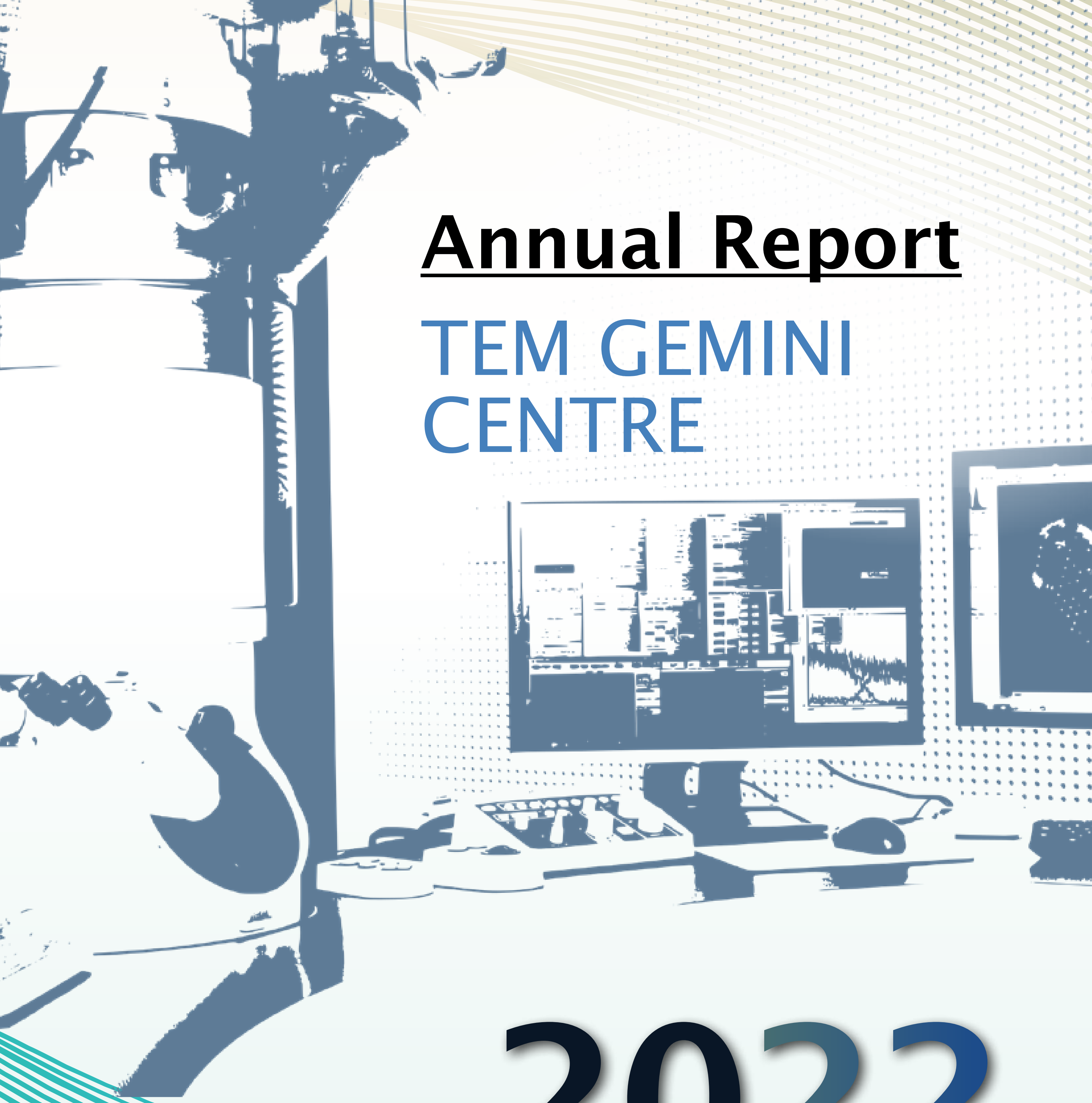
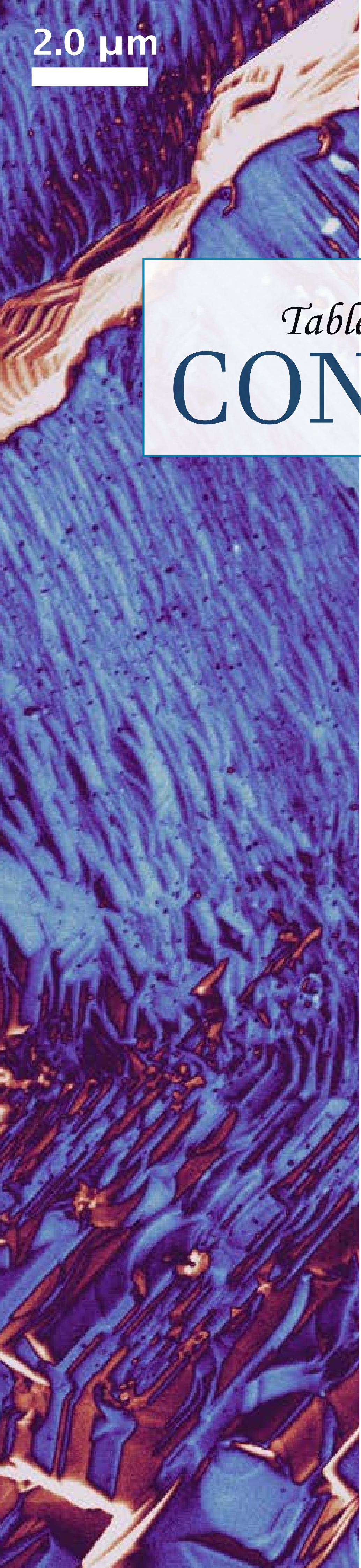


Annual Report

TEM GEMINI CENTRE

2022

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



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Graphic design:
Jørgen A. Sørhaug & Marthe Linnerud.
All images (including TEM images), in this report are taken by members at the TEM Gemini Centre.

***Images on both pages**: surface of Ag, by T. Bergh.*


2.0 µm

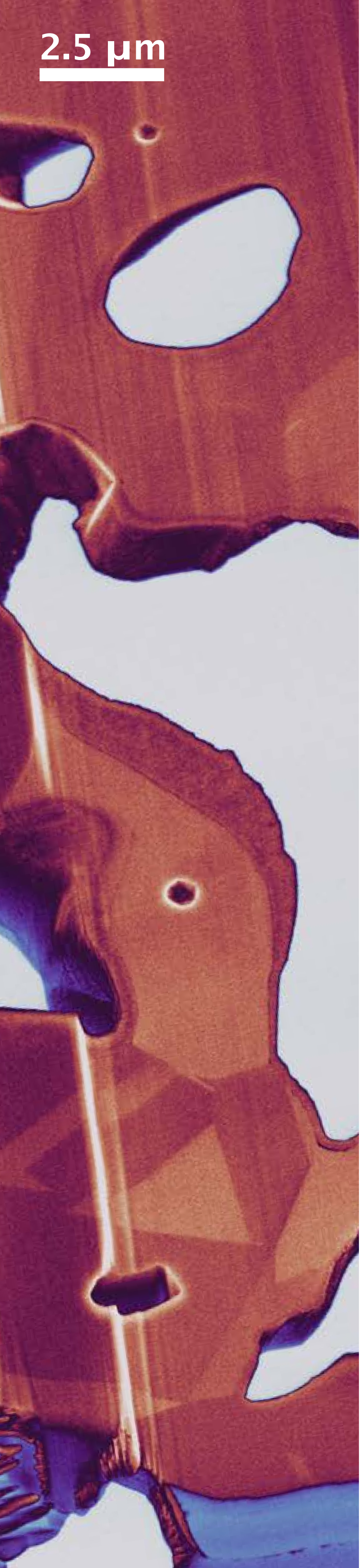


Image: *FIB-prepared TEM sample of Ag, by T. Bergh.*

INTRODUCTION

2022 has been a good year for the TEM Gemini Centre. 🙂 Again, we see that the structural and strategic efforts in the Centre enable users to perform quality work efficiently and fast. Together, NTNU and SINTEF have collaborated 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 the highest ever, and we have started the work to get a new microscope into the facility. 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, and the NORTEM consortium is now in a tender process to secure access to state-of-the-art instrumentation in the years to come. 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 is in no doubt crucial in this important task. This grant is the result of a 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 infrastructure that was financed by the RCN and the partners in 2011 has been working under 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 the use of the three instruments increasing steadily, (but with high variations over the years) and the resulting high scientific throughput, we recognize the importance of strategic collaboration. We updated our strategy document, now running 2023-2026. 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. Continuous reinvestments are crucial and through an internal NTNU grant, two new detectors have been installed on the top instrument in 2022.

In 2022 we received more visitors, after the pandemic in which travel was more restricted. However, in the last years the Centre has still facilitated transnational access to TEM through its participation in the ESTEEM3 EU Horizon 2020 network with several other world-leading TEM laboratories. This has increased the visibility in Europe in a time where conferences and workshops have been cancelled or changed to less interactive formats. The Gemini Centre participates in a broad range of projects, including national, public, industrial and EU funded ones. The Centre is involved in four long-term SFI projects – Centre of advanced structural studies (CASA), Sustainable innovations for automated manufacturing of multi-material products (SFI-Manufacturing), Industrial catalysis science and innovation (iCSI), and the 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. As a part of the INTPART project with Japanese aluminium industry and academia, we have had 3 Japanese exchange students visiting in the fall. It is good to see that international collaborations again rise after the pandemic.

We organized a very successful physical workshop on electron diffraction in June 2022 with 26 participants from 8 European countries. In September we had a big celebration in Trondheim for the 2020 and 2022 Kavli laureates in nanoscience and neuroscience. Harald Rose, Knut Urban, Max Haider and Ondrej Krivanek got the 2020 Kavli prize in nanoscience for their developments of aberration correction in transmission and scanning transmission electron microscopy. The prize ceremony in Oslo was postponed for two years because of the pandemic, and the winners came to Trondheim for the nano-symposium. After this they stayed one extra day in Trondheim to attend a seminar in the TEM Gemini Centre. This is described elsewhere in this report.

In October we were renominated as a Gemini Centre for four new years. As documented in this report, the Centre had 47 active hands-on users/operators, 9 users through operators and served 96 different projects, whose results have contributed to 62 journal publications (plus 5 in press/preprints) in 2022. 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 and 11 MSc candidates were educated with TEM as a substantial part of their theses in 2022. The TEM lab activity in scheduled courses is taken up again, and 5 courses used TEM in lab exercises last year. The annual TEM introduction course has continued in digital form and is available on the net. 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 2022. For more details, see our home page.

TEM Gemini Centre management,
February 2023.

