim (Senior Engineer, DP, NTNU)
- Hedda Christine Soland (Master student, DP, NTNU)
- Peder Stokkan (Master student, DP, NTNU)
- Jørgen Sørhaug (PhD student, DP, NTNU)
- Aurora Teien (Master student, DP, NTNU)
- Tor Inge Thorsen (PhD student, DP, NTNU)
- Elisabeth Thronsen (Research Scientist, SINTEF, DP, NTNU)
- Mats Topstad (Master student, DP, NTNU)
- Kristian Tveitstøl (Master student /PhD student, DP, NTNU)
- Per Erik Vullum (Senior research scientist, SINTEF and Assoc. Prof. II, DP, NTNU)
- Sigurd Wenner (Research scientist, SINTEF)
- Håkon Wiik Ånes (PhD student, DMSE, NTNU)

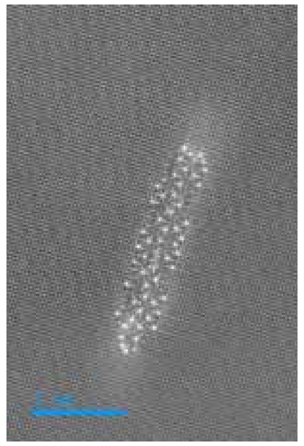
THE NORTEM PROJECT

NORTEM (Norwegian Centre for Transmission Electron Microscopy) was a nationally coordinated largescale infrastructure project (2011-2020) with three partners - SINTEF, NTNU and UiO, funded by the Research Council of Norway (RCN) and the three partners. The budget for new equipment and the re-building in the project was about 75 MNOK in total in two geographical nodes, Trondheim and Oslo. We have now been running the facility for ten years. The support to NORTEM from the Research Council ended in 2016, but the project continued to the end of 2020. In November 2020 we applied for a reinvestment (NORTEM II project) after the first application for reinvestment was not granted. In December 2021 we received the good news from RCN that the funding was granted, thus securing access to world-leading TEM in Norway for another decade. This proposal included upgrades of existing infrastructure and new instruments in both nodes. During 2023 there has been a common tender process for the Oslo and Trondheim node, where a competitive dialogue was done with JEOL, Thermo Fisher Scientific and Gatan companies. This ended with contract signing in December 2023. In the Trondheim node, we will get a JEOL GrandARM2, a new state-of-the-art probe corrected Level 1 instrument with modern cutting-edge direct detectors, CEFID energy filter, 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.

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 established a sound running model for the infrastructure. This has been established and is running well. Further work has been focused on securing the required resources for operating TEM in the best way. The funding of NORTEM II secures the necessary future upgrades, and our job is now to get the best out of the huge and complex investment ahead. For more information on NORTEM see the NORTEM webpages.

The Trondheim node of the NORTEM facility has three senior engineers, Bjørn Gunnar Soleim, Emil Frang Christiansen and Ursula Ludacka, supporting maintenance, training, competence, and techniques. Ursula had a temporary position from May 2023. The microscopes have a high uptime. Per Erik Vullum and Ruben Bjørge have been working as adjunct (affiliated) professors (20 %) at NTNU in 2023. This contributes to developing the interaction between NTNU and SINTEF.



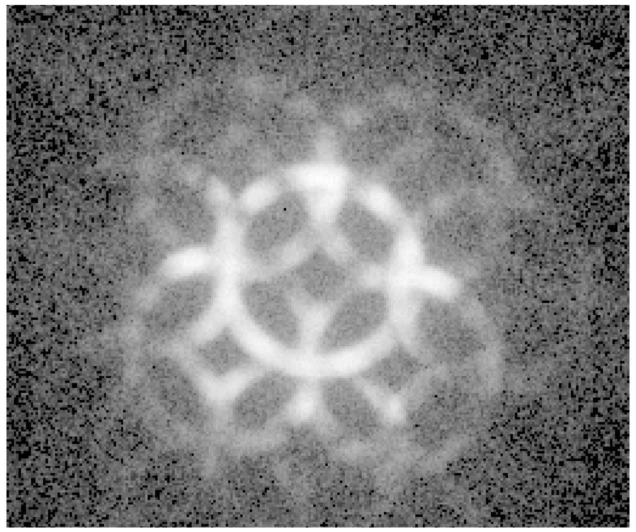


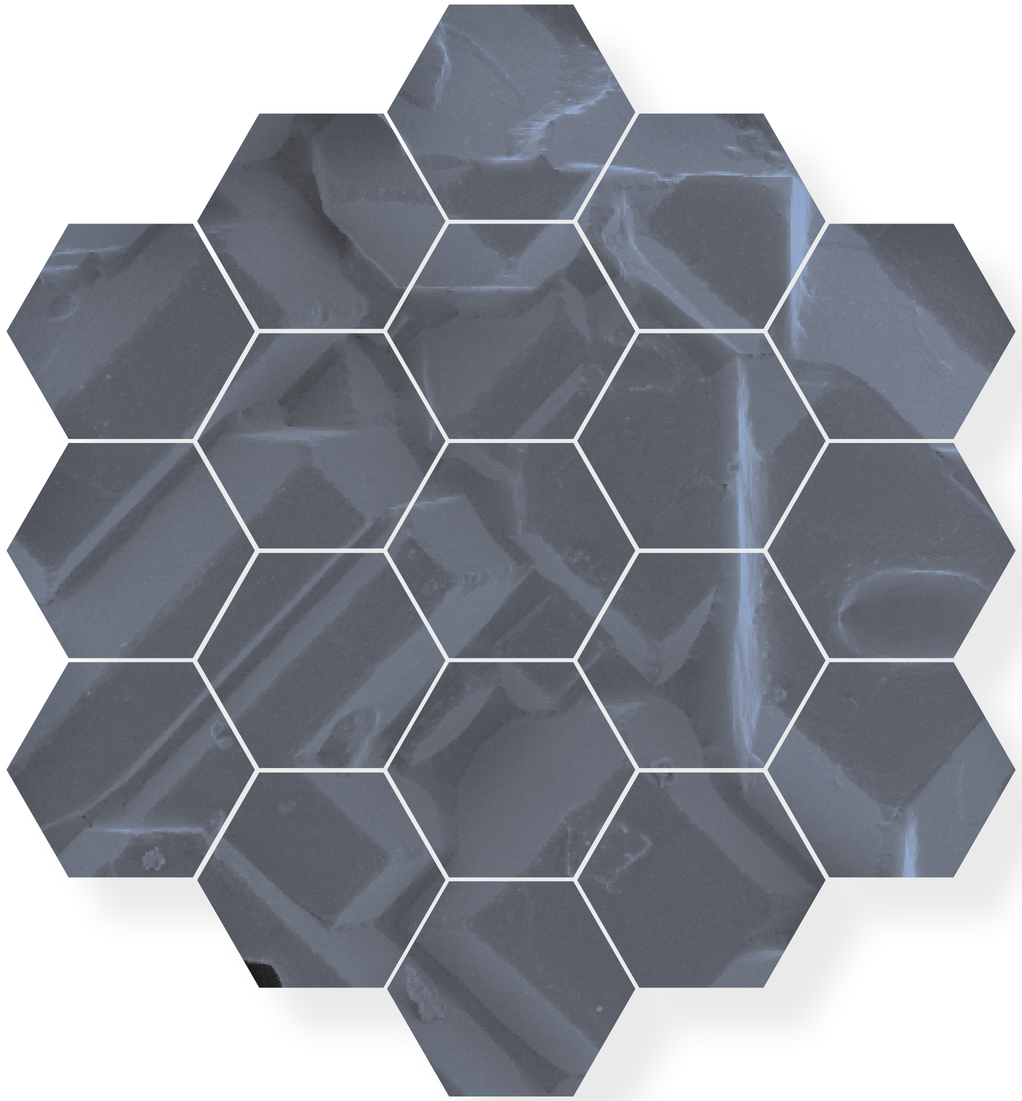
THE TEM GEMINI CENTRE

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 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 renominated in November 2018 for a new period of 4 years. In October 2022, the Centre was through a new renomination. Since DMSE does not have a TEM instrument anymore, and this department in principle does not have a different role related to TEM compared to other departments at NTNU, we agreed that DMSE should leave the Gemini Centre. However, since this department represents a large part of the external users of the TEM facility, we decided to keep the board, which still has three members, from DP, DMSE and SINTEF.

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 TEM. The overall objective of the TEM Gemini Centre is *'to build and secure a robust scientific environment within TEM with high international profile as a sound basis for growth, not only for the Centre itself, but also for other parts of NTNU and SINTEF and academic and industrial partners.'*

Parallel to and together with this, the large nationally coordinated infrastructure project, NORTEM, has given a broader identity to the Centre's TEM infrastructure. The Gemini centre is the NORTEM Trondheim node. The collaboration in general represents a model for strategic research coordination between parallel research groups at SINTEF, NTNU and UiO. The aim is to develop large-scale technical centres that produce higher quality results collectively than the individual groups would achieve independently. The Gemini collaboration enables collaborating groups to grasp new opportunities to create values and improve profitability. High-quality technical centres are in great demand internationally from both commercial clients and students. The shared vision of Gemini Centers is: *"Global excellence together"*. The groups must undertake joint strategic processes as the basis for their research planning and technical coordination in connection with large-scale projects. Furthermore, they must create joint fora for concept development and information exchange and share approaches to investment and operation of laboratories and equipment. The strategic plan includes all aspects of the collaboration model, from teaching and research to commercial research projects, entrepreneurship, recruitment, and internationalization.





Instrumentation

JEOL double corrected JEM-ARM200F (cold FEG)

This is currently the top instrument in the TEM Gemini centre. 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.

JEOL JEM-2100F

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

JEOL JEM-2100

The 2100 LaB6 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 TEM INSTRUMENTS IN TRONDHEIM

The TEM Gemini Centre has three TEMs installed as part of the NORTEM project in 2012/2013 - a JEM-2100 LaB6, a JEM-2100F and a double corrected JEM-ARM200F.

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

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

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)

Instrument photos by O. M. Melgård

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, reflecting the broad range of materials studied. The Centre has different types of grinders, dimplers, saws, an ultrasonic cutter, and other tools for TEM specimen preparation of metal and ceramic cross-sectional specimens. The Centre has three Gatan PIPS instruments, including a PIPS II, to make more high-quality and reproducible specimens. A routine has been developed to polish focused ion beam (FIB)-made TEM lamellas to obtain the highest specimen quality and the best possible TEM results. Many TEM projects utilize the FIB instrument 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 prep together with NTNU NanoLab. Here a special application area is advanced characterization of battery materials. Recently, a plasma FIB has been installed in the Department of Mechanical and Industrial Engineering, which is good to use for TEM sample prep. The electro-polisher at DP is essential in producing high quality aluminium 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. A recent development is utilizing chemical methods at NTNU NanoLab to remove thin film substrates, allowing for the creation of large field-of-view plan view samples. This “back-etching” technique requires the thin film to be deposited on a chemical etch stop, like Si_3N_4. In addition to the large field-of-view, it will enable the fabrication of customized in-situ TEM samples when paired with in-situ TEM holders. Relevant materials for this are ferroelectric thin films, magnetic materials and other potential device concepts which can be fabricated onto Si_3N_4.

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, two tomography holders, two tilt-rotation holders and back-up double tilt holders. Another special holder is the MEMS based heating holder, which can also be used for biasing.