Prof. Erik Walström

Inga Ringdalen

Prof. Ida Westermann

Prof. Randi Holmestad

Prof. Antonius T. J. van Helvoort

Bjørn Gunnar Soleim

Emil Frang Christiansen

Prof. Yanjun Li

Assoc. prof. Per Erik Vullum

Ruben Bjørge

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
- Yanjun Li, Materials Science and Engineering, NTNU
- Per Erik Vullum, Materials Physics, SINTEF Industry
- Ruben Bjørge, Materials Physics, SINTEF Industry

Image on next page: *Group picture of members of the TGC, by I.-E. Nylund.*

PEOPLE IN THE TEM GEMINI CENTRE IN 2022

- Sigmund J. Andersen (Senior research scientist, SINTEF)
- Rajith Aravinth (Master student, DP, NTNU)
- Julie Marie Bekkevold (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)
- Torbjørn Bogen-Storø (Master student, DP, NTNU)
- Joseph Vincent Broussard (Master student, DP, NTNU)
- Dipanwita Chatterjee (Postdoc, DP, NTNU)
- Emil Frang Christiansen (Senior Engineer, DP, NTNU)
- Sivert Johan Vartdal Dagenborg (PhD student, DP, NTNU)
- Jonas Frafjord (Postdoc, DP, NTNU)
- Jesper Friis (Senior research scientist, SINTEF)
- Kristin Frøystein (Master student, DP, NTNU)
- Erik Gaupseth (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)
- Supreet Kaur (Master student, DP, NTNU)
- Håkon Longva Korsvold (Master student, DP, NTNU)
- Petter Lervik (Master student, DP, NTNU)
- Martin Bakken Lesjø (Master student, DMSE, NTNU)
- Yanjun Li (Prof., DMSE, NTNU)
- Marthe Linnerud (Master Student/PhD student, DP, NTNU)
- Ursula Ludacka (Postdoc, DMSE, NTNU)
- Hogne Lysne (PhD student, DP, NTNU)
- Calin D. Marioara (Senior research scientist, SINTEF)
- Knut Marthinsen (Prof., DMSE, NTNU)
- Anders Christian Mathisen (Master student, DP, NTNU)
- Magnus Nord (Assoc. Prof. DP, NTNU)
- Gregory Nordahl (PhD student, DP, NTNU)
- Brynjar Morka Mæhlum (Master student, DP, NTNU)
- Inger-Emma Nylund (PhD student /Postdoc, DMSE, NTNU)
- Ding Peng (Postdoc, DP, NTNU)
- Andreas Rosnes (Master student, DP, NTNU)
- Oskar Ryggetangen (PhD student, DP, NTNU)
- Armand Sepehri (Master student, DP, NTNU)
- Mari Sofie Skomedal (Master student, DP, NTNU)
- Bjørn Gunnar Soleim (Senior engineer, DP, NTNU)
- Hedda Christine Soland (Master student, DP, NTNU)
- Jørgen Sørhaug (PhD student, DP, NTNU)
- Tor Inge Thorsen (PhD student, DP, NTNU)
- Elisabeth Savitri Thrane (Master student, DP, NTNU)
- Elisabeth Thronsen (PhD student/ Research Scientist, SINTEF, DP, NTNU)
- Kristian Tveitstøl (Master student, DP, NTNU)
- Per Erik Vullum (Senior research scientist, SINTEF and Assoc. Prof. II, DP, NTNU)
- Sigurd Wenner (Research scientist, SINTEF)
- Yingda Yu (Senior engineer, DMSE, NTNU)
- Iryna Zelenina (Postdoc, DP, NTNU)
- Hedda Øye (Master student, DP, NTNU)
- 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 nine 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 includes upgrades of existing infrastructure 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 direct detectors, 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. The tender was published in July 2022, and a competitive dialogue has been started with the vendors, with a plan to have a contract ready in June 2023. For more information on NORTEM see the NORTEM webpages.

The Trondheim node of the NORTEM facility has two senior engineers, Bjørn Gunnar Soleim and Emil Frang Christiansen supporting maintenance, training, competence, and techniques. The microscopes have a high uptime. Per Erik Vullum and Ruben Bjørge (from September) have been working as adjunct (affiliated) professors (20 %) at NTNU in 2022. This contributes to developing the interaction between NTNU and SINTEF.

Key numbers for NORTEM:

- 3 partners - SINTEF, NTNU and UiO;
- 2 nodes - Trondheim and Oslo;
- 58 MNOK from INFRA in 2011;
- New grant in 2021;
- 5 microscopes - 2 top level from 2012/2013;
- ~ 150 projects served annually;
- > 50 000 h used since 2012;
- ~ 60 users annually;
- ~ 60 papers annually (2/3 international co-authors);
- ~ 15 permanent staff in core research groups;

Image: *private concert for Kavli winners with family and guests. The concert was held at Ringve museum by Linus Nord-Varhaug.*

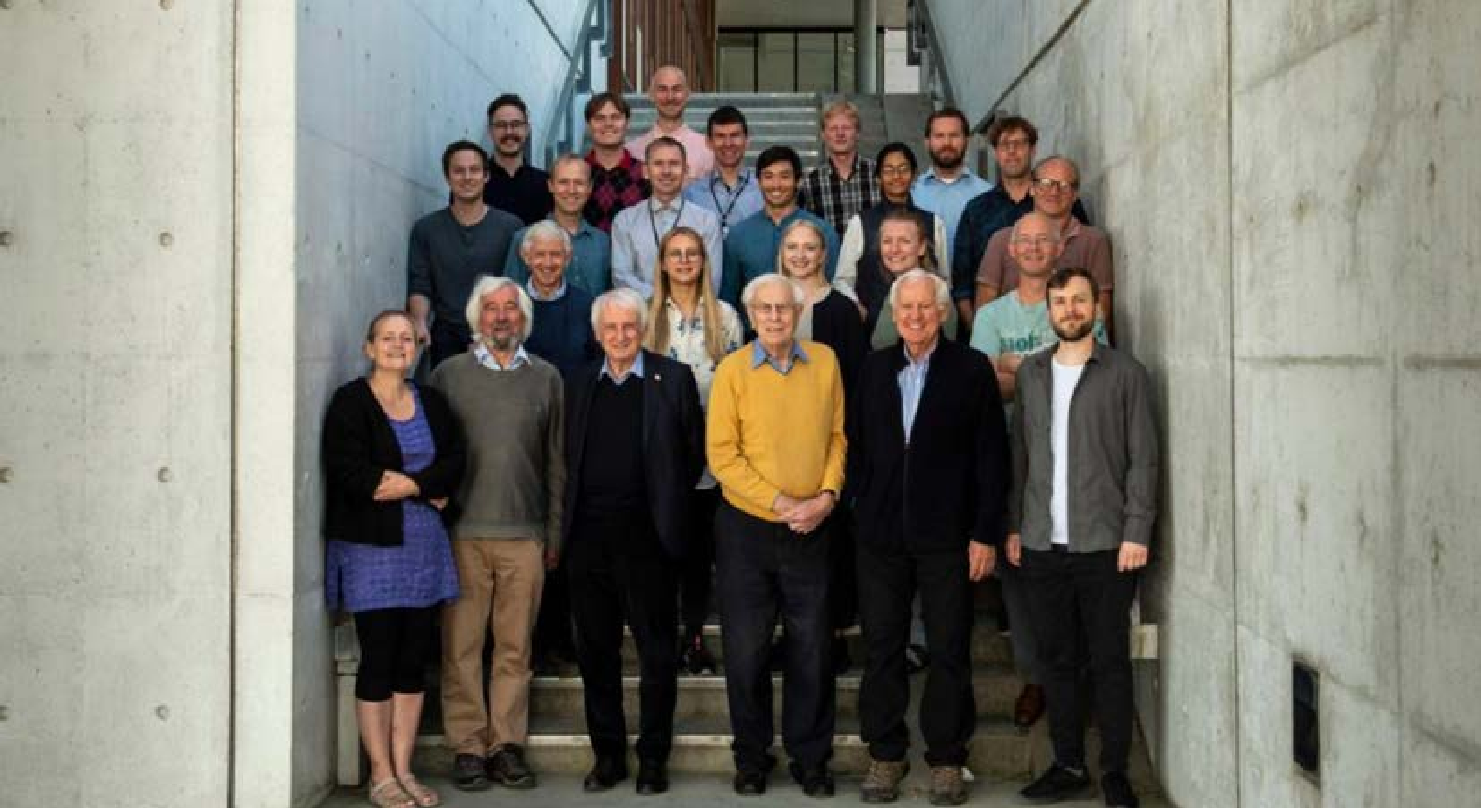


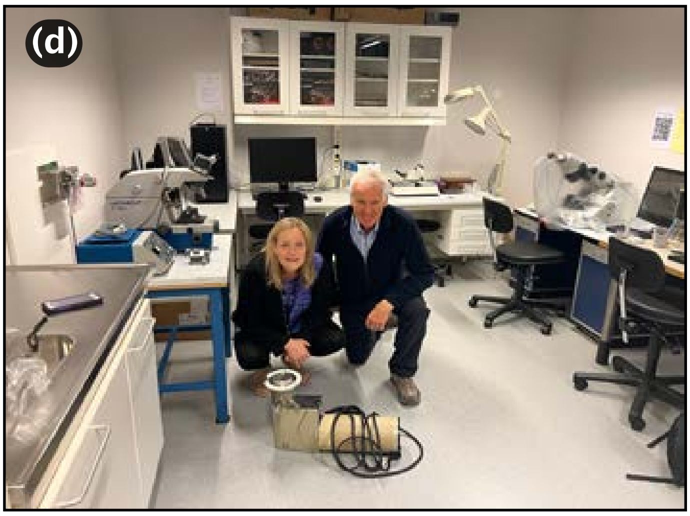
Image: *Group picture of members of the TGC and the Kavli price winners, by I.-E. Nylund.*

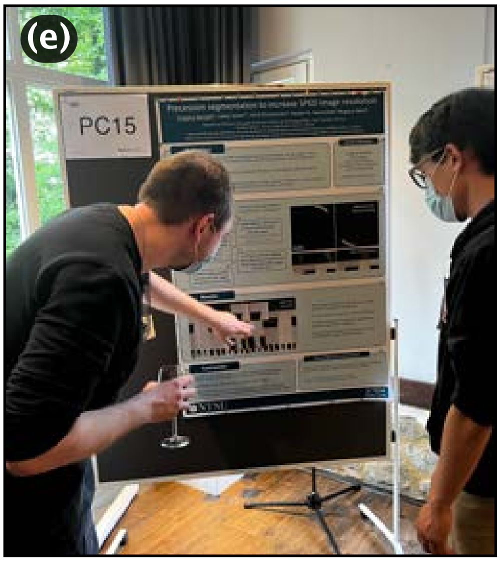
THE TEM GEMINI CENTRE

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 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.

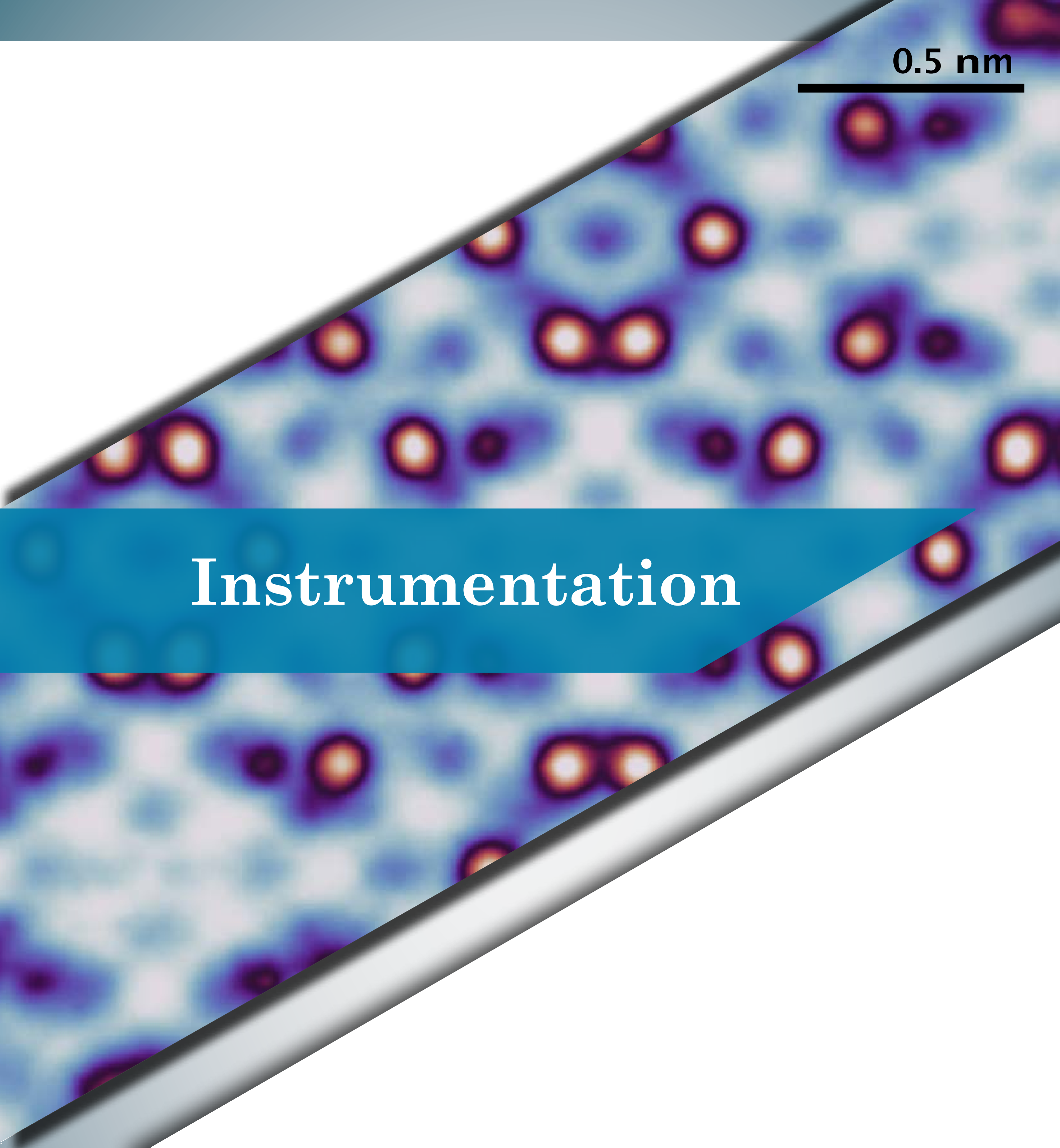
The Centre's research groups work within materials physics and materials science, studying a broad range of materials down to the nanometer and atomic level, where the main tool is the TEM. The overall objective of the TEM Gemini Centre is to build and secure a robust scientific environment within TEM 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 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 enables collaborating groups to grasp new opportunities to create values and improve 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 to work, 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.





Images above: (a) *Group picture of all participants on the ESTEEM3 electron diffraction workshop in Trondheim, June 2022, by I.-E. Nylund;* ***(b)*** *Our engineer Bjørn G. Soleim showing the Kavli winners our old TEM Phillips T400* ***(c)*** *Ton van Helvoort chatting with the Kavli winners at NTNU Gløshaugen;* ***(d)*** *Randi Holmestad and Ondrej Krivanek inspecting spectrometer Ondrej modified to one of the first GIFs in Trondheim 30 years ago;* ***(e)*** *Gregory Nordahl speaking about how his SPED segmentation method to enhance image resolution at the PICO conference;* ***(f)*** *Sivert Dagenborg presenting his work on improving STEM-DPC data analysis at the PICO conference;* ***(g)*** *Ton van Helvoort and Randi Holmestad at a rainy summer walk-and-talk;* ***(h)*** *Randi Holmestad, Knut Marthinsen and Paul Sanders visiting Elkem and Leander Michels in Kristiansand.*



Instrumentation

Image: *High-resolution STEM image of atoms, by E. Thronsen.*

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 atomic scale spectroscopy and imaging with CCD, CMOS and direct electron detectors. 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) with direct electron detection, 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
- Prefilter Gatan 4k RIO (in-situ)
- MerlinEM 4R Medipix direct electron detector from Quantum Detectors
- 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

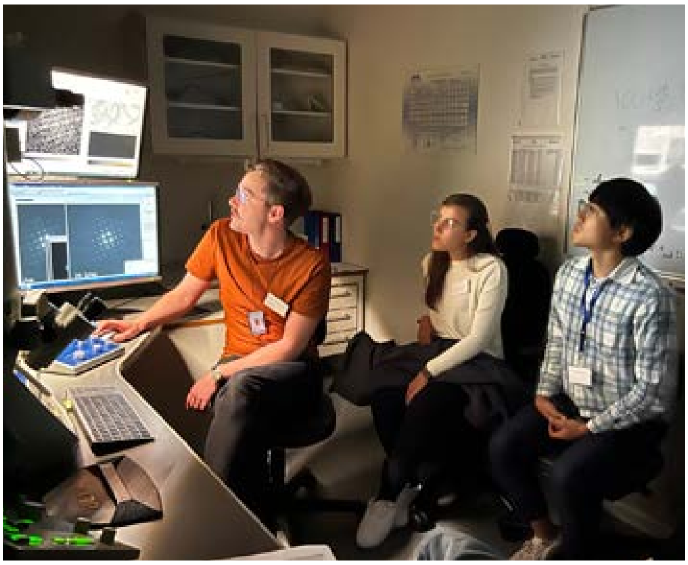
The 2100F features:

- 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
- MerlinEM 1S Medipix direct electron detector from Quantum Detectors
- Gatan TEM/STEM tomography

The 2100 features:

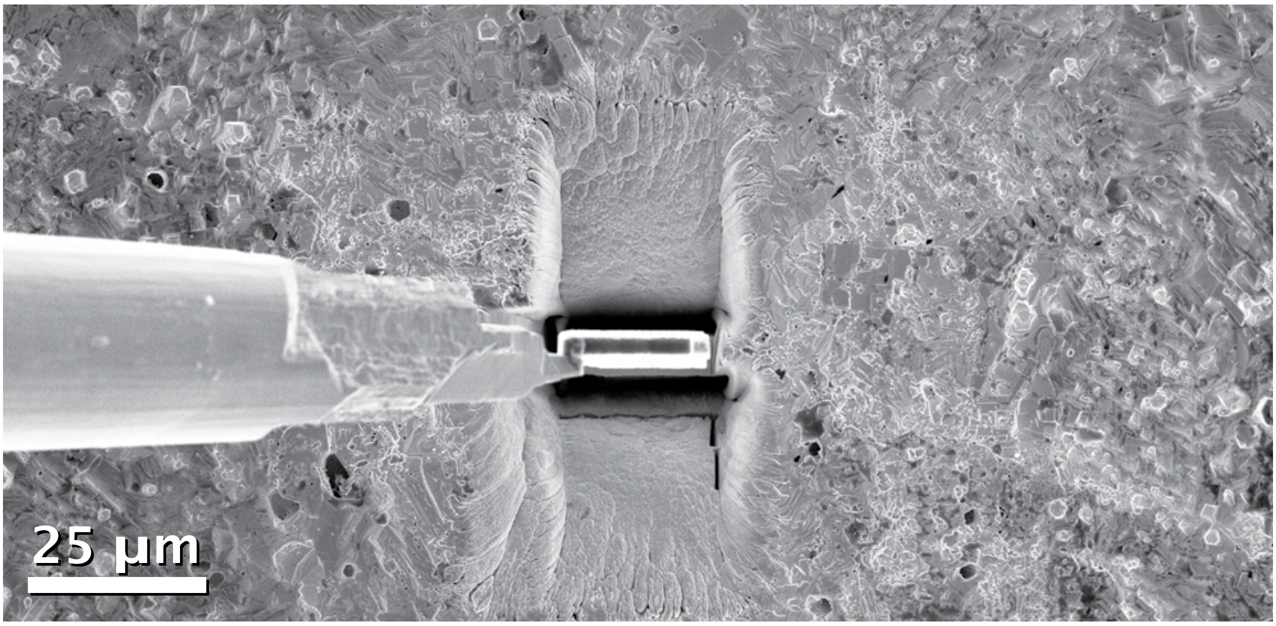
- Gatan 2k Orius CCD (side mounted)
- Scanning option with BF and HAADF detector
- Scanning option with BF and HAADF detector (DigiScan)
- GIF 2002 with 2k CCD post-filter
- Oxford X-Max 80 SDD EDS (solid angle 0.23 sr)

Instrument photos by O. M. Melgård and precession pattern with descan off by J. Sørhaug.



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.



Images on top page: *H. W. Ånes (left image) and E. F. Christiansen (right image) demonstrating the open source Python package kikuchipy for EBSD data processing and analysis, and SPED data acquisition during the ESTEEM3 workshop in Trondheim.*
Image above: *TEM sample preparation using FIB lift-out, by T. Bergh.*

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. We also have a holder tip for TEM characterization of atom probe tomography needles to enable correlated structural and chemical studies of 100 nm^3 sized volumes.

SUPPORTING FACILITIES

With the double 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. In addition, there is a data transfer room with additional facilities such 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. Lately, 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 2022

The total registered used time for the three instruments in 2022 was 3569 hours, including 154 non-paid hours used for testing, competence development, demonstrations, and guided tours. Of the 3415 paid hours, the use by NTNU corresponds to 76 %, externals (with NTNU operator) 1 %, and SINTEF 23 %. NTNU's use is divided over four departments, where the main use is from Department of Physics (82 % of NTNUs paid hours). 96 different projects used TEM in 2022. The infrastructure had in total 55 users, of which 47 were hands-on operators. 5 of the users were based at SINTEF, 16 were PhD candidates and 21 were master students.

Microscope use (hours)	ARM-200F	2100F	2100	Sum
SINTEF	441	129	226	796
NTNU - Physics	361	783	718	1862
NTNU - Other departments	128	120	193	441
NTNU - Visitors from abroad	47	8	26	81
NTNU - Teaching lab	0	119	81	200
External	0	18	17	35
NTNU - Setup/testing/training/ demonstrations	95	59	0	154
Total use	**1072**	**1236**	**1261**	**3569**

Activities, research and events

Image: *grass formation from ICP-RIE etching of silicon for TEM membrane fabrication, by M. Linnerud.*

PhysMet | sfi Centre for Research-based Innovation manufacturing | CASA Centre for Advanced Structural Analysis