USER STATISTICS FOR 2023

The total registered used time for the three instruments in 2023 was 3781 hours, including 205 non-paid hours used for testing, competence development, demonstrations, and guided tours. Of the 3576 paid hours, the use by NTNU corresponds to 73 %, externals (with NTNU operator) 5 %, and SINTEF 22 %. NTNU's use is divided over five departments, where the main use is from Department of Physics (78 % of NTNUs paid hours). 81 different projects used TEM in 2023. The infrastructure had in total 53 users, of which 38 were hands-on operators. 5 of the users were based at SINTEF, 17 were PhD candidates and 18 were master students.

4 courses, with in total ~200 students, have had lab exercises using the TEM instrumentation in 2023, these are,

. TFY4220 - Solid State Physics
. TFY4255 - Materials Physics
. TFY4330 - Nanotools
. TMT4166 - Experimental Materials Chemistry and Electrochemistry

Table 1: User statistics, TEM Gemini Centre 2024

Microscope use in hours	ARM200F	2100F	2100	SUM
SINTEF	437	187	176	800
NTNU – Physics	394	822	515	1731
NTNU – Other departments	274	140	114	528
NTNU – Visitors from abroad	108	61	8	177
NTNU – Teaching lab	0	174	5	179
External	118	43	0	161
NTNU – Set-up/testing/ training/demonstrations	116	65	24	205
Total use	1447	1492	842	3781

Activities

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 five focus areas, which have been important for the TEM Gemini Centre activities since the Centre was formed. These focus areas were also in the core NORTEM I and II proposals and are specified in the Centre's recent strategy document. Within these areas we still see potential for further growth and tackling unsolved issues with TEM. They are light metals, energy materials, nanotechnology, magnetic materials, and catalysis, and will be strategically important for Norway also in the future. The TEM Gemini Centre had activities in all these four areas in 2023. In addition, we have high and increasing activity connected to data analysis. The next sections describe these activities. Activities in aluminium alloy research are the largest. In all areas the use of advanced data processing has gained significance.

ALUMINIUM - LIGHT METALS

The study of aluminium alloys using TEM has been a pillar in the Trondheim TEM environment for many years, and there have been many successful projects. All projects have been jointly between NTNU and SINTEF and supported by the Research Council of Norway and Norwegian light metal industry, in particular Hydro Aluminium. We were in 2023 involved in 3 SFI Centers and one competence project (KPN) with aluminium research, in addition to the INTPART project with Japanese universities and aluminium industry. SFI CASA, headed by Prof. Magnus Langseth at the Structural Engineering department in the NTNU Engineering Faculty, ended in 2023. Here we were involved at the "lowest scale" of the multiscale activities, including TEM and atomistic calculations of precipitates, grain boundaries, precipitation free zones and dislocations in deformed, mostly industrial, Al alloys. The project leader of the SINTEF part of CASA Lower scale is Inga G. Ringdalen. SFI CASA has made a promotion video where TEM on aluminium has a central part. SFI Manufacturing, headed by Dr. Sverre Guldbrandsen-Dahl from SINTEF Manufacturing, also ended in 2023. Here, we had some activity on joining of aluminium with other materials in multi-material products. In 2023, Ruben Bjørge's II position was financed from this project.

The newest SFI the TEM Gemini Centre is involved in, is SFI PhysMet - *Centre for Sustainable and Competitive Metallurgical and Manufacturing Industry* - based in the Department of Materials Science and Engineering with Prof. Knut Marthinsen as a Centre director. TEM is the main topic in Research Area 1 (RA1), Multi-scale materials analysis, headed by Randi Holmestad. SINTEF is involved in this Research area, leaded by Sigurd Wenner. Tor Inge Thorsen was a PhD student here studying different joining techniques and additive manufactured materials but ended his engagement in 2023. A common problem for the thermal welding processes is the creation of heat affected zones where the strength of the material is significantly reduced. The effects of alloying elements, nanoparticles and heat treatments was studied. Håkon L. Korsvold was hired at DMSE in RA1 in August 2023, supervised by Yanjun Li, working on combined TEM/APT study to understand grain boundary segregation in aluminium alloys.

The FRIPRO project 'QUATRIX' - *Quasicrystal nucleation in a metallic matrix* started in 2021. Much attention has been directed towards quasicrystals since their discovery, but many aspects of these peculiar structures are still unknown. Specifically, the nucleation and growth of quasi-crystalline particles in metallic host matrices is understood to a very limited extent. The QUATRIX project aims to shed light on the structures and precipitation mechanisms of quasicrystals within a selection of alloy systems, and thus to produce generic knowledge about quasicrystal growth and structure. QUATRIX is mainly a SINTEF project, with Dr. Ruben Bjørge as a project leader and with one PhD at NTNU - Oskar Ryggetangen. He focuses on acquiring and analysing electron diffraction data from quasi-crystalline phases, aided by advanced high-resolution TEM.

The Nano2021 project 'In-Sane' - *In-situ studies of highly conductive bonded interfaces between aluminium and copper at the nanoscale* –is a collaboration with Department of Mechanical and Industrial Engineering (MTP) where Randi Holmestad is the project leader. The idea is to perform nanoscale joining in the FIB at the nanoscale, 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 micro-joints with strong and sharp interfaces for battery power packs. PhD student Jørgen Sørhaug does advanced TEM in this project. One PhD student (Ambra Celotto) works at MTP and focuses mostly on making the joints in the FIB. SINTEF is involved through Per Erik Vullum.

We have currently one competence project on aluminium - SumAl - *Solute cluster manipulation for optimized properties in Al-Mg-Si based Al alloys* - with industry partners from Norway (Hydro, Benteler and Raufoss Technology), Austria (Neuman), Sweden (Hydro) and Germany (Speira). 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 the project performs both TEM experiments and modelling within the TEM Gemini Centre. PhD student Christoph Hell does advanced TEM in this project, focusing on effects of heat treatments on clustering and precipitates in 6xxx alloys. Jonas Frafjord is working as a postdoc. He is doing density functional theory (DFT) and molecular dynamics in combination with other higher scale methods to explore clustering in Al alloys. SINTEF has a big part of this project, doing TEM, in addition to APT and modelling.

The NAPIC (NTNU aluminium product innovation Centre) was established in 2017, and Håkon Wiik Ånes has been working as a PhD student in this Centre, based in DMSE to study nucleation of recrystallization using SEM and TEM. The SINTEF part is heavily involved in the Green Platform project *AluGreen*, where many industry partners are brought together, and recycled aluminium is studied and utilized on several scales. How to increase the amount of recycled aluminium is an important task for the green transition which all industries are facing.

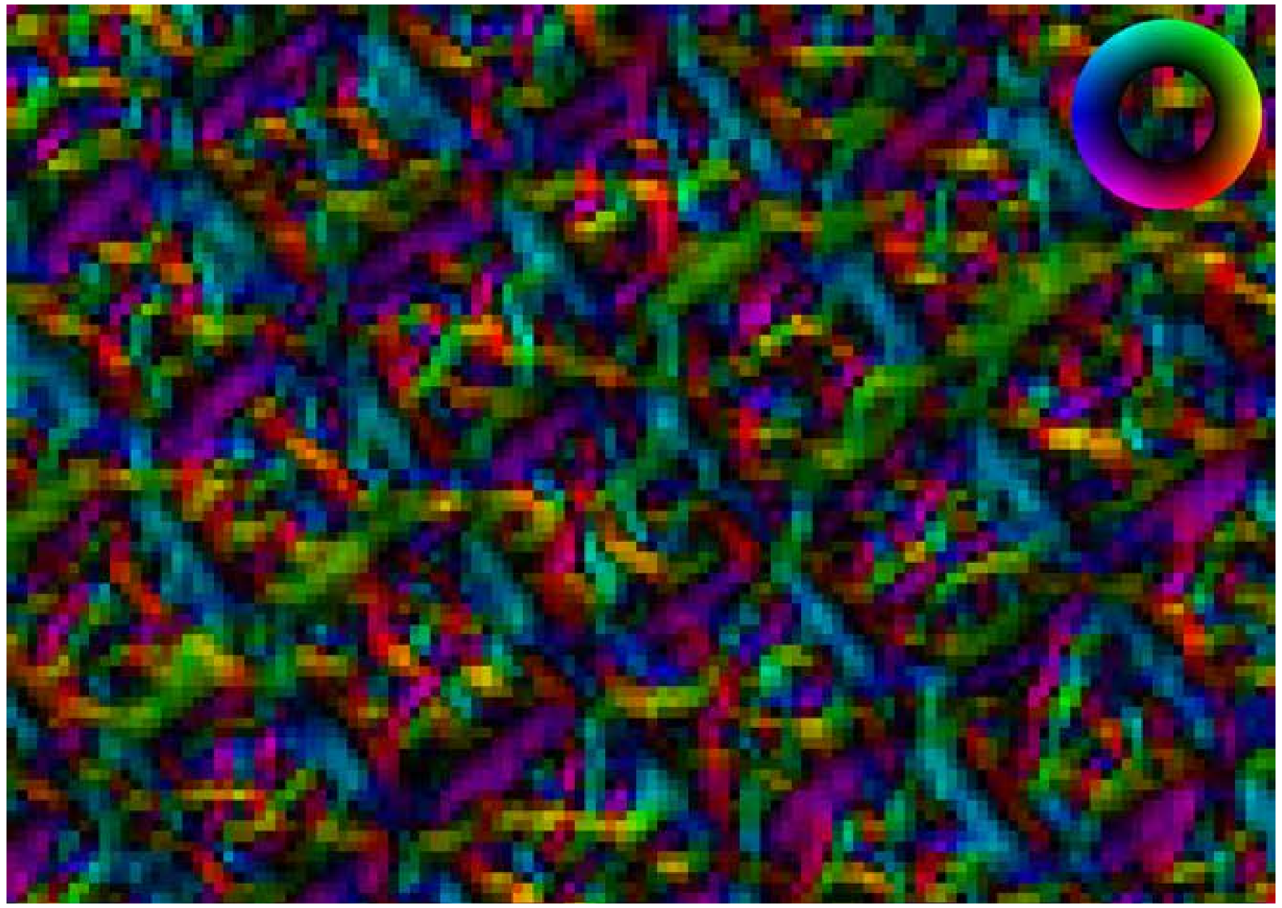
During the last years, we have had several aluminium alloy related collaborations abroad. The largest is the Japanese collaboration with academia and industry, where the INTPART project was renewed for 4 new years in 2019 and extended with new partners. This is further presented elsewhere in this report.

As seen from the publication lists of the TEM Gemini Centre, we have the last year had many invited talks about aluminium activities at international conferences, both material and microscopy conferences – SCANDEM in Sweden, Thermec in Austria, M&M in USA and IMC in South Korea - which shows that our work on aluminium is internationally recognized.

ENERGY MATERIALS

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 finished his PhD in 2023 in the FME, working on TiO_2 thin film growth and characterization with Turid Reenaas as main supervisor.

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 aim is to develop Si/graphite-based composites as anodes in commercial Li-ion batteries. TEM is the primary tool to characterize and understand the behaviour of the anode composites as a function of structure, morphology and cycling conditions. From the beginning of 2022 this R&D is continued through the Green Platform project SUMBAT. Another IPN project, "Coloumbus", 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 behaviour 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, Vianode, 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. "SAGA" ended in 2022, and this work is now followed up as one of the sub-projects in the Green Platform project "SUMBAT". TEM is also used in several other projects related to development of Li-ion battery technologies. Such projects include the Horizon2020 project "Hydra", and the RCN funded projects "HighCath" and "LongLife". In FME MoZEES TEM is also used to characterize and understand the fundamental behaviour of the battery electrodes as a function of electrode and electrolyte compositions, synthesis and cycling conditions. Postdoc Inger-Emma Nylund works here on advanced TEM characterization of $LiMn_{2-x}Ni_xO_4$ as high voltage cathode materials in Li-ion batteries.