50 nm

FOCUS AREAS

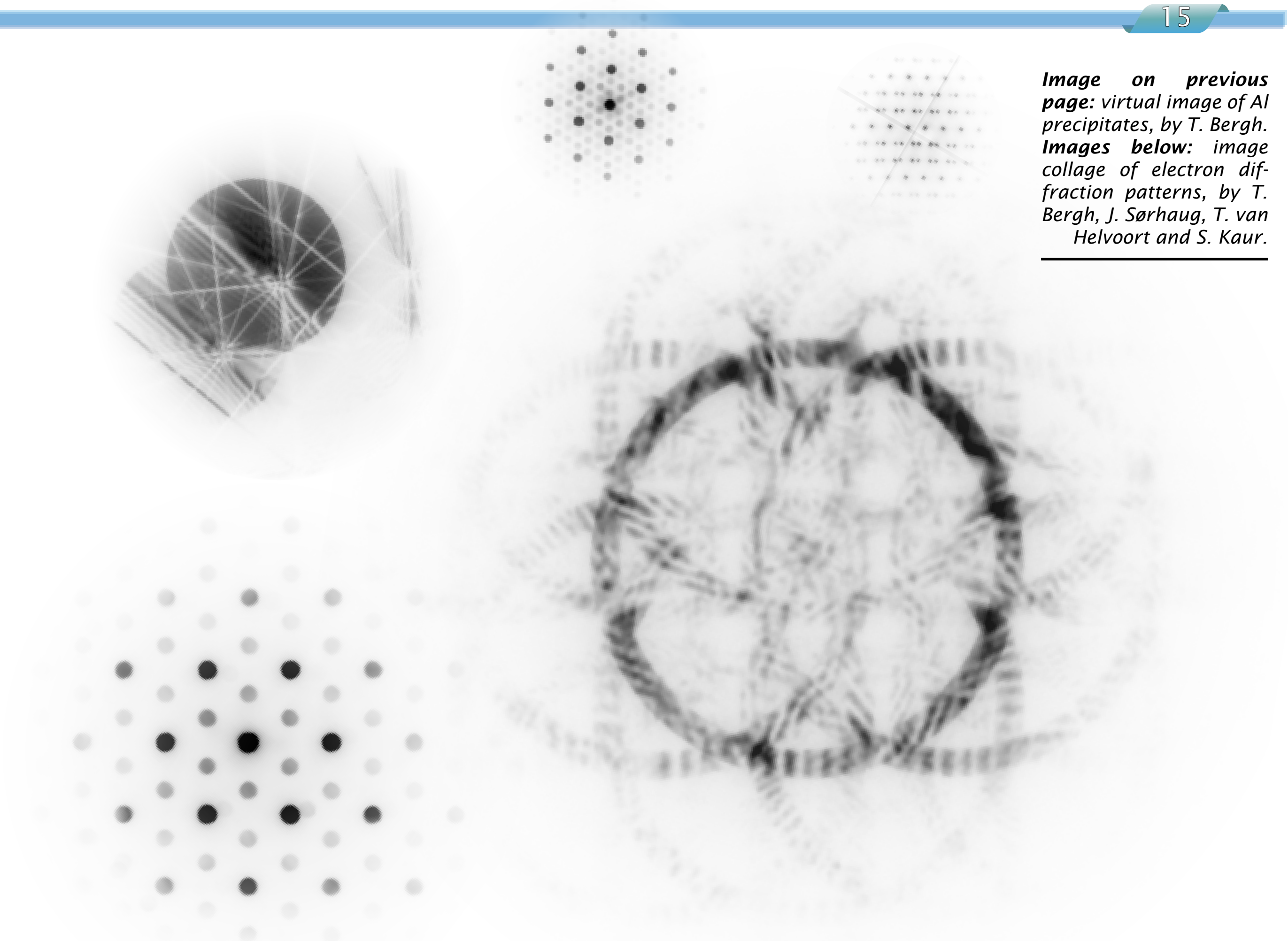
TEM is a powerful technique for fundamental and applied research in the physical sciences, in different fields from geology, metallurgy and semiconductor industry to fundamental chemistry and physics. NORTEM has identified four focus areas, which have been important for the TEM Gemini Centre activities since the Centre was formed. These focus areas were also in the core NORTEM I and II applications and the 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, catalysis, energy materials and nanotechnology**, and will be strategically important for Norway also in the future. The TEM Gemini Centre had activities in all these four areas in 2022. In addition, we have some special topics of interest, like magnetic materials and data analysis, where we recently have increased activity. 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. The Research Council is in 2022/2023 running an evaluation of the natural sciences (EVALNAT). In connection to this the TEM Gemini Centre has submitted an 'Impact Case' where the activities in aluminium alloys between 2011 and 2021 are described. 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 are currently involved in 3 SFI Centers, one competence project (KPN) and one Digitalization project in 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, is ending next year. Here we are involved in 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. There is no PhD or Postdoc hired on this scale now, but there is some SINTEF activity. 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, is also ending next year. Here, we have had some activity on joining of aluminium with other materials in multi-material products. In 2022, Ding Peng worked as a postdoc up to March, and studied additive manufactured materials in the TEM.

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, Multi-scale materials analysis, headed by Randi Holmestad. Tor Inge Thorsen is a PhD student here and will study different joining techniques and additive manufactured materials. 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 will be studied.

Image on previous page: virtual image of Al precipitates, by T. Bergh. *Images below:* image collage of electron diffraction patterns, by T. Bergh, J. Sørhaug, T. van Helvoort and S. Kaur.



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 is a PhD student in this project and focuses on acquiring and analyzing electron diffraction data from quasi-crystalline phases, aided by advanced high-resolution techniques, like APT and 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.

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 Neuman), 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 is 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 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 INT-PART project was renewed for 4 new years in 2019 and extended with new partners. This is further presented elsewhere in this report. Another aluminium project is the NTNU financed Digitalization project AllDesign with Prof. 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 was a PhD student on this project and worked on TEM of early-stage clustering and precipitation. She defended her thesis in September 2022.

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 (online!) – ICAA in Japan, M&M in USA, PICO in the Netherlands - which shows that our work on aluminium is internationally recognized. Furthermore, at the international conference on aluminium alloys (ICAA18) in Japan in September, two people from our group got recognised with an early career researcher (ECR) award based on the quality of their scientific research – Tina Bergh and Elisabeth Thronsen both got ECR awards for Postdoc and PhD student, respectively. Congratulations!

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 TiO_2 thin film growth and characterization with Turid Reenaas as main supervisor. In addition, Andreas Rosnes took his MSc with TEM in this topic in 2022.

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 behavior 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 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, 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 under-

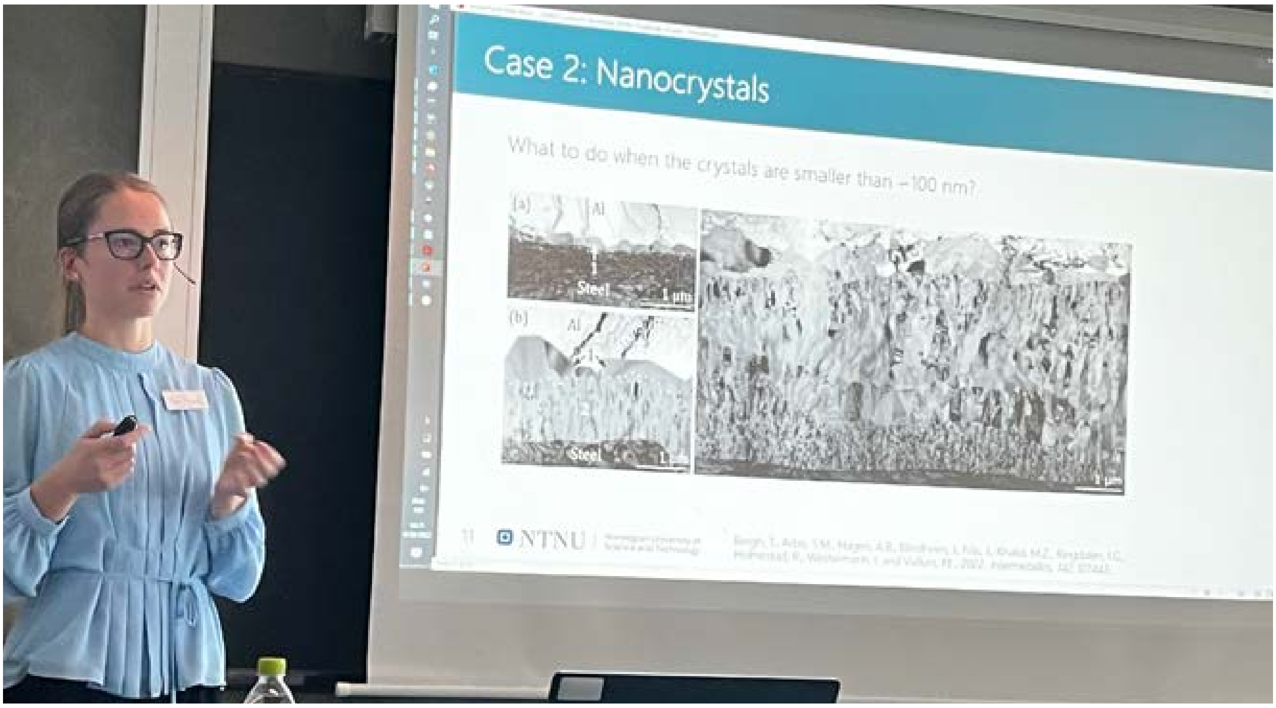


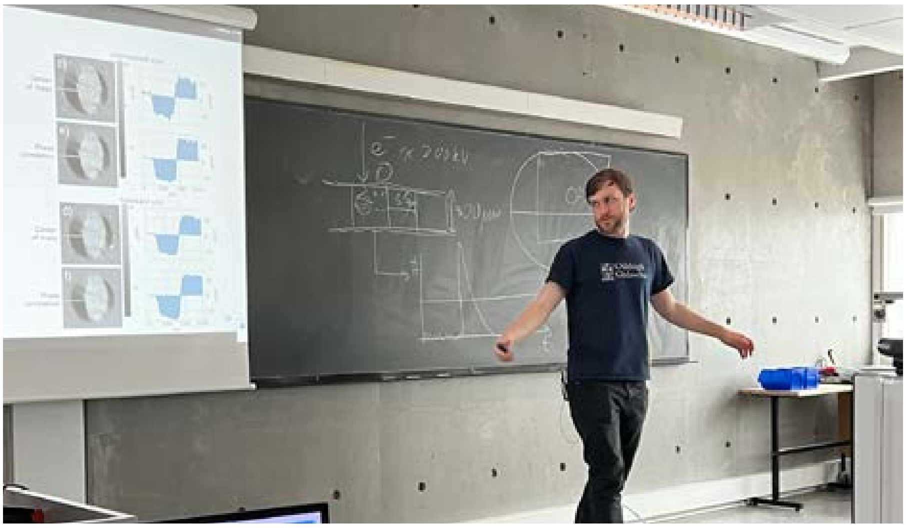
Image: *T. Bergh presenting some of her work on phase identification of nanocrystals during the ESTEEM3 workshop in Trondheim, 2022.*

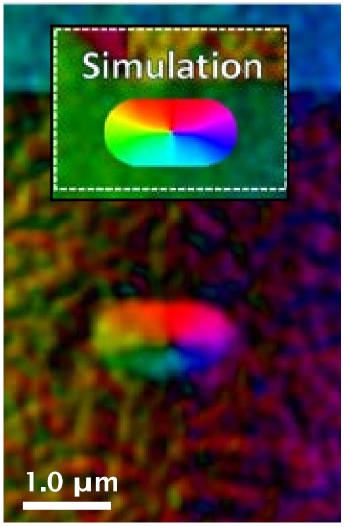
stand the fundamental behavior of the battery electrodes as a function of electrode and electrolyte compositions, synthesis and cycling conditions. Postdoc Inger-Emma Nylund works here on advanced TEM characterization of $LiMn_{2-x}Ni_xO_4$ as high voltage cathode materials in Li-ion batteries.

NANOTECHNOLOGY

For nanotechnology and nano-sciences TEM plays an essential role. TEM is important because it can analyze structure and composition on the same small volume and thereby contribute to understand and tailor the properties of nanomaterials. This was excellently displayed in the celebrations of the Kavli prizes, both to academic and general public (see elsewhere in this annual report). 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 neighbor 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 materialized by Xe-based FIB (owned and run by Department of Mechanical and Industrial Engineering). It is important for TEM preparation of materials like Al that is difficult to prepare with a Ga FIB. 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. Most 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. Also, from the publication list at the end of the annual report our role in studying nanomaterials. In the TEM Gemini Centre, both NTNU and SINTEF have worked with start-up companies within nanotechnology such as CrayoNano. SINTEF is collaborating with CrayoNano to develop light emitting diodes (LEDs) that can emit in the deep UV-C wavelength region. TEM is here the only tool able to both describe the crystal structure, including various types of defects, and the chemical composition along the nanowires in the LEDs. Hence, TEM is used to understand the lasing properties of the LEDs and to further modify and improve the devices.