NANOTECHNOLOGY

For nanotechnology and nano-sciences TEM plays an essential role. TEM is important because it can analyse structure and composition on the same small volume and thereby contribute to understand and tailor the properties of nanomaterials. The TEM Gemini Centre activities at all levels cover nanoparticles, 1D-nanostructures (i.e. nanowires, carbon nanotubes, cellulose fibrils), 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 complementary equipment in the cleanroom. Especially, the FIBs are important for the TEM Gemini Centre. New possibilities have materialized by using the Xe-based FIB (owned and run by Department of Mechanical and Industrial Engineering). With collaborators, primarily in Trondheim, we develop methods for correlated studies where TEM is directly combined with SEM, EBSD, Cathodoluminescence, Photoluminescence and Scanning Probe Microscopy techniques. Hereby, more all-round characterization of nanomaterials is realized. NorFab is an important partner for the TEM Gemini Centre in many projects. Many of our MSc students follow the Nanotechnology study program, a further demonstration that TEM is integrated part of practical nanotechnology. These MSc projects include both practical as well as theoretical/computational focus. In the TEM Gemini Centre, both NTNU and SINTEF have worked with start-up companies within nanotechnology such as CrayoNano. TEM is here the only tool able to both describe the crystal structure in nanostructures, including various types of defects, and the chemical composition for example in heterostructured nanowires and thin films.

MAGNETIC MATERIALS - IMAGING ELECTROMAGNETIC 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 associate professor Magnus Nord is to improve the STEM-DPC technique. Utilizing the recently installed MerlinEM fast pixelated direct electron detectors, together with PhD student Gregory Nordahl, Marthe Linnerud, Sivert Dagenborg and Kristian Tveitstøl, as part of the Young Research Talents project InCoMa - *In-situ correlated nanoscale imaging of magnetic fields in functional materials* - from RCN in 2021. The newly installed 4D-STEM detector at the ARM, has given another microscope which can perform STEM-DPC. 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. These types of magnetic experiments can require a great deal of manual user input. A recent focus area is scripting control of the TEMs to enable a greater degree of automation, as part of the HORIZON Europe IMPRESS project. The aim is not only to do more advanced *in situ* magnetic experiments, but also to extend this to other types of TEM characterization.

CHEMICAL ENGINEERING - CATALYSIS AND MEMBRANE MATERIALS

The Centre has continued a strong interaction with the national catalysis environment, including the NTNU Chemical Engineering department and SINTEF Industry. The SFI *Industrial catalysis science and innovation* (iCSI), which ended in 2023, headed by Prof. Hilde Johnsen Venvik, hired Tina Bergh as a postdoc (2021-2025) to provide a platform for further applications of TEM in both academic and industrial catalysis research. The main goal is to develop and use improved (S)TEM methodologies for specimen preparation, in-situ heating and gas reactions, and data collection and analysis. When it comes to the materials studied, the main focus in 2023 was placed on silver catalysts for the methanol to formaldehyde reaction. The silver catalysts were studied after exposure to various reaction atmospheres at elevated temperatures by FIB-SEM tomography and STEM methods, in particular SPED. Also, the newly installed fast TEM camera on the ARM in combination with in-situ holders, will allow studying dynamics.

ADVANCED DATA PROCESSING

Science and technological developments are data driven. The ongoing revolution within TEM research is digital and data-hungry and in the TEM Gemini Centre data processing is a corner stone. This includes machine learning and smart algorithms for analysis of images, spectral data and electron diffraction. In 2022 the TEM Gemini Centre installed its second direct electron detector. This is placed on the double-corrected ARM, and its main task is 4DSTEM techniques with probes down to lattice dimensions. In addition, the 2k CCD was replaced by a 4k CMOS camera with in-situ capacities for fast (big) data acquisition in both TEM and STEM mode and is now fully integrated/operative. These upgrades triggered a necessary restructuring of the lab's data transfer handling, for which we received good practical support from NTNU IT services. The TEM Gemini Centre has a share in the NTNU cluster IDUN, and is utilizing this, in particular for 4DSTEM data analysis. In addition, users have access to local workstations. With all the developments efficient and transparent data processing is getting more important. We benefit from the fact that our MSc and PhD students have a solid education regarding numerical and computational techniques. Many student projects have a (strong) data handling component in them. They actively contribute to open-source initiatives such as the Python library HyperSpy (hyperspy.org) and especially for electron diffraction pyXem (github.com/pyxem) as detailed in the publication list at the end of this report Open science is growing and pushed by EU and other authorities. It is a key element of modern science and innovation.

A new project in 2023 was the IMPRESS project, a Horizon Europe EU project – headed by Regina Ciancio. Kristian Tveitstøl was hired as a PhD student from August 2023 and will work on scripting control and automation of the TEMs at the TEM Gemini Centre. This to enable more advanced experiments, but also to increase the efficiency through automation of the TEMs.

MAKING TEM MORE ACCESSIBLE AND USER FRIENDLY!

IMPRESS stands for Interoperable electron Microscopy Platform for advanced RESearch and Services. Our goal is to design and deliver TEM instrumentation conceived at the highest level of open standards and interoperability. The architecture of this innovative platform will be based on interchangeable components that can be readily customized by scientists and further adjusted, taking into account the needs of users from different scientific communities. We aim to make TEMs flexible so they can be adapted to a diversity of multimodal experiments, instead of adapting experiments to TEMs. (https://e-impress.eu/about/)

RESEARCHERS' NIGHT, SCHOOL VISITS AND OUTREACH

The TEM Gemini Centre has contributed to many high school visits and Researchers Night during the last year. At Researchers Night in September 2023 a big delegation from the TEM group participated, some remotely from the basement of KJ1.

THE EU NETWORK PROJECT ESTEEM3

The TEM Gemini Centre has been a partner in the EU Horizon 2020 INFRAIA initiative ESTEEM3. ESTEEM3 was 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 ended in June 2023. NTNU was 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 University of Science and Technology in Krakow, Poland. In addition, we were affiliated to the work packages 'Diffraction' and 'Data analysis'. The main part of ESTEEM3 was the TA, where we were involved in more than 20 projects. These projects were initiated from Germany, Romania, UK, Spain, Sweden and Japan. TA exchanges did not only include data acquisition on the TEM, but also data handling. The website of ESTEEM3 (esteem3.eu) is still active and gives info about the project. Dr. Dipanwita Chatterjee was 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' together with AGH, Krakow, Poland.

NTNU is also involved in the work on creating a sustainable electron microscopy infrastructure collaboration in Europe and is one of the founders of the eDREAM (see https://e-dream-eu.org/) initiative.

NordTEMhub

The Nordic network in transmission electron microscopy (TEM) and materials science – NordTEMhub – was granted in 2020. This is a network funded by NordForsk, gathering the TEM groups in physical sciences from seven universities in the Nordic countries: Linköping University, Stockholm University, Chalmers, DTU, Aalto University, University of Oslo and NTNU – for utilizing complementary instruments, cooperating and working together, running workshops, having student exchange, finding best practice in lab management etc.

The aim is to establish collaborations, provide access, optimize instrument use, and build and utilize Nordic competence on advanced microscopy. Common to all the nodes are recent and significant investments in state-of-the-art transmission electron microscopes. This initiative adds value to academia and industry in the Nordic countries and strengthens the Nordic competence in electron microscopy within materials, physics, chemistry, and adjacent disciplines.

Because of Covid, the start of the Hub was very delayed, but the project is prolonged to 2025, and hopefully we will see some more good Nordic collaboration in the years to come. NTNU will organize a new workshop in 2024. As a part of the NordTEMhub, and also supported by NTNU NanoLab, we have started a collaboration with Stockholm University and Tom Willhammar on 3-dimensional electron diffraction (3DED).

SIDI

One new project in the TEM Gemini Centre in 2023 was the SIDI project 'Sustainable graphite Inoculants for Ductile cast Iron'. This is an innovation project for the industrial sector (IPN) with Elkem in Kristiansand. Elkem aims at a fundamental understanding at atomic level of the graphite nucleation that determines the final properties of the cast iron, systematize that knowledge, and offer scientifically based recommendations for internal product development and client foundries. To achieve that goal, the team of experts from Elkem and Mandal Castings will work closely with research scientists from NTNU and SINTEF providing access to top level experimental facilities, advanced characterization, and numerical modelling.

INTERNATIONAL COLLABORATION

2023 has been the first 'normal' year after Covid, and international travel is almost back to normal. Most of our international network is therefore back, and we hope for more exchange in the years to come!

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. Through the ESTEEM3 project, the facility has been used by several renowned researchers across Europe. In addition to the ESTEEM3 project, the INTPART project ensures international collaboration, in this case with Japan. We had students from Japan in the first part of 2023 from Tokyo Institute of Technology and Kyushu University. We thank all our international collaborators for the productive and stimulating (digital) interactions and hope we can be able to continue the cooperation in the coming years

List of visitors:

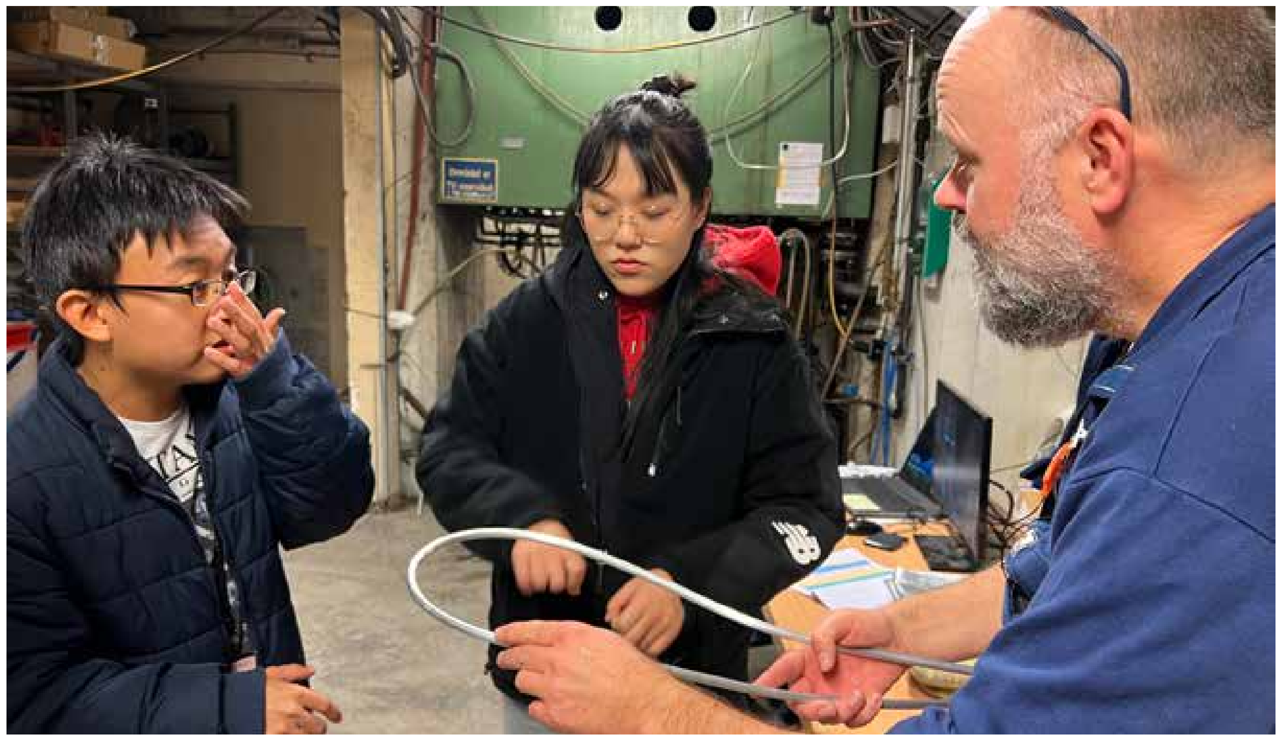
- Kenji Kaneko, Kyushu University, Japan, Jan. 23
- Ginevra Lalle, Sapienza University of Rome, Italy, April-July, 23
- Tom Willhammar, Stockholm University, Sweden, April 23
- Evgeniia Ikonnikova, Stockholm University, Sweden, April 23
- Aleksander Mosberg, Super-STEM, UK, April 23
- Yasuhito Kawahara, Kyushu University, Japan, Sept. 22 - Febr. 23
- Yujin Rhee, Tokyo Institute of Technology, Japan, Sept. 22- March 23
- Eiji Abe, Tokyo University, March 2023
- Ernst Kozeschnik, TU Wien, May 2023
- Seungwon Lee, University of Toyama, May 2023
- Stefan Pogatscher, Montanuniversität Leoben, Austria, May 2023
- William Curtin, Brown University, US /EPFL, Switzerland, May 2023
- Alexis Deschamps, Univ. Grenoble Alpes, May 2023

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 objectives of this project are 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. Due to the pandemic, there has been extremely low activity in the INTPART project during the project period, and this project was prolonged to June 2023. Two Japanese students stayed in Trondheim until February/March - PhD students Yasuhito Kawahara from Kyushu University and Yujin Rhee from Tokyo Institute of Technology - doing TEM work and using the NORTEM facility. In January, Prof. Kenji Kaneko from Kyushu came for a visit.

In 2023 most Covid measures were gone, and 11.-20. April a large delegation from NTNU and SINTEF (2 professors (Holmestad, Marthinsen, 3 researchers from SINTEF (Mariaora, Bjørge and Thronsen), 3 PhD candidates, 2 Postdocs and 2 MSc students) and one representative from Hydro, Sunndalsøra (Thrane), visited Japan, including visits to several universities (Tokyo University, Osaka University and Kyushu University) and industry (Toyo and UACJ) companies. Moreover, a 2-day workshop was organized at Tokyo Institute of Technology with 50 participants from aluminium industry, professors and colleagues, postdocs, PhD and MSc students from the partners of the INTPART project.

In October 2023, Knut Marthinsen and Randi Holmestad participated in the CAMRIC conference by Zoom. Knut and Randi also gave lectures online for Japanese students. We can hopefully manage to find other ways or projects to continue the collaborations for the next years.



The venue for the seminar was beautiful Bårdshaug Herregård in Orkanger, outside of Trondheim.
Invited and local key speakers at the seminar; Lee (Toyama), Frafjord (NTNU), Marioara (SINTEF), Kozeschnik (TUWien), Bergh (NTNU), Pogatscher (Leoben), Curtin (EPFL/Brown), Deschamps (Grenoble), Holmedal (NTNU).

CLUSTERING SEMINAR IN MAY 2023

46 participants attended a successful seminar on clustering and precipitation in aluminium alloys MAY 10-11, 2023. The seminar was organized by the TEM Gemini Centre through the KPN project SumAl and coordinated by Randi Holmestad and Jonas Frafjord. Talks and topics included advanced experimental characterization techniques (TEM, APT, DSC, SAXS) at the nanoscale and novel atomistic and mesoscopic scale modelling in aluminium alloy design with focus on nucleation and early stages, and how to manipulate thermomechanical processing to obtain the best alloy properties. Participants were researchers, postdocs and PhD students interested in these topics as well as representatives from industry partners. 10 of the participants came from the aluminium industry (Hydro, Speira, Neuman, Raufoss Technology) and also gave presentations on what is important, seen from an industrial point of view. The role of clusters was discussed in the context of industrial processing and modelling. The seminar was a great opportunity to have formal and informal discussions bringing us closer to understanding clustering and precipitation in aluminium alloys.

LIFE DEVELOPMENTS

Two central people in the TEM Gemini Centre turned 50 years in 2023, Calin Marioara in February and Ton van Helvoort in June. Both were celebrated with cake! Congratulations! In November, Sigmund Andersen retired from SINTEF. In 2023 we also had three new TEM citizens – Emil got two baby girl twins in November and Elisabeth gave birth to girl in December! Congratulations to all!

PHD DEFENSES IN THE TEM GEMINI CENTRE 2023

Kasper Aas Hunnestad, 23.11 2023.

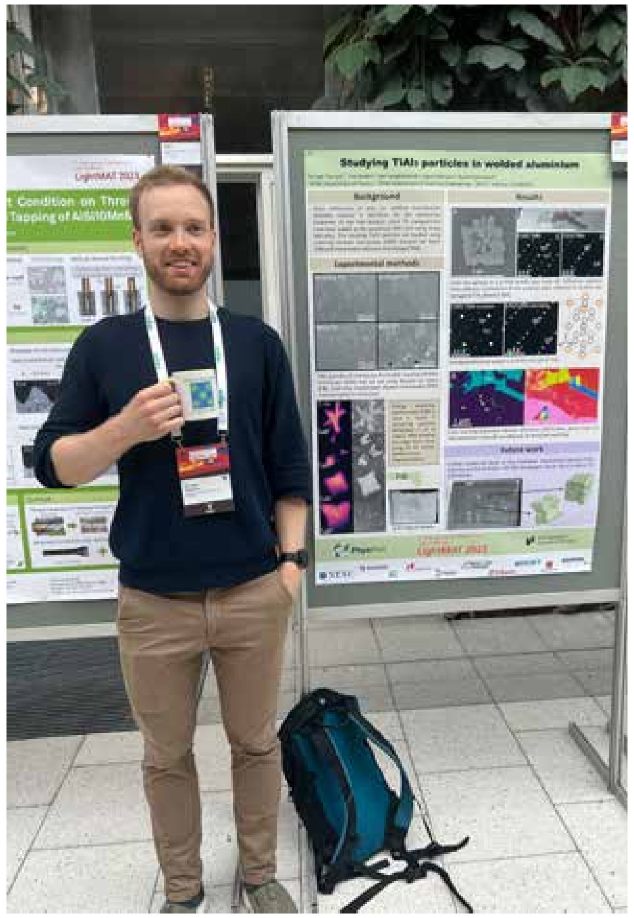
Kasper Aas Hunnestad worked in the FACET group in the Materials Science and Engineering Department and wrote a thesis with the title 'Nanoscale Chemical Analysis of Ferroic Materials and Phenomena'. Kasper did groundwork on applying atom probe tomography (APT) to study functional oxides, specifically ferroelectric oxides, demonstrating the potential of APT for detailed characterisation for oxides. The work included APT process parameter optimalization, doping and studying small compositional variations at domain walls, grain boundaries and superstructures. Kasper used in his work the national facilities NorFab, MiMac and NORTEM as the work required combining different advanced techniques. The work led to several high impact publications. Kasper joined the TEM group in 2018 as project student and has been a respective and active TEM group member.

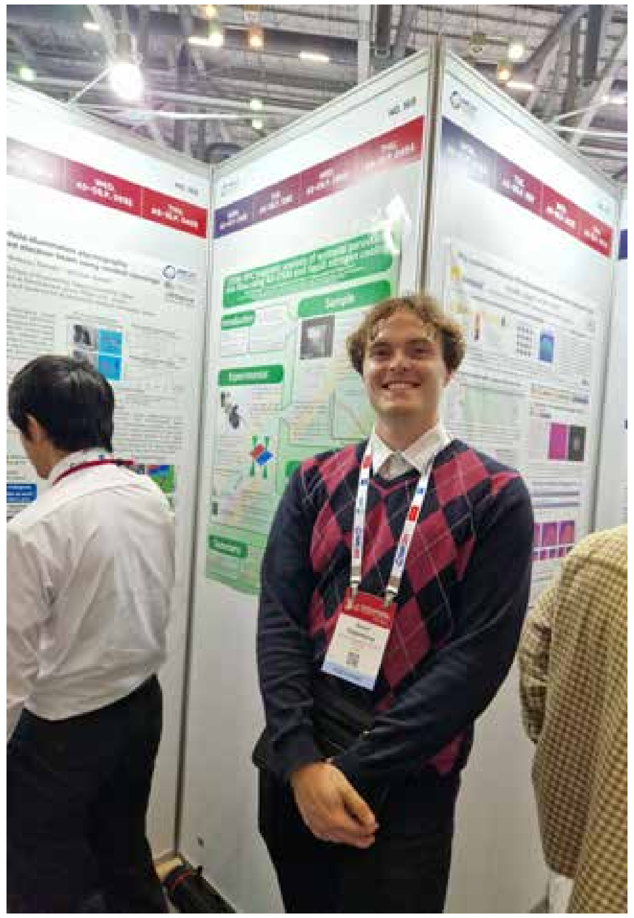
Kasper had Dennis Meier as supervisor and Ton van Helvoort as co-supervisor. The examination committee consisted of Dr Miryam Arredondo-Arechavala (The Queen's University of Belfast, UK), Professor Baptiste Gault (Max-Planck-Institut für Eisenforschung, Germany), and Associate Professor Hendrik Bentmann, (NTNU, Norway), who acted as administrator for the defence.
Kasper is currently postdoc at the Department of Electronic systems, NTNU, working at a different length scale.

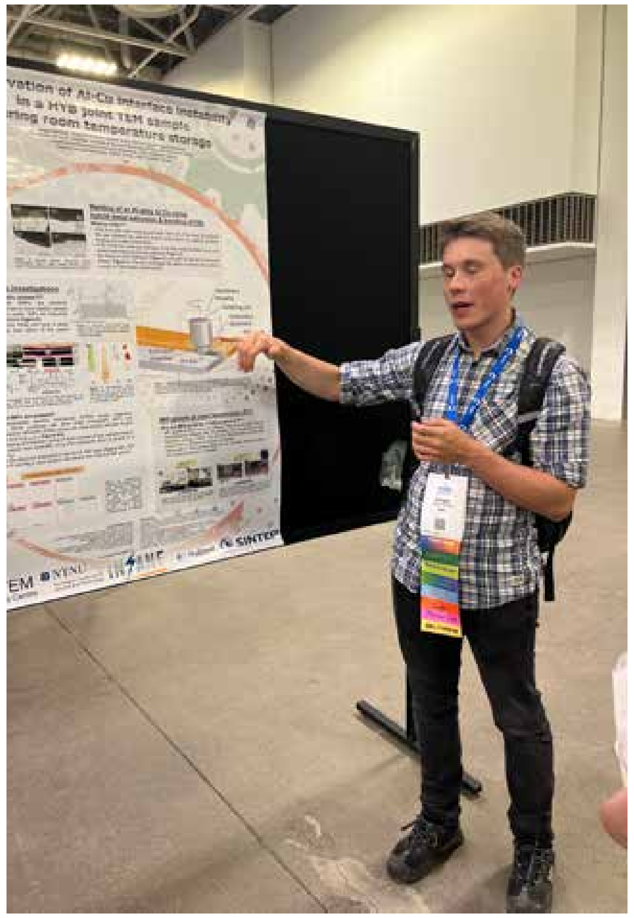
Hogne Lysne, 05.07 2023

Hogne Lysne defended his PhD 'Design and fabrication of (Cr + N) co-doped TiO2 films with a continuous compositional spread' 5th July 2023. Hogne's supervisor was Turid Reenaas, with Marisa De Sabatino and Randi Holmestad as co-supervisors. Hogne worked on intermediate band solar cells where you can increase the conversion efficiency by creating an additional, intermediate, energy band between the valence band and conduction band. The aim of this PhD project was to utilize combinatorial pulsed laser deposition (PLD) to deposit (Cr + N) co-doped TiO2 with a continuous compositional spread (CCS). The films were deposited on silicon substrates, and the amount of doping was controlled by separate ablation of a TiO2 target and a CrN target. An improved method for the design of films made using combinatorial PLD was developed. The characterization of the optical properties of the CCS films showed a sub-bandgap optical absorption at energies relevant for a TiO2 based intermediate band material, and in good agreement with what has been reported in literature. The characterization of the micro-structure of the films revealed that the sub-bandgap absorption correlated with the local crystal quality of the film and could be associated with a crystalline phase present for high Cr and N doping concentrations.