During 2022, SINTEF has also had a self-financed project (SEP) related to 2D materials for logic in electronics. Again, TEM has here been crucial in order to describe both structure and chemistry of the designs and to couple structural properties on the nm scale to the macroscopic, functional properties.





Images - left image: *M. Nord presenting some work on magnetic field imaging done at the TGC during the ESTEEM3 workshop in Trondheim.* ***Right image****: STEM-DPC image showing magnetic contrast in a nanostructured magnet, corresponding well with simulations, by H. C. Soland.*

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 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 and Sivert Dagenborg as part of the Young Research Talents “In-situ correlated nanoscale imaging of magnetic fields in functional materials” (InCoMa) project from RCN in 2021. Iryna Zelenina was hired as a postdoc for 7 months in 2022, to work on chip-based in-situ sensors. 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.

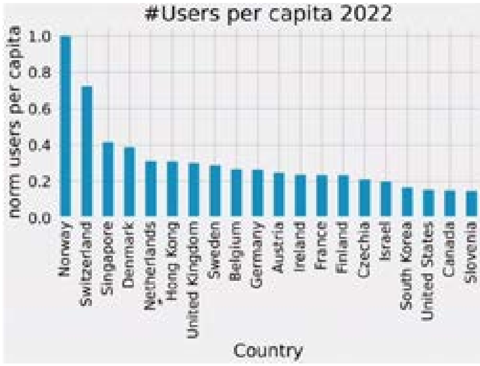
CHEMICAL ENGINEERING - CATALYSIS AND MEMBRANE MATERIALS

The Centre has continued a strong interaction with the national catalysis environment, including the NTNU Chemical Engineering department, SINTEF Industry and Chemistry Oil and Gas Process Technology Departments. The SFI Industrial catalysis science and innovation (iCSI), 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 focus in 2022 was placed on silver catalysts for the methanol to formaldehyde reaction. 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. The newly installed fast TEM camera on the ARM will allow studying dynamics in combination with in-situ holders.

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. In 2022 the TEM Gemini Centre installed it's second direct electron detector. This is placed on the double-corrected ARM, is 4 times as large as the first one, and its main task is 4DSTEM techniques. 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. 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). This can be seen in the statistics in the figure below, presented by Francisco de la Peña in a recent ESTEEM3 seminar. Open-science, even in these troubled times, is growing and pushed by EU and other authorities. It is a key element of modern science and innovation. Beside ongoing activities, new projects primarily focused on advanced data handling and with a tide link towards modelling (e.g. DFT) will start in 2023.



RESEARCHERS' NIGHT, SCHOOL VISITS AND OUTREACH

The TEM Gemini Centre has contributed to many high school visits and Researchers Nightduring the last year. At Researchers Night in September 2022 a big delegation from the TEM group participated, some remotely from the basement of KJ1.

Image below: *People from the TGC during Researchers' night at NTNU Gløshaugen.*
(Back left to right, top): B. G. Soleim, S: Dagenborg, G. Nordahl, E. F. Christiansen, M. Linnerud and H. C. Soland.
(Front left to right, bottom): C. Hell, T. I. Thorsen and S. Boucher.

This project has received funding from the European Union's Horizon 2020 research and innovation program under grant agreement no 823717.

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 TA and we have now been involved in around 20 projects. These projects were initiated from Germany, Romania, UK, Spain, Sweden and Japan. 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' together with AGH University of Science and Technology in 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. ESTEEM3 ends in June 2023 and for the TEM Gemini Centre it has been a great success so far.

ESTEEM3 WORKSHOP ON ELECTRON DIFFRACTION FOR SOLVING ENGINEERING PROBLEMS

21.-23. June 2022 NTNU in Trondheim organised an ESTEEM3 workshop on 'Electron diffraction for solving engineering problems'. 26 participants from eight European countries came to Trondheim for the workshop, in addition we had 16 in local organising committee and 14 participants online. Lectures were given by Paul Midgley (Cambridge University, UK) and Stefan Zaefferer (MPI Düsseldorf, Germany) in addition to lectures from the local NTNU teachers Tina Bergh and Magnus Nord. Practicals were given by Stefan Zaefferer, Shao-Pu Tsai (MPI Düsseldorf, Germany), Joonatan Laulainen (Cambridge University, UK), and NTNU people; Håkon Wiik Ånes, Tina Bergh, Magnus Nord, Emil Frang Christiansen, Gregory Nordahl, Dipanwita Chatterjee, Joseph Broussard and Tor Inge Thorsen. Two lab sessions were on microscopes – one on TEM and one on SEM. The rest of the labs was on coding, and participants had to install the software (Hyperspy and Pyxem) and did analysis themselves on their own computers The workshop was very well received, and the participants said they had learned a lot.

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 in 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 has been very delayed. However, activities start to increase, and DTU in Denmark organised an in-situ workshop in July 2022, where Tina Bergh from our group participated. there are still some years left, and hopefully we will see some more good Nordic collaboration in the years to come.

ACTIVE PROJECTS IN 2022

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 96 different projects using the facilities in 2022.

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.	1 postdoc NTNU, 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, 1 postdoc	2015-23
	Partners: 7 research institutions including both SINTEF and NTNU, 7 public bodies, 26 industrial partners		
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, Speira		
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
FRIPRO	In-situ correlated nanoscale imaging of magnetic fields in functional materials (InCoMa)	1 PhD, 1 postdoc NTNU	2021-25
NTNU Nano Enabling technologies	Enabling nanoscale in-operando studies of advanced epitaxial functional thin film materials using nanofabrication	1 PhD, NTNU	2022-26
IPN/ENERGIX	UVC LEDs based on nanowires-on-graphene: CrayoNano, SINTEF, NTNU	SINTEF	2022-24
IPN/ENERGIX	Surface treatment of Artificial Graphite for Anodes in Lithium-Ion Batteries.	SINTEF	2020-22
	Partners: SINTEF, Vianode, IFE		
IPN/ENERGIX	HAST.	SINTEF	2020-22
	Partners: Cenate, Dynatec, SINTEF, IFE		
NTNU Digital trans-formation	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	2017-2022
INTPART	Norwegian-Japanese Aluminium alloy Research and Education Collaboration (NJALC) – II.	NTNU, SINTEF, Travel, exchange students	2019-23
	Partners: NTNU, SINTEF, Hydro, University of Toyama, Tokyo Institute of Technology		
EU	ESTEEM3 - https://www.esteem3.eu/	1 postdoc NTNU, prof IIs	2019-23
IPN/PETROMAKS2	AMRREX	SINTEF	2018-21
SINTEF/SEP	Molecular structure of organic nanomaterials (MOSON)	SINTEF	2021-22
SINTEF/SEP	NeuroMorph	SINTEF	2022
IPN	Novel Failure Monitoring System for Marine Applications by including Acoustic Emission (AE-MON)	SINTEF	2019-22
IPN	Catch & Kill.	SINTEF	2020-23
	Partners: SINTEF, Standard Bio, USN, Uni. New South Wales		
EU/H2020	SAFE-N-MEDTECH	SINTEF	2019-23
EU/H2020	Hydra	SINTEF	2020-24
	Partners: SINTEF, Uppsala Universitet, CEA, UCL, FAAM, DLR, ISCI, Solvionic, Corvus, POLITO, Elkem, Johnsen Matthey		
Grønn plattform	AluGreen	SINTEF	2021-24
	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		
IPN	IPN Coulombus	SINTEF	2022-23
Eu/H2020	MatCHMaker	SINTEF	2022-26
	Partners: CEA, RINA-C, CSM, SINTEF, AIMEN, SIMAVI, HeidelbergCement, GENVIA, Toyota, TU Wien, ASRO.		
Grønn plattform	SUMBAT	SINTEF	2022-24
	Partners: SINTEF, NTNU, UiO, UiA, IFE, Freyr, Vianode, Elkem, Morrow, Norsk Hydro, Corvus		

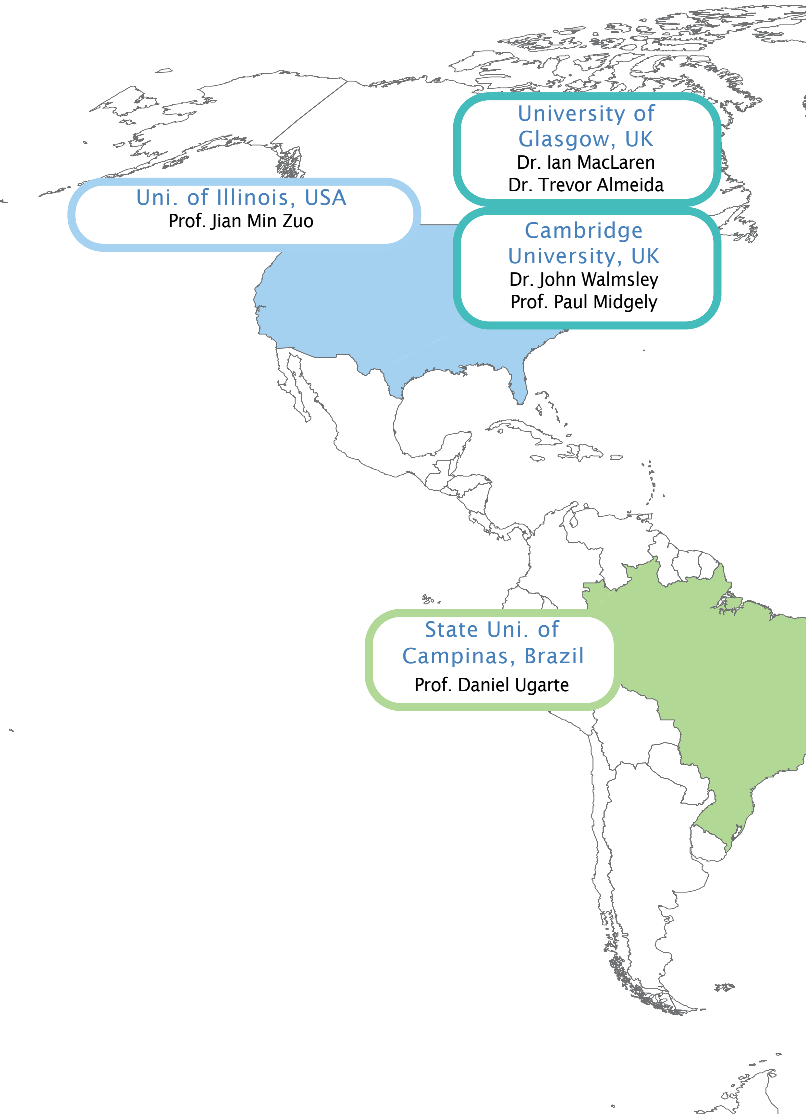
VISIT FROM KAVLI PRIZE WINNERS

The Kavli Prize honours scientists for breakthroughs in astrophysics, nanoscience and neuroscience – transforming our understanding of the big, the small and the complex. Harald Rose, Knut Urban, Max Haider and Ondrej Krivanek were awarded the 2020 Kavli Prize in Nanoscience for '*sub-ångström resolution imaging and chemical analysis using electron beams*'. More specific (citing the award committee) the got the prize for

- **Harald Rose**, for proposing a novel lens design, the Rose corrector, enabling aberration correction in transmission electron microscopy that can be applied to both conventional and scanning microscopes.
- **Maximilian Haider**, for the realization of the first sextupole corrector, based on Rose's design, and for his role in the implementation of the first aberration corrected conventional transmission electron microscope.
- **Knut Urban**, for his role in the implementation of the first aberration corrected conventional transmission electron microscope.
- **Ondrej L. Krivanek**, for the realization of the first aberration corrected scanning transmission electron microscope with sub-ångström resolution, well suited for spatially resolved chemical analysis. This was obtained using a quadrupole-octupole corrector.

Because of the pandemic, the award ceremony was postponed to 2022, and the winners came to Oslo and Norway to receive the prize in September. They gave their scientific talks in Trondheim in the Nano-symposium organised by NTNU NanoLab. The laureates and their families stayed one day extra in Trondheim and participated in a seminar organised by the TEM Gemini Centre and came to guided tour, concert, and dinner at Ringve Museum. We had many good discussions with the impressive scientists and gründers and are very honoured by the visit!

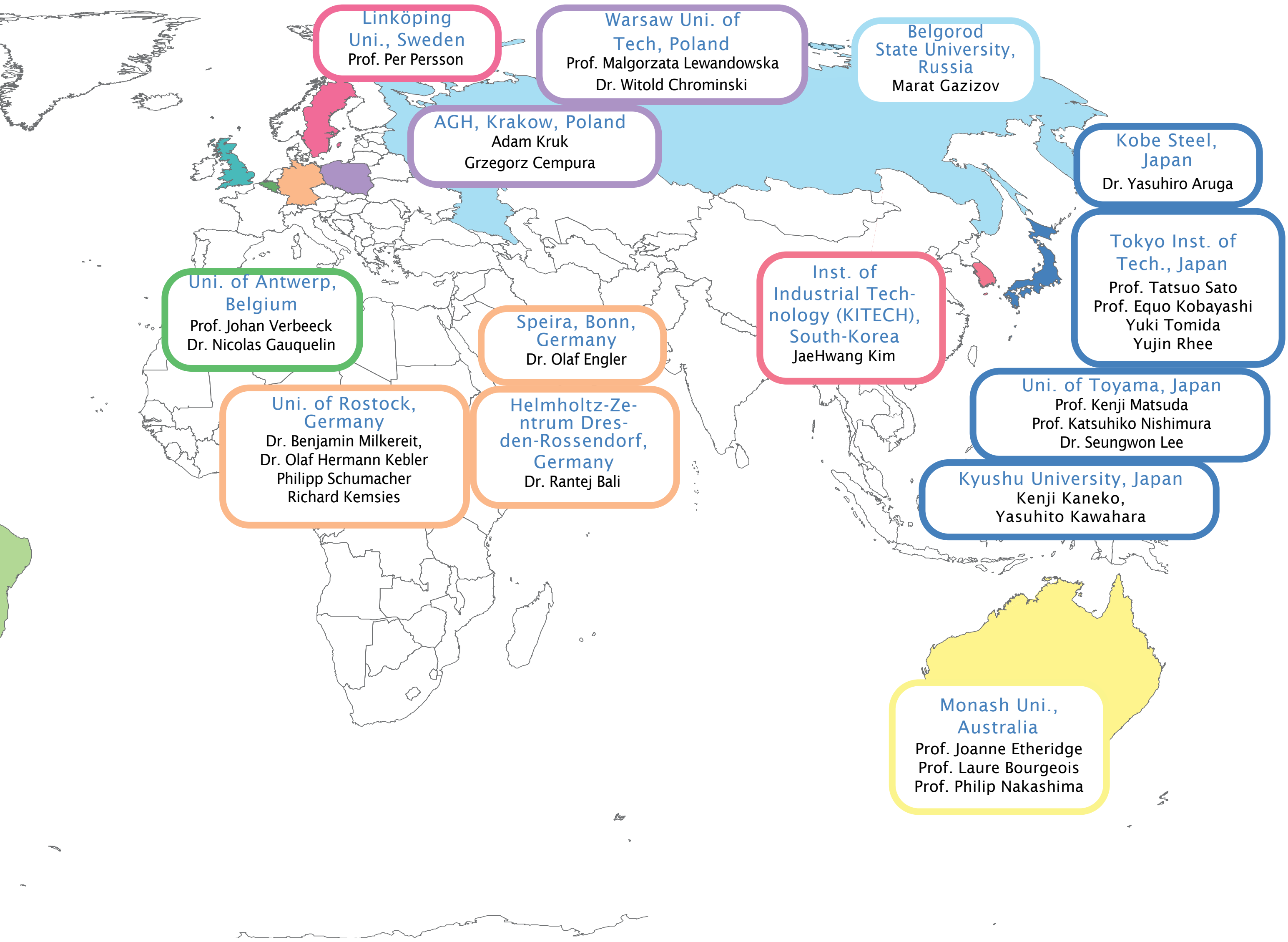
Ondrej Krivanek and his wife stayed a few days longer in Trondheim and visited the DNT cabin Imarbu in Nordmøre together with a few members of the group for social and informal further discussions.



INTERNATIONAL COLLABORATION

Also 2022 has been affected by the pandemic, but travel and visits are slowly turning back to normal. We have managed to maintain most of our international network and 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. Three students from Tokyo Institute of Technology and Kyushu University visited the group in the fall of 2022. 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:

- Elizabeth C. Dickey, Carnegie Mellon University, US, April 22
- Paul Midgley, Cambridge University, UK, June 22
- Stefan Zaefferer, Max-Planck-Institute Düsseldorf, Germany, June 22
- Shao-Pu Tsai, Max-Planck-Institute Düsseldorf, Germany, June 22
- Joonatan Laulainen, Cambridge University, UK, June 22
- Yasuhito Kawahara, Kyushu University, Japan, Sept. 22 - Febr. 23
- Yuki Tomida, Tokyo Institute of Technology, Japan, Sept. 22 – Dec. 22
- Yujin Rhee, Tokyo Institute of Technology, Japan, Sept. 22- March 23
- Normand Mousseau, University of Montreal, Candana, Sept. 22
- Stefan Pogatscher, Montanuniversität Leoben, Austria, Sept 22
- Joke Hadermann, EMAT, University of Antwerp, Belgium, Sept 22
- Xiaoqing Pan, University of California, Irvine, US, Sept 22
- Dierk Raabe, Max-Planck-Institute Düsseldorf, Germany, Nov. 22

Images: *(left image) Y. Kawahara, Y. Tomida and Y. Rhee visiting the TGC at NTNU Gløshaugen, Trondheim (1-5 months); (Top right) Visiting researchers joining yearly Bymarka walk; (Bottom right) Jostein Røyset from Hydro showing the visiting researchers how they extrude alloys in the press in Perleporten.*

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. Objective 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. However, we see from the publication list that the collaboration still is there, as we had many common publications the last years. 2022 still had covid measures in Asian countries, but three from the TEM Gemini Centre (Tina Bergh, Christoph Hell and Tor Inge Thorsen) managed to get business visa and went to the ICAA conference in Toyama in September. In October, Knut Marthinsen and Randi Holmestad participated in the CAMRIC conference by Zoom. Knut and Randi also gave lectures online for Japanese students. Three Japanese students came to Norway in September 2022. PhD student Yasuhito Kawahara from Kyushu University, PhD student Yujin Rhee and MSc student Yuki Tomida from Tokyo Institute of Technology stayed one month at Hydro Sunndalsøra in Sept-Oct and studied aluminium alloys. From mid-October they have stayed in Trondheim and done TEM work using the NORTEM facility. The INTPART project is prolonged for one year, to June 2023. and the plan is to go to Japan in April 2023. Unfortunately, we will not manage to get back on track or spend the resources before the project ends, but we can hopefully manage to find other ways or projects to continue the collaborations.

PHD DEFENSES

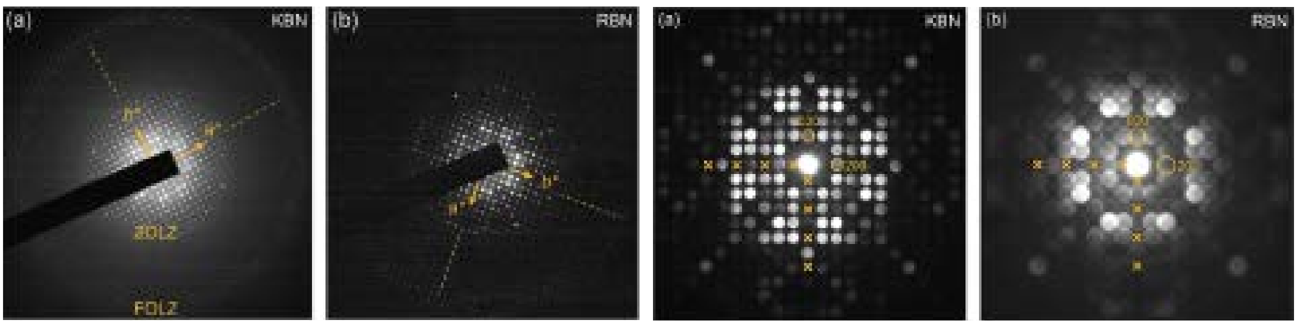
IN THE TEM GEMINI CENTRE 2022

INGER-EMMA NYLUND, 22. APRIL 2022

Inger-Emma Nylund worked in the FACET group in the Materials Science and Engineering Department and wrote a thesis with the title 'Transmission electron microscopy of ferroic materials'. In her thesis work, three structurally complex ferroelectric materials were studied by TEM, taking advantage of the possibility to perform imaging, diffraction, and spectroscopy within the same instrument. The three materials systems investigated were ferroelectric $BaTiO_3$films, tetragonal tungsten bronzes (TTB) with various chemical compositions, and improper ferroelectric $Gd_2(MoO_4)_3$. A thorough investigation of the misfit dislocations in the $BaTiO_3$ films was performed, demonstrating that strain in the films were completely relaxed by the dislocations. $K_4Bi_2Nb_{10}O_{30}$ and $Rb_4Bi_2Nb_{10}O_{30}$ were investigated by convergent-beam electron diffraction (CBED) to determine the crystal symmetry. Lastly, high quality STEM images were obtained for the beam sensitive improper ferroelectric $Gd_2(MoO_4)_3$, where the goal was to obtain the polarization orientation from atomically resolved images. This turned out to be a challenge but motivated a study of the improper phase transition of $Gd_d(MoO_4)_3$ by temperature dependent XRD. The work presented in this thesis has demonstrated that the TEM is a very useful tool in the study of ferroic materials. Local variation in the structure and chemistry at an atomic scale were investigated in oxide films as well as bulk materials, which would have been difficult to obtain by other experimental techniques. Determination of the centrosymmetric crystal symmetry of two compounds, previously not determined, was also demonstrated by CBED.

Image: *(From back left to right) M. Nord, P. E. Vullum, T. Grande. (From front left to right) Ø. Prytz (UiO), M.-A. Einarsrud, E. C. Dickey (CMU) and fresh Dr. I.-E. Nylund.*

The examination committee consisted of Prof. Elizabeth C. Dickey, Carnegie Mellon University, US, Prof. Øystein Prytz, UiO and A. Prof. Magnus Nord, NTNU. Inger-Emma's supervisors were Tor Grande with co-supervisors Per Erik Vullum and Mari-Ann Einarsrud. Inger-Emma is now hired as a postdoc in the battery group in the Department of Materials Science and Engineering, working with advanced characterization, and is therefore still a member of the TEM Gemini Centre.