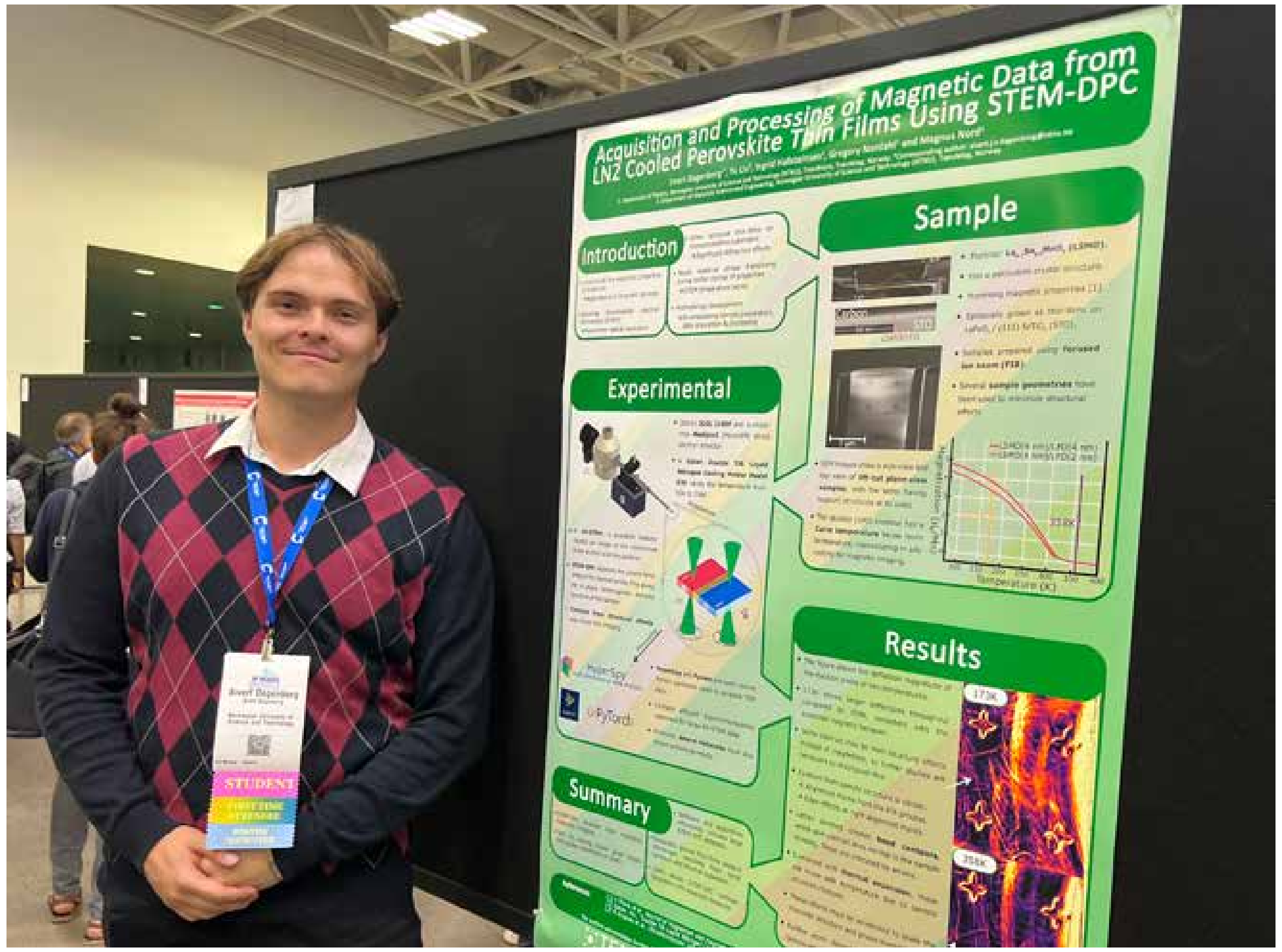
The examination committee consisted of Gertjan Koster, University of Twente, Patricia Almeida Carvalho, SINTEF, with Raffaela Cabriolu from NTNU as administrator. Hogne is now working in Alcatel.

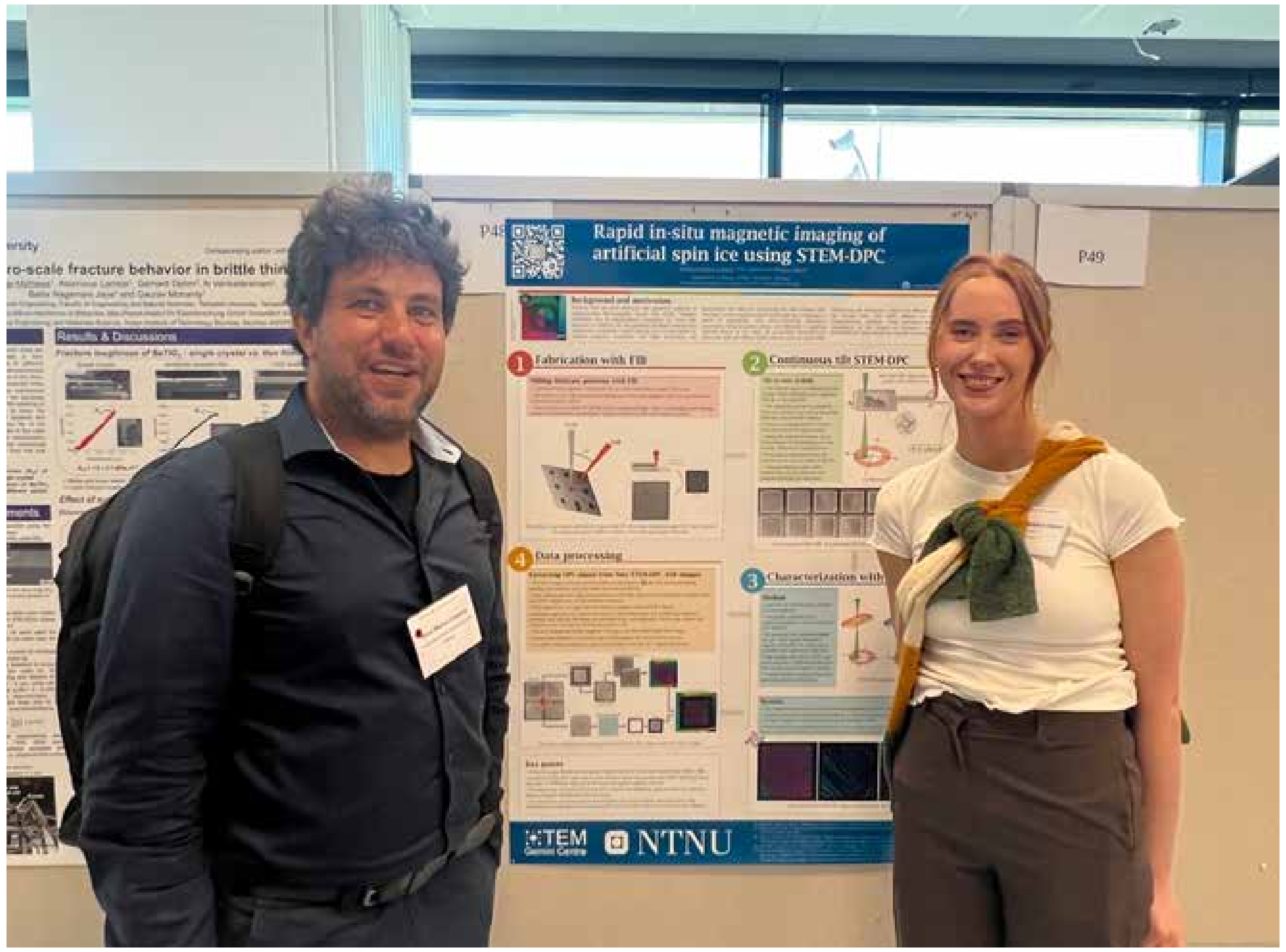

Studying TiAl3 particles in welded aluminium
Background
Results
Experimental methods






M&M 2023
MICROSCOPY & MICROANALYSIS
MSA


Acquisition and Processing of Magnetic Data from LN2 Cooled Perovskite Thin Films Using STEM-DPC
Introduction
Sample
Experimental
Results
Summary


Rapid in-situ magnetic imaging of artificial spin ice using STEM-DPC
Fabrication with FIB
Continuous tilt STEM-DPC
Data processing
TEM Gemini Centre
NTNU
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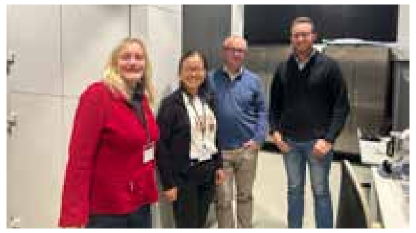

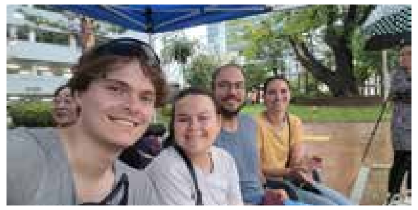

Publications 2023

JOURNAL PUBLICATIONS 2023

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CONTRIBUTIONS TO OPEN-SOURCE CODE

Various group members, from project students to academics, have actively contributed to open-source packages dedicated to electron microscopy data handling. Some contributions are listed below:

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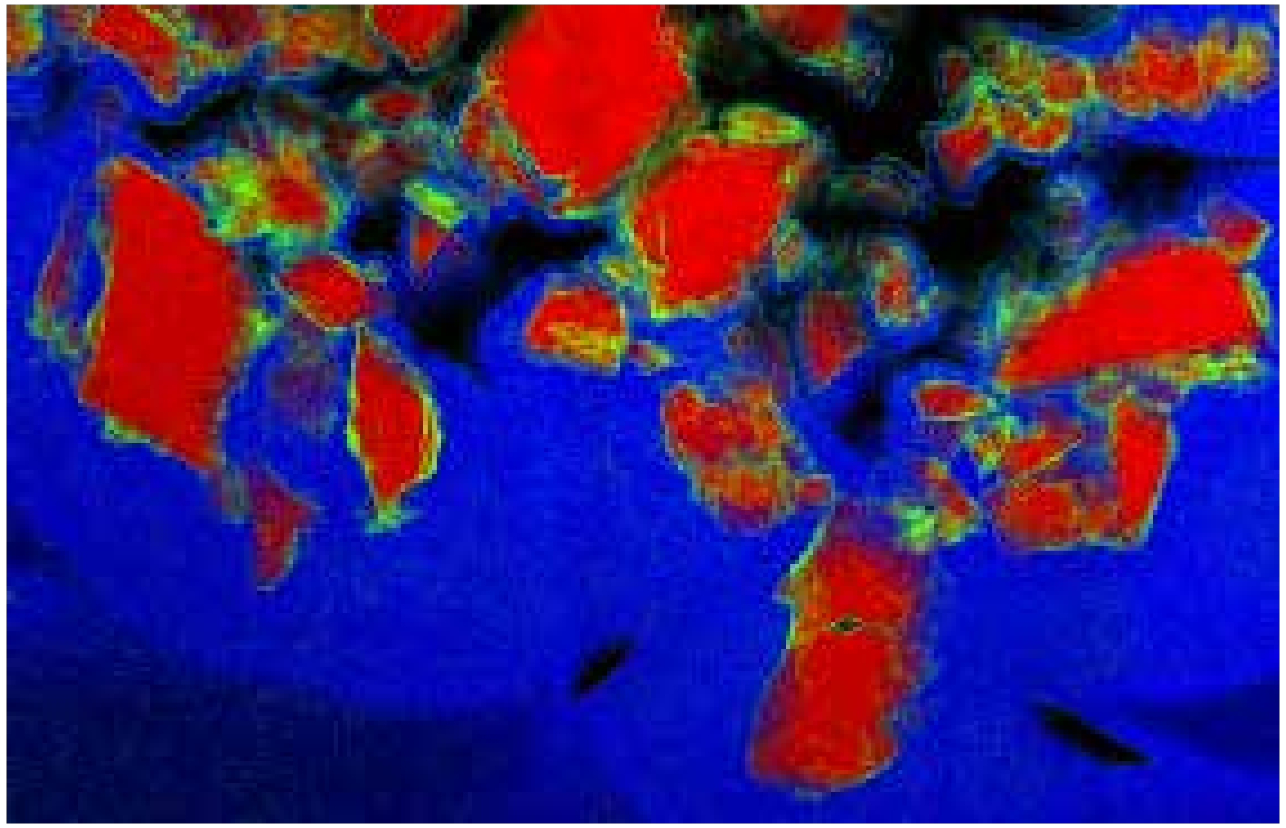

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- Kristin Frøystein, Scanning precession electron diffraction data analysis of in-situ precipitate evolution in an Al-Mg-Si-Cu alloy, January 2023 (Supervisor Randi Holmestad).
- Susanne Aspen Boucher, Characterization of perovskite oxide superlattices using HRTEM, STEM-EELS, 4D-HOLZ-STEM and Segmented SPED, June 2023 (Supervisor Magnus Nord).
- Håkon Longva Korsvold, Investigating the precipitates in the heat affected zone of a HYB welded Al-Mg-Si-Cu alloy using transmission electron microscopy, June 2023 (Supervisor Randi Holmestad).
- Anders Christian Mathisen, Scanning precession electron diffraction of ferroelectric polycrystalline h-$ErMnO_3$, June 2023 (Supervisor A.T.J. van Helvoort).
- Brynjar Mæhlum, Finding performance parameters and implementing bulk corrections for SEM EDS data in open-source software, June 2023 (Supervisor A.T.J. van Helvoort).
- Simen Skurdal, Characterization of Aluminium TiC nano-composite filler wire inoculated weld metal, June 2023 (Supervisor Jens K. Werenskiold).
- Hedda Christine Soland, Using STEM-DPC with a Conventional ADF Detector to Explore FIB Patterned Artificial Spin Ice Structures in Permalloy, June 2023 (Supervisor Magnus Nord).
- Mats Topstad, Charged ferroelectric domain walls in $K_2MgWP_2O_{10}$ and $K_3Nb_3B_2O_{12}$ studied by transmission electron microscopy, June 2023 (Supervisor A.T.J. van Helvoort).
- Kristian Tveitstøl, Using EELS to measure the local conductivity in Aluminium, June 2023 (Supervisor Randi Holmestad).
- Supreet Kaur: TEM Characterization of Strontium Barium Niobate (SrxBa1-xNb2O6) thin films, August 2023 (Supervisor Randi Holmestad).

PROJECT THESES

- Kaja Eggen Aune, Orientation mapping of next-generation Li-ion battery cathode material, December 2023 (Supervisor Ruben Bjørge).
- Thea Marie Dale, A structural characterization using TEM of a BiTe thin film grown on SrTiO3(111), December 2023 (Supervisor Magnus Nord).
- Viljar Johan Femoen, Relating holder axes and template matching for grain orientation analysis, December 2023 (Supervisor A.T.J. van Helvoort).
- Håvard Holm Fyhn, Phase and orientation analysis of illite-muscovite by scanning precession electron diffraction and template matching, December 2023 (Supervisor A.T.J. van Helvoort).
- Evy Gjedrem, Calculations of energy barriers for atomic migrations in Al-Sc alloys, December 2023 (Supervisor Jonas Frafjord).
- Espen J. Gregory, The origin of the double hardness peak in a Cu-containing 6082 aluminium alloy, December 2023 (Supervisor Randi Holmestad).
- Olav Hellebust Haaland, Orientation mapping of polycrystalline crystals by using kinematical diffraction theory and template matching of scanning precession electron diffraction data, January 2024 (Supervisor A.T.J. van Helvoort).
- Sindre Vie Jørgensen, Characterization of Ce-phases in Cast Austenitic Stainless Steel, December 2023 (Supervisor Randi Holmestad).
- Inga Dahlen Konow, Electron Microscopy on Jet Milled Silicon Powders, December 2023 (Supervisor Randi Holmestad).
- Peder Stokkan, Creating a Protocol for Performing an In Situ Heating Experiment on FIB Patterned Artificial Spin Ice Structures using TEM, January 2024 (Supervisor Magnus Nord).
- Aurora Teien, 3D electron diffraction tomography: From electron diffraction tomo datasets to detailed structural information, December 2023 (Supervisor A.T.J. van Helvoort).

ACTIVE PROJECTS IN TEM GEMINI CENTRE IN 2023

The table below shows the larger projects connected to TEM within the Gemini Centre. They are listed by project type, title and research partners and duration. Smaller projects, both academic and with direct industrial support, are not listed and run in parallel. In total the Centre had 81 different projects using the facilities in 2023.

Project type	Prosjekt title	Involved with TEM	Duration
SFI	SFI PhysMet (Centre for Sustainable and Competitive Metallurgical and Manufacturing Industry) Partners: NTNU, SINTEF, Statens vegvesen, Forsvarsbygg, Hydro, Elkem, Neuman Aluminium, Equinor, Benteler, ThermoCalc Software	~3 PhDs NTNU, SINTEF	2020-28
SFI	SFI CASA Centre for Advanced Structural Analysis 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	1-2 PhDs NTNU, SINTEF	2015-23
SFI	SFI Manufacturing Partners: SINTEF, NTNU, Benteler, Brødrene AA, Ekornes, GKN Aerospace, Hexagon composites, Kongsberg Automotive, Nammo, Raufoss Neuman, Plastal, Plasto, Rolls Royce, Teeness, Hybond, Hydro	1 PhD NTNU, 1 postdoc NTNU, SINTEF	2015-23
SFI	SFI iCSI - Industrial Catalysis Science and Innovation for a Competitive and Sustainable process Industry. Partners: Yara Norge, K.A. Rasmussen, Dynea INOVYN Norge, Haldor Topsøe AS	1 postdoc NTNU, SINTEF	2015-23
FME	SuSolTech – The Research Center for Sustainable Solar Cell Technology Partners: IFE, NTNU, SINTEF, University of Oslo (UiO), CleanSi, Dynatec, Elkem Solar, Mosaic, Norsun, Norwegian Crystals, Quartz Corp, REC Silicon, REC Solar, Semilab	1 PhD, NTNU, SINTEF, UiO	2017-25
FME	Mobility Zero Emission Energy Systems - MoZEES Partners: 7 research institutions including both SINTEF and NTNU, 7 public bodies, 26 industrial partners	SINTEF, 1 postdoc	2015-23
KPN/BIA	SumAl - Solute cluster manipulation for optimized properties in Al-Mg-Si based Al alloys. Partners: NTNU, SINTEF, Hydro, Benteler, Neuman, Speira	1 PhD, 1 Postdoc, SINTEF	2019-24

Project type	Prosjekt title	Involved with TEM	Duration
Research project/ Nano2021	In-Sane - In-situ studies of highly conductive bonded interfaces between aluminium and copper at the nanoscale	1 PhD NTNU, SINTEF	2020-24
FRIPRO	QUATRIX - Quasicrystal nucleation in a metallic matrix	SINTEF, 1 PhD NTNU	2021-25
FRINATEK	In-situ correlated nanoscale imaging of magnetic fields in functional materials (InCoMa)	2 PhD NTNU	2021-25
NTNU Nano Enabling technologies	Enabling nanoscale in-operando studies of advanced epitaxial functional thin film materials using nanofabrication	1 PhD, NTNU	2022-2026
INTPART	Norwegian-Japanese Aluminium alloy Research and Education Collaboration (NJALC) – II. Partners: NTNU, SINTEF, Hydro, University of Toyama, Tokyo Institute of Technology	NTNU, SINTEF, Travel, exchange students	2019-23
IPN	SIDI - Sustainable graphite Inoculants for Ductile cast Iron, Partners: Elkem, SINTEF, NTNU	Engineer time NTNU, SINTEF	
IPN/ENERGIX	UVC LEDs based on nanowires-on-graphene: Partners; CrayoNano, SINTEF, NTNU	SINTEF	2022-24
IPN	Catch & Kill. Partners: SINTEF, Standard Bio, USN, Uni. New South Wales	SINTEF	2020-23
IPN	IPN Coulombus	SINTEF	2022-23
EU/H2020	ESTEEM3 - https://www.esteem3.eu/	1 postdoc NTNU, prof Ils	2019-23
EU/H2020	SAFE-N-MEDTECH	SINTEF	2019-23
EU/H2020	Hydra, Partners: SINTEF, Uppsala University, CEA, UCL, FAAM, DLR, ISCI, Solvionic, Corvus, POLITO, Elkem, Johnsen Matthey	SINTEF	2020-24
EU/HEU	MatCHMaker, Partners: CEA, RINA-C, CSM, SINTEF, AIMEN, SIMAVI, HeidelbergCement, GENVIA, Toyota, TU Wien, ASRO.	SINTEF	2022-26
EU/HEU	IMPRESS - Interoperable electron Microscopy Platform for advanced RESearch and Services.	1 PhD	2023-2027
Green platform	AluGreen, Partners: Hydro, SINTEF, NTNU, Oshaug, Metall, Christie & Opsahl, Overhalla, Betongbygg, Norcable, Corvus Energy, Ocean Sun, Kodyna, Leirvik, Nexans,Benteler, Metallco Aluminium, Prodtex, Statnett SF Nordic Office Of Architecture Dr Techn Olav Olsen	SINTEF	2021-24
Green platform	SUMBAT, Partners: SINTEF, NTNU, UiO, UiA, IFE, Freyr, Vianode, Elkem, Morrow, Norsk Hydro, Corvus	SINTEF	2022-24

Selected Papers

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Materials Science & Engineering A

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Local mechanical properties and precipitation inhomogeneity in large-grained Al–Mg–Si alloy

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Al–Mg–Si (6xxx series) alloys show excellent mechanical properties due to the precipitates formed during heat treatment. However, heat treatment of these alloys results in a soft precipitation free zone (PFZ) close to grain boundaries that weakens them and promotes fracture, and thereby reduces the ductility of the material. This study provides quantitative insights into the mechanical properties and underlying plasticity behavior of Al–Mg–Si (6xxx series) alloys through combined nanoindentation hardness measurements and in-depth characterization of the microstructure adjacent to the PFZ region and in the grain interior. Experimental nanoindentation, transmission microscopy (TEM) and electron channeling contrast imaging results confirm the weakening effect from PFZ by means of a reduced hardness close to grain boundaries. The nanoindentation hardness mapping also revealed an increase in hardness a few micrometers from the grain boundary with respect to the grain interior. Precipitate quantification from TEM images confirms that the hardness increase is caused by a locally higher density of precipitates. To the authors' best knowledge, this harder zone has not been recognized nor discussed in previously reported findings. The phenomenon has important implications for the mechanical properties of large-grained (>100 µm) aluminium alloys.