Images: *Diffraction patterns (selected area and convergent beam) used for phase identification and space group determination of KBN and RBN. See https://doi.org/10.1021/acs.chemmater.2c01944*

ELISABETH THRONSEN, 29. SEPTEMBER 2022

Image: *(Left to right) J. Hadermann (EMAT), fresh Dr. E. Thronsen, S. Pogatscher (MU Leoben) and P. Kontis (NTNU).*

Elisabeth Thronsen worked in the AllDesign project under the Digital Transformation initiative at NTNU, and wrote a thesis entitled 'The effect of natural ageing on clustering and precipitation in heat-treatable aluminium alloys - An advanced TEM study'. Her work concerned age-hardenable aluminium alloys which gain their strength from metastable, nanoscale precipitates formed during the last stage of the alloy's thermomechanical treatment. The precipitates hinder dislocation motion, thereby significantly increasing the strength of the alloy. The precipitates are preceded by solute clusters or Guinier-Preston (GP) zones formed in the initial stages of artificial ageing or during room temperature storage prior to artificial ageing. Understanding the atomic structure of clusters and GP zones and how they evolve to precipitates is important for developing new alloys and for modelling the mechanical properties of Al alloys.

Among the main findings, the previously unknown crystal structure of the GPI zones in Al-Zn-Mg(-Cu) alloys were solved by analyzing atomically resolved scanning transmission electron microscopy (STEM) images. The atomic models were compared with electron diffraction patterns to ensure their validity. Further, the effect of the GPI zones on the precipitation during artificial ageing was investigated and it was concluded that the GPI zones undergo partial reversion upon ageing above a critical temperature. Moreover, the effect of pre-deformation on precipitation was investigated on heavy deformed Al-Mg-Cu-Si alloys and it was found that higher solute content in the matrix increased the precipitation away from the deformation induced defects. A major part of this work relied on scanning electron diffraction combined with advanced data analysis to obtain statistics of the precipitate distribution or to obtain diffraction patterns of single particles. Part of this work is dedicated to implementing and improving data analysis approaches of such data, which typically rely on analysing 400 000 diffraction patterns. The examination committee consisted of Prof. Stefan Pogatscher, Montanuniversität Leoben, Austria, Prof. Joke Hadermann, EMAT, University of Antwerp, Belgium and A. Prof. Paraskevas Kontis, NTNU. Elisabeth's supervisor was Randi Holmestad, co-supervisors were Sigurd Wenner, Ton van Helvoort and Calin Marioara. Elisabeth is still in the TEM Gemini Centre, as she was hired as a research scientist in the Materials Physics group in Trondheim in SINTEF Industry. We are very happy to still have Elisabeth around!

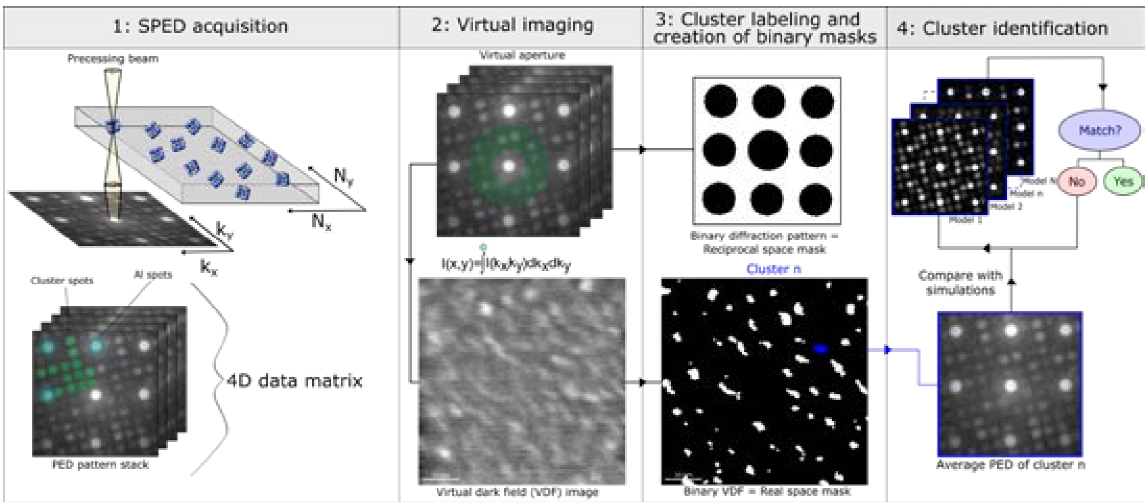
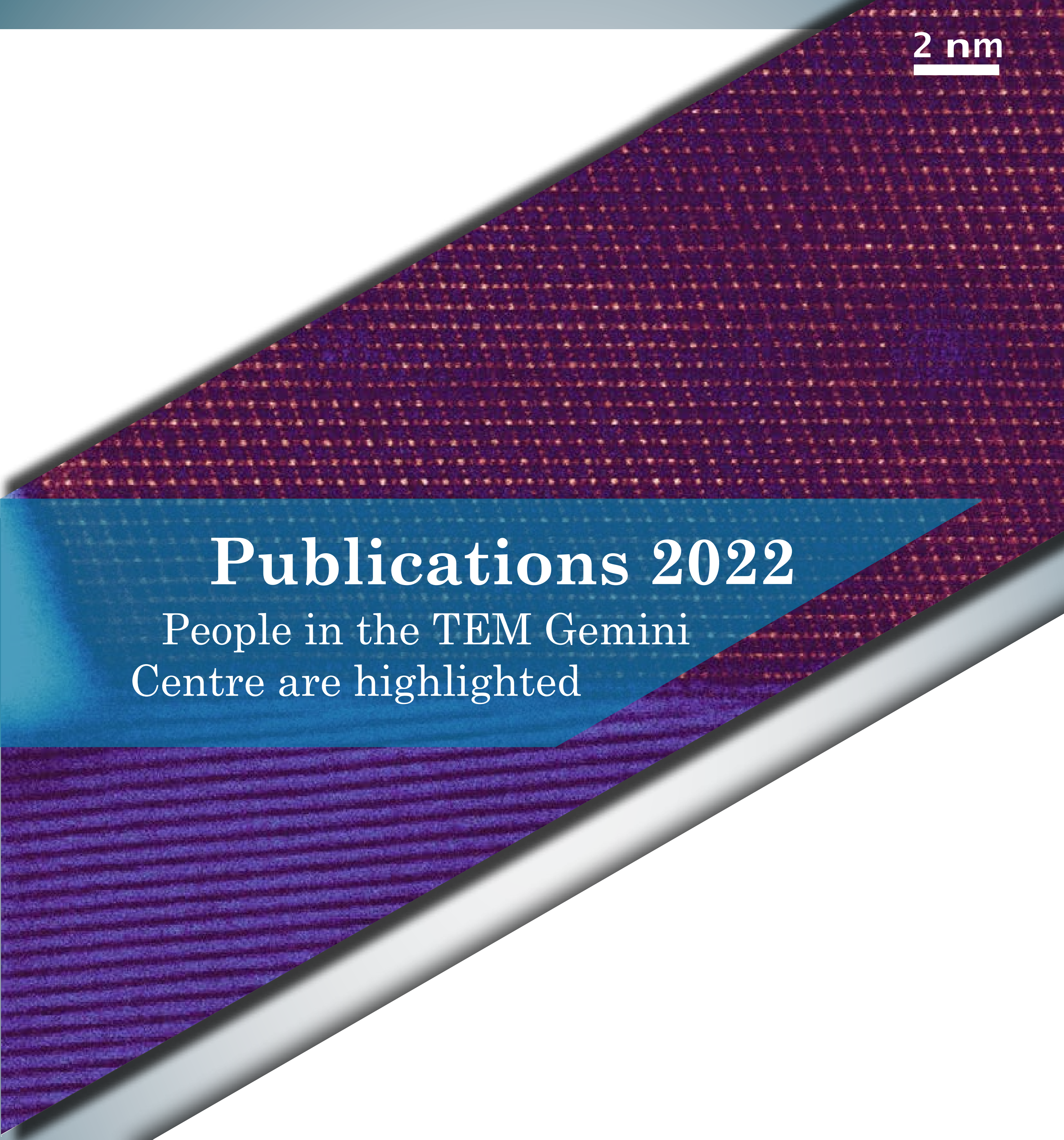


Figure: *Workflow of SPED experiments and data analysis for studying GP zones and phase identification, by E. Thronsen.*
See https://doi.org/10.1016/j.matchar.2021.111675org/10.1016/j.matchar.2021.111675 org/10.1016/j.matchar.2021.111675



Publications 2022

People in the TEM Gemini Centre are highlighted

Image: *grain boundary in the Li-ion material $LiNi_{0.5}MMn_{1.5}O_4$ (LNMO), by I.-E. Nylund.*

TEM GEMINI CENTRE PUBLICATIONS 2022

**: Not from TGC infrastructure. (Some are TEM papers, but TEM not taken in TRD).

JOURNAL PUBLICATIONS

** Akselsen, O. M.; **Bjørge, R.**; **Ånes, H. W.**; Ren, X.; Nyhus, B. Microstructure and Properties of Wire Arc Additive Manufacturing of Inconel 625. Metals **2022**, 12, 1-18. http://dx.doi.org10.3390/met12111867

Asheim, K.; **Vullum, P. E.**; Wagner, N. P.; Andersen, H. F.; Maehlen, J. P.; Svensson, A. M. Improved electrochemical performance and solid electrolyte interphase properties of electrolytes based on lithium bis(fluorosulfonyl)imide for high content silicon anodes. Rsc Advances **2022**, 12, 12517-12530. https://doi.org/10.1039/D2RA01233B

Bakardjieva, S.; Mares, J.; Koci, E.; Tolasz, J.; Fajgar, R.; Ryukhtin, V.; Klementova, M.; Michna, S.; Bibova, H.; **Holmestad, R.**; et al. Effect of Multiply Twinned Ag-(0) Nanoparticles on Photocatalytic Properties of TiO_2 Nanosheets and TiO_2 Nanostructured Thin Films. Nanomaterials **2022**, 12. https://doi.org/10.3390/nano12050750

Bakken, K.; Blichfeld, A. B.; **Nylund, I.-E.**; Chernyshov, D.; Glaum, J.; Grande, T.; Einarsrud, M.-A. Tailoring Preferential Orientation in $BaTiO_3$-based Thin Films from Aqueous Chemical Solution Deposition. Chemistry–Methods **2022**, 2, e202100064. https://doi.org/10.1002/cmtd.202100064

Bergh, T.; Arbo, S. M.; Hagen, A. B.; Blindheim, J.; **Friis, J.**; Khalid, M. Z.; Ringdalen, I. G.; **Holmestad, R.**; Westermann, I.; **Vullum, P. E.** On intermetallic phases formed during interdiffusion between aluminium alloys and stainless steel. Intermetallics **2022**, 142. https://doi.org/10.1016/j.intermet.2021.107443

Biserova-Tahchieva, A.; **Chatterjee, D.**; **van Helvoort, A. T.J.**; Llorca-Isern, N.; Cabrera, J. M. Effect of the nanostructuring by high-pressure torsion process on the secondary phase precipitation in UNS S32750 Superduplex stainless steel. Materials Characterization **2022**, 183, 111639. https://doi.org/10.1016/j.matchar.2021.111639

Bisht, P.; Kumar, A.; Ghosh, A.; **Vullum, P. E.**; Sunding, M. F.; Belle, B. D.; Mehta, B. R. Tailoring the Vertical and Planar Growth of 2D WS_2 Thin Films Using Pulsed Laser Deposition for Enhanced Gas Sensing Properties. Acs Applied Materials & Interfaces **2022**, 14, 36789-36800. https://doi.org/10.1021/acsami.2c07759

Bjørkedal, O. H.; Regli, S. K.; Nuguid, R. J. G.; **Vullum, P. E.**; Kröcher, O.; Ferri, D.; Rønning, M. One-pot synthesis of highly dispersed mesoporous Cu/ZrO_2 catalysts for NH3-SCR. Catalysis Today **2022**, 384-386, 113-121. https://doi.org/10.1016/j.cattod.2021.05.010

** Burgmann, S.; Lid, M.; Chaikasetsin, S.; Bjordal, D. S.; Prinz, F.; Provine, J.; Berto, F.; **van Helvoort, A. T. J.**; Torgersen, J. Approaching the Limits of Aspect Ratio in Free-Standing Al_2O_3 3D Shell Structures. Advanced Engineering Materials **2022**, 24, 2200444. https://doi.org/10.1002/adem.202200444

** Cautaerts, N.; Crout, P.; **Ånes, H. W.**; Prestat, E.; Jeong, J.; Dehm, G.; Liebscher, C. H. Free, flexible and fast: Orientation mapping using the multi-core and GPU-accelerated template matching capabilities in the Python-based open source 4D-STEM analysis toolbox Pyxem. Ultramicroscopy **2022**, 237, 113517-113517. https://doi.org/10.1016/j.ultramic.2022.113517

Ciancio, R.; Dunin-Borkowski, R. E.; Snoeck, E.; Kociak, M.; **Holmestad, R.**; Verbeeck, J.; Kirkland, A. I.; Kothleitner, G.; Arbiol, J. e-DREAM: the European Distributed Research Infrastructure for Advanced Electron Microscopy. Microscopy and Microanalysis **2022**, 28, 2900-2902. https://doi.org/10.1017/S1431927622010893

Dehghan-Niri, R.; Tsakoumis, N. E.; Voronov, A.; Holmen, A.; **Holmestad, R.**; **Vullum, P.-E.**; Borg, O.; Rytter, E.; Ronning, M.; **Walmsley, J. C.** Nanostructural Analysis of Co-Re/gamma-Al_2O_3 Fischer-Tropsch Catalyst by TEM and XRD. Chemcatchem **2022**. https://doi.org/10.1002/cctc.202101931

Dull, S. M.; Vinogradova, O.; Xu, S.; Koshy, D. M.; **Vullum, P. E.**; Torgersen, J.; Kirsch, S.; Viswanathan, V.; Jaramillo, T. F.; Prinz, F. B. Alloyed Pt–Zn Oxygen Reduction Catalysts for Proton Exchange Membrane Fuel Cells. ACS Applied Energy Materials **2022**. https://doi.org/10.1021/acsaem.2c00816

Elkjaer, A.; **Sørhaug, J. A.**; Ringen, G.; **Bjørge, R.**; Grong, Ø. Electrical and thermal stability of Al-Cu welds: Performance benchmarking of the hybrid metal extrusion and bonding process. Journal of Manufacturing Processes **2022**, 79, 626-638. https://doi.org/10.1016/j.jmapro.2022.04.029

** Gawel, K.; **Wenner, S.**; Jafariesfad, N.; Torsaeter, M.; Justnes, H. Portland cement hydration in the vicinity of electrically polarized conductive surfaces. Cement & Concrete Composites **2022**, 134, 9. https://doi.org/10.1016/j.cemconcomp.2022.104792

** Gayen, M.; Ariyadka, S.; Agarwal, S.; **Chatterjee, D.**; Singh, A.; Ravishankar, N. Tuning Catalytic Activity in Ultrathin Bimetallic Nanowires Via Surface Segregation: Application in Electrochemical Methanol Oxidation. In ECS Meeting Abstracts, **2022**; IOP Publishing: p 1702. https://iopscience.iop.org/article/10.1149/MA2022-01381702mtgabs/meta

Gazizov, M. R.; Boev, A. O.; **Marioara, C. D.**; **Holmestad, R.**; Gazizova, M. Y.; Kaibyshev, R. O. Edge interfaces of the Omega plates in a peak-aged Al-Cu-Mg-Ag alloy. Materials Characterization **2022**, 185. https://doi.org/10.1016/j.matchar.2022.111747

Hagen, A. B.; **Wenner, S.**; **Bjørge, R.**; Wan, D.; **Marioara, C. D.**; **Holmestad, R.**; **Ringdalen, I.** G. Local mechanical properties and precipitation inhomogeneity in large-grained Al-Mg-Si alloy. Materials Science and Engineering: A **2022**, 144222. https://doi.org/10.1016/j.msea.2022.144222

Hansen, H. E.; Seland, F.; Sunde, S.; Burheim, O. S.; Pollet, B. G. Frequency controlled agglomeration of pt-nanoparticles in sonochemical synthesis. Ultrasonics Sonochemistry 2022, 85. https://doi.org/10.1016/j.ultsonch.2022.105991

Heinig, M. F.; **Chatterjee, D.**; **van Helvoort, A. T. J.**; Wagner, J. B.; Kadkhodazadeh, S.; **Ånes, H. W.**; Niessen, F.; da Silva Fanta, A. B. High resolution crystal orientation mapping of ultrathin films in SEM and TEM. Materials Characterization **2022**, 189, 111931. https://doi.org/10.1016/j.matchar.2022.111931

Hell, C. M.; Søreide, H.-S.; **Bjørge, R.**; **Marioara, C. D.**; **Li, Y.**; **Holmestad, R.** Influence of Natural Aging and Ramping before Artificial Aging on the Microstructure of two different 6xxx Alloys. Journal of Materials Research and Technology **2022**, 21, 4224-4240 https://doi.org/10.1016/j.jmrt.2022.10.139

Hunnestad, K. A.; Hatzoglou, C.; Khalid, Z. M.; **Vullum, P. E.**; Yan, Z.; Bourret, E.; **van Helvoort, A. T. J.**; Selbach, S. M.; Meier, D. Atomic-scale 3D imaging of individual dopant atoms in an oxide semiconductor. Nature Communications 2022, 13, 7, https://www.nature.com/articles/s41467-022-32189-0

Kessler, O.; Zhuravlev, E.; **Wenner, S.**; Heiland, S.; Schaper, M. Correlation between Differential Fast Scanning Calorimetry and Additive Manufacturing Results of Aluminium Alloys. Materials **2022**, 15, 7195. https://doi.org/10.3390/ma15207195

Knies, J.; Schonenberger, J.; Zwingmann, H.; van der Lelij, R.; Smelror, M.; **Vullum, P. E.**; Bronner, M.; Vogt, C.; Fredin, O.; Muller, A.; et al. Continental weathering and recovery from ocean nutrient stress during the Early Triassic Biotic Crisis. Communications Earth & Environment **2022**, 3, 12. https://www.nature.com/articles/s43247-022-00480-z

** Liu, S.; Wan, D.; **Peng, D.**; Lu, X.; Ren, X.; Fu, Y.; Wang, F.; **Li, Y.**; Zhang, Z.; He, J. Effect of heat input on nanomechanical properties of wire-arc additive manufactured Al 4047 alloys. Materials Science and Engineering: A **2022**, 144288. https://doi.org/10.1016/j.msea.2022.144288

Lu, F.; **Sunde, J. K.**; **Marioara, C. D.**; **Holmestad, R.**; Holmedal, B. An improved modelling framework for strength and work hardening of precipitate strengthened Al-Mg-Si alloys. Materials Science and Engineering A-Structural Materials Properties Microstructure and Processing **2022**,

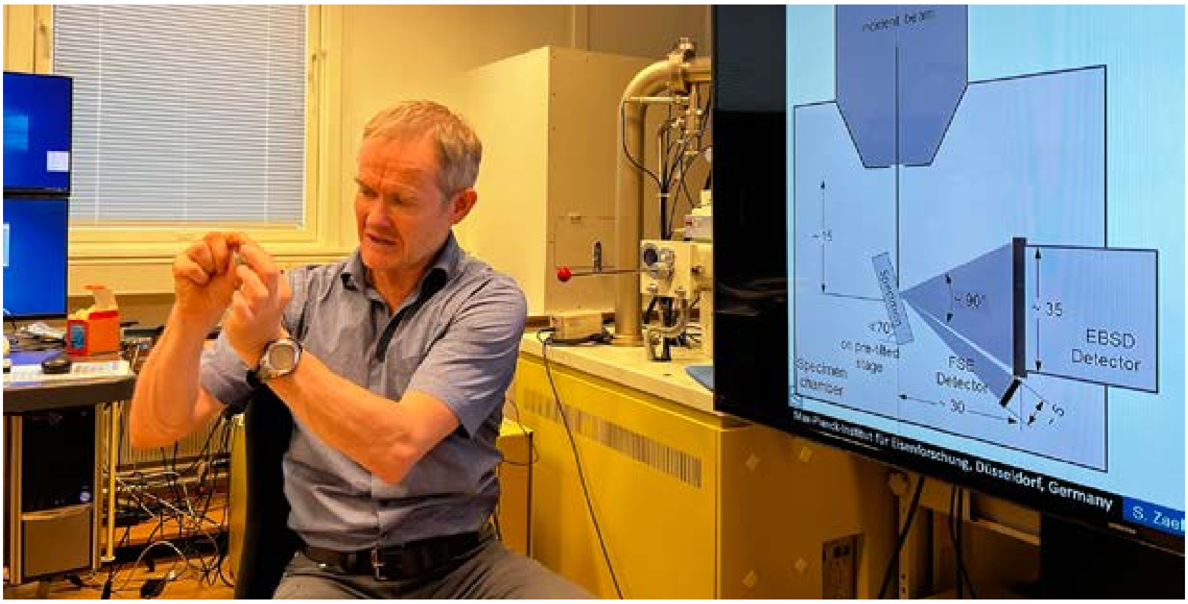


Image: *Stefan Zaefferer lecturing about the physics behind EBSD signal generation and data acquisition during the ESTEEM3 workshop in Trondheim.*

832. https://doi.org/10.1016/j.msea.2021.142500

Lu, X.; Ma, Y.; **Peng, D.**; Johnsen, R.; Wang, D. In situ nanomechanical characterization of hydrogen effects on nickel-based alloy 725 under different metallurgical conditions. Journal of Materials Science & Technology **2023**, 135, 156-169, https://doi.org/10.1016/j.jmst.2022.07.006

Mazumder, J. T.; Lenka, T. R.; Tripathy, S. K.; **Vullum, P. E.**; Menon, P. S.; Lin, F.; Aberle, A. G. Investigation of structural, morphological, and optoelectronic properties of Ga-doped TiO_2 nanoparticles for electron transport layer in solar cell applications: An experimental and theoretical study. Journal of Physics and Chemistry of Solids **2022**, 161, 110410. https://doi.org/10.1016/j.jmst.2022.07.006

McEnroe, S. A.; Church, N.; Fabian, K.; Stange, M. F.; **van Helvoort, A. T. J.** An Enigma in Rock Magnetism: Can Microstructures in Magnetite Cause a Threefold Increase in the Efficiency of NRM Acquisition in the Stardalur Basalts? Geophysical Journal International **2022**, 231, 835–855 https://doi.org/10.1093/gji/ggac224

Nilsen, J. S.; **van Helvoort, A. T. J.** Composition Analysis by STEM-EDX of Ternary Semiconductors by Internal References. Microscopy and Microanalysis **2022**, 28, 61-69. https://doi.org/10.1017/S1431927621013672

** **Nylund, I.-E.**; Tsoutsouva, M.; Grande, T.; Meier, D. Observation of cation-specific