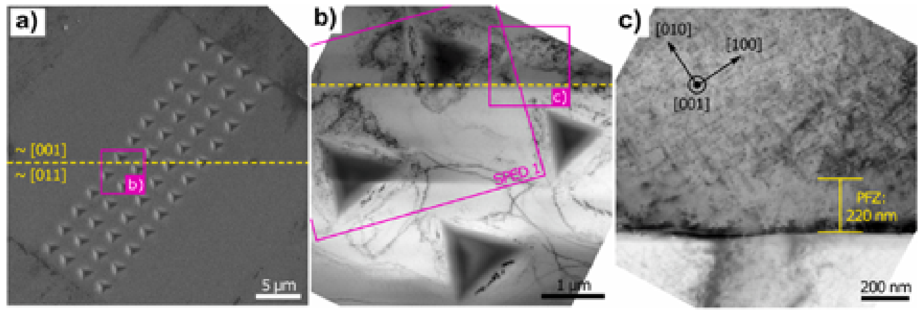

Fig. 8. TEM overview of a 4 × 12 nanoindentation array around GB1 in T6AC. (a) SEM image of the array. (b) Bright-field TEM image of four indents close to GB1. (c) Bright-field image of precipitate microstructure close to GB1, in a [001] orientation.

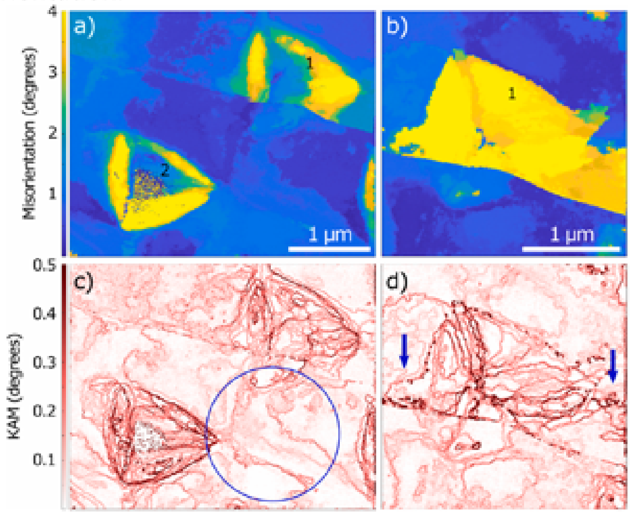

Fig. 9. Results from SPED analysis of T6AC. (a,c) Map with two indentations close to GB1. (b,d) Map from indentation 1, in a [001] orientation. (a–b) Angle of misorientation from the average value in each grain. (c–d) Kernel average misorientation. The blue circle highlight strain fields in-between indentations and the blue arrows point towards abrupt orientation changes between indentation and GB.

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RESEARCH ARTICLE

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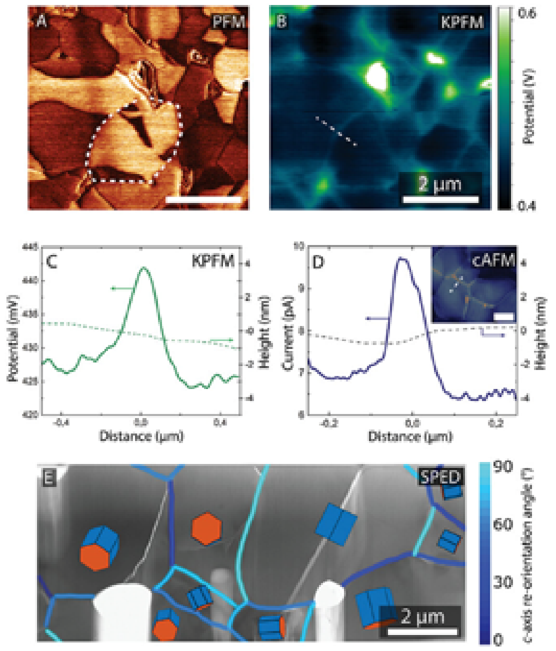
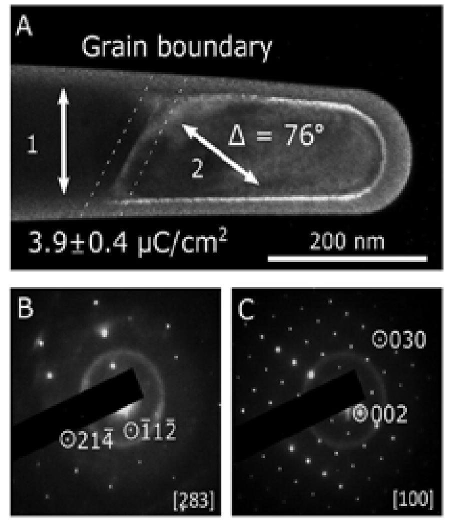
Quantitative Mapping of Chemical Defects at Charged Grain Boundaries in a Ferroelectric Oxide

Kasper A. Hunnestad, Jan Schultheiß, Anders C. Mathisen, Ivan N. Ushakov, Constantinos Hatzoglou, Antonius T. J. van Helvoort, and Dennis Meier**

Polar discontinuities, as well as compositional and structural changes at oxideinterfaces can give rise to a large variety of electronic and ionic phenomena.In contrast to earlier work focused on domain walls and epitaxial systems,this work investigates the relation between polar discontinuities and the localchemistry at grain boundaries in polycrystalline ferroelectric ErMnO3 . Usingorientation mapping and scanning probe microscopy (SPM) techniques, thepolycrystalline material is demonstrated to develop charged grain boundarieswith enhanced electronic conductance. By performing atom probetomography (APT) measurements, an enrichment of erbium and a depletionof oxygen at all grain boundaries are found. The observed compositionalchanges translate into a charge that exceeds possible polarization-driveneffects, demonstrating that structural phenomena rather than electrostaticsdetermine the local chemical composition and related changes in theelectronic transport behavior. The study shows that the charged grainboundaries behave distinctly different from charged domain walls, givingadditional opportunities for property engineering at polar oxide interfaces.



Microstructural characterization. A) Out-of-plane PFM image, displaying pronounced bright and dark contrast consistent with ferroelectric 180° domains as discussed in ref. [24]. The dashed line highlights the boundary of a representative grain. B) KPFM map of the same area as shown in (A), where grain boundaries display a brighter contrast (higher potential) than the bulk. A profile across one grain boundary is extracted along the dashed line and shown in (C), along with the topography. D) A representative cAFM profile and map (inset) of a grain boundary along with the topography. Scale bar: 500 nm. E) SPED is used to obtain the crystal orientation of the grains, as represented by a hexagonal prism with the ferroelectric polarization lying along the hexagonal *c*-axis. The calculated *c*-axis re-orientation angle between neighboring grains is indicated by different shades of blue.



Grain boundary extraction and analysis. A) DF-TEM of an APT needle showing the grain boundary configuration. The crystal orientation of the two grains is identified via the selected-area electron diffraction patterns in (B) and (C), with the viewing direction oriented to keep grain 2 on the (100) zone axis and the 2D projected polarization directions drawn in with white arrows. The resulting change in *c*-axis orientation across the grain boundary is about 76°, corresponding to a bound charge $|\rho| = 3.9 \pm 0.4\ \mu C\ cm^{-2}$. For the DF-TEM image in (A), the 002 reflection in (C) was used.

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Original Article

The evolution of precipitates in an Al–Zn–Mg alloy

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The precipitation sequence in Al–Zn–Mg alloys has been subject to many revisions over the years as more structural details of the early-stage Guinier-Preston (GP) zones and precipitates have been uncovered. To further investigate this, a 7003 aluminium alloy naturally aged for one year was subjected to artificial ageing at 140 °C to investigate the evolution of early-stage precipitates. STEM was coupled with APT and hardness measurements to characterise the precipitate structure and chemistry of the alloy at different stages in the heat treatment. The naturally aged condition contained a dense population of GPI zones, co-existing with a smaller distribution of η' precipitates. The number densities of clusters were similar in the two conditions, while the average size of the clusters had increased. The average Zn/Mg ratio of the clusters decreased, and the clusters no longer exhibited the GPI zone atomic structure, indicating that the GPI zones had dissolved. Concurrently, the fraction of precipitates having η' structure increased.

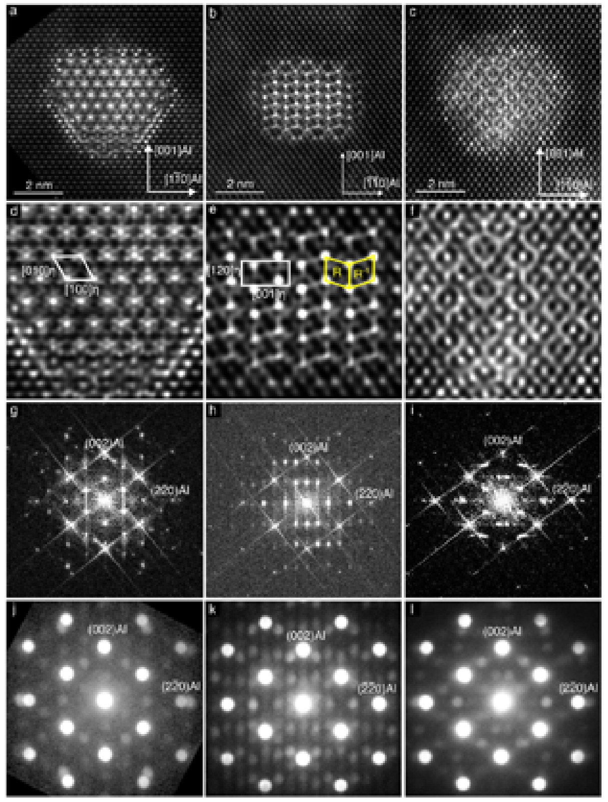

Fig. 8. Precipitates in the peak condition viewed along $\langle 110 \rangle_{Al}$. a: η_1 precipitate viewed along $[001]_\eta$, b: η_1 viewed along $[100]_\eta$ and c: T′ phase. d, e and f: Enlarged, FFT filtered regions of the HAADF-STEM images in a, b and c, respectively. g, h and i: FFTs of a, b and c, respectively. j, k and l: PED patterns exhibiting a good correspondence with the FFTs in g, h and i, respectively. Rhomobohedral and vertically mirrored rhomobohedral layers are indicated with R and R^{-1}, respectively.

MATERIALS TRANSACTIONS

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Special Issue on Aluminium and Its Alloys for Zero Carbon Society, ICAA 18

Intermetallic Phase Layers in Cold Metal Transfer Aluminium-Steel Welds with an Al–Si–Mn Filler Alloy

Tina Bergh, Håkon Wiik Ånes, Ragnhild Aune, Sigurd Wenner, Randi Holmestad, Xiaobo Ren, Per Erik Vullum

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Keywords: aluminium-steel welds, cold metal transfer, intermetallic phases, transmission electron microscopy, electron backscatter diffraction

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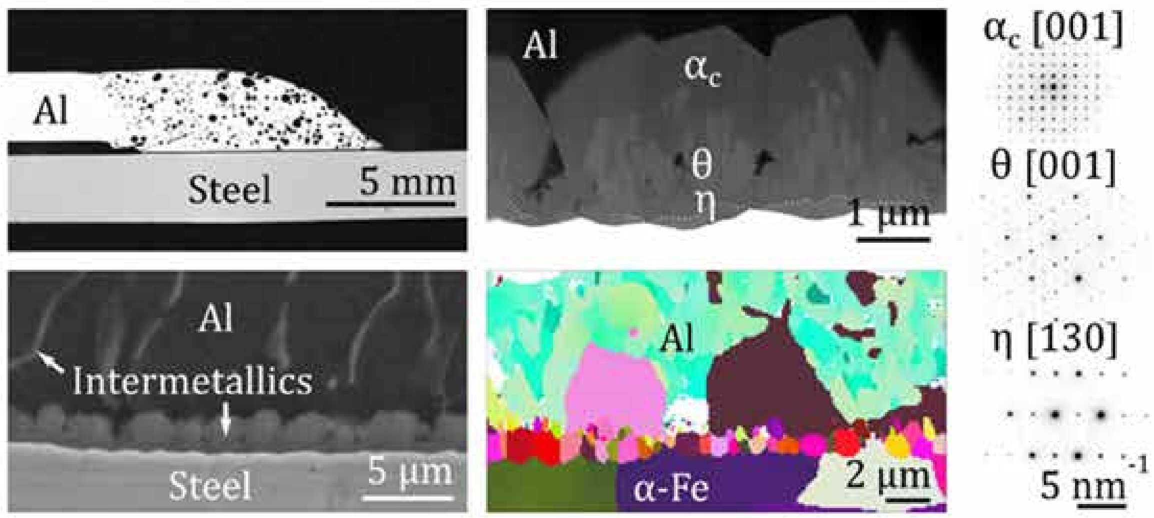
In welding of aluminium (Al) alloys to steels, a major challenge is excessive growth of brittle intermetallic phases along the bonded Al-steel interfaces. The formation and growth of these phases are influenced by the heat input and the alloying elements present. This work focuses on the phases formed between a low alloyed steel and an Al–Si–Mn alloy that was used as the filler wire in a cold metal transfer joint. During lap shear testing of the joint, fracture ran through the melted Al, and the joint reached a strength of 174 ± 21 MPa. Scanning and transmission electron microscopy showed that the formed ~2.5 µm thick intermetallic phase layer consisted of polyhedral α_c-Al–(Fe,Mn)–Si, elongated or rounded θ-Fe_4Al_{13} and near equiaxed η-Fe_2Al_5 grains. Electron backscatter diffraction was used to study the crystal orientations of the formed phases. Altogether this work aims to contribute to better understanding of the formation and growth of intermetallic phases in Al-steel welds where the Al alloy contains Si and Mn.



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Correcting for probe wandering by precession path segmentation

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Precession electron diffraction has in the past few decades become a powerful technique for structure solving, strain analysis, and orientation mapping, to name a few. One of the benefits of precessing the electron beam, is increased reciprocal space resolution, albeit at a loss of spatial resolution due to an effect referred to as 'probe wandering'. Here, a new methodology of precession path segmentation is presented to counteract this effect and increase the resolution in reconstructed virtual images from scanning precession electron diffraction data. By utilizing fast pixelated electron detector technology, multiple frames are recorded for each azimuthal rotation of the beam, allowing for the probe wandering to be corrected in post-acquisition processing. Not only is there an apparent increase in the resolution of the reconstructed images, but probe wandering due to instrument misalignment is reduced, potentially easing an already difficult alignment procedure.

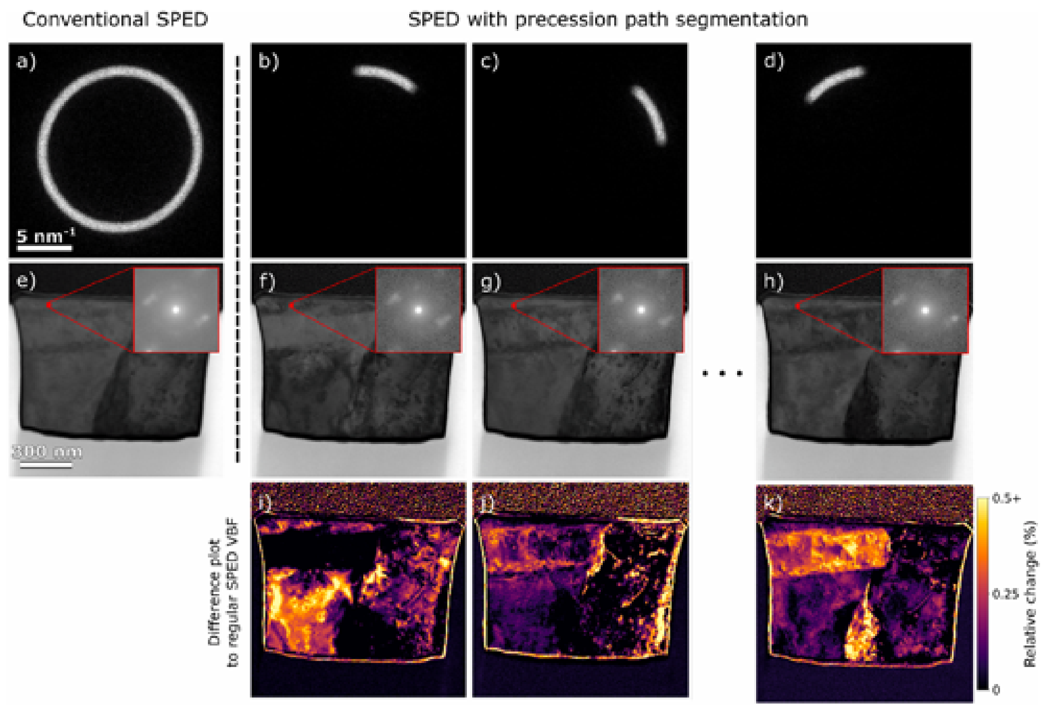

Fig. 1. A comparison between a regular SPED scan, and a precession path segmented SPED scan with n=8 segments. Example of diffraction patterns with de-rocking switched off for (a) the regular SPED scan, and (b)–(d) three precession path segments. VBF images of a (e) regular SPED scan, and (f)–(h) three segments. Insets show diffraction patterns for 4 × 4 selected scan points in the VBF images. VBF intensity difference plots between each of the presented segment VBF images in (f)–(h) compared to the regular SPED VBF in (e) are shown in (i)–(k). The diffraction patterns in (a)–(d) are from a different precession path segmentation scan than the VBF images and difference plots in (e)–(k).

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Focused ion beam lithography for position-controlled nanowire growth

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To exploit the promising properties of semiconductor nanowires and ensure the uniformity required to achieve device integration, their position on the growth substrate must be controlled. This work demonstrates the direct patterning of a SiO_2/Si substrate using focused ion beam (FIB) patterning to control self-catalyzed GaAsSb nanowire growth in molecular beam epitaxy (MBE). Besides position control, FIB patterning parameters influence nanowire yield, composition and structure. Total ion dose per hole is found to be the most important parameter. Yield of single nanowires ranges from ≈34% to ≈83%, with larger holes dominated by multiple nanowires per hole. Areas exposed to low ion beam doses are selectively etched by routine pre-MBE HF cleaning, enabling patterning and nanowire nucleation with minimal damage to the Si substrate. The optical and electronic properties of nanowires are found to depend on the ion dose used during patterning, indicating the potential for FIB patterning to tune nanowire properties. These findings demonstrate the possibility for a FIB lithography protocol which could provide a rapid and direct patterning process for flexible controlled nanowire growth.

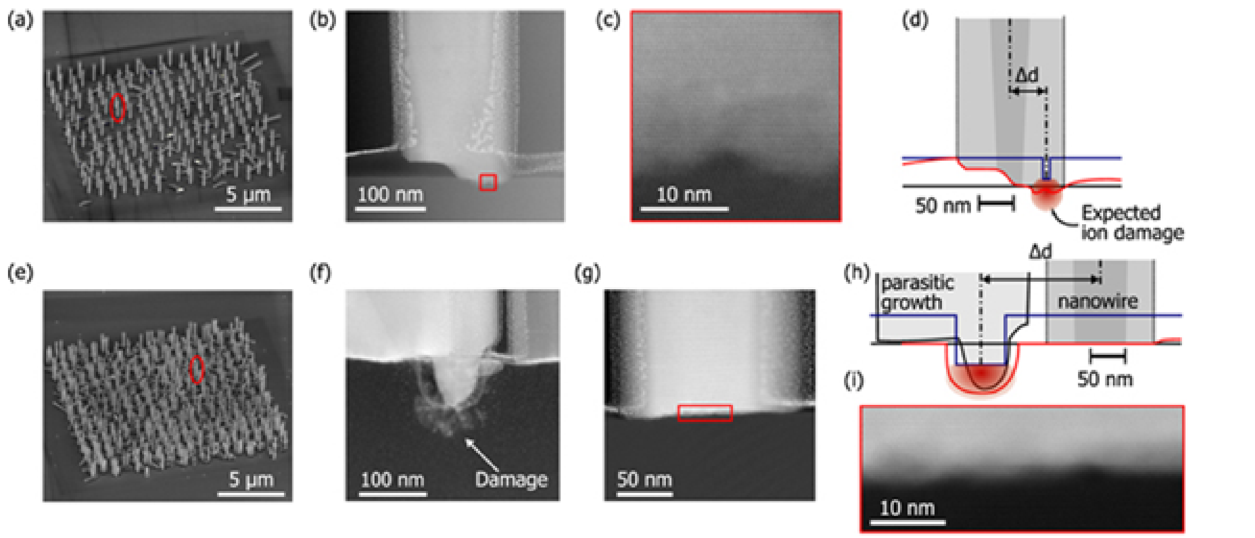

Figure 2. Ion-beam dependent nanowire-substrate interfaces for different milling parameters. TEM lamella specimens taken from the fields marked by white squares in figure 1(a) (bottom-left and top-right in the 8 × 8 growth matrix): (a)–(d) SEM, HAADF-STEM and schematic of nanowire from representative low-dose array. (e)–(i) SEM, HAADF-STEM and schematic from representative high-dose array. Nanowires in (b) and (g) are marked with red circles in (a) and (e), respectively. High-resolution HAADF-STEM of their interfaces at the marked red rectangles is presented in (c) and (i). In the schematic presentations (d) and (h), the solid horizontal dark blue line marks the location and shape of FIB milling while the red line marks the final observed interface after HF etching, annealing and nanowire growth. Red areas mark the expected extent of ion implantation from simulation. The distance Δd between dash-dot lines denote the nanowire displacement (distance between targeted hole center and nanowire center axes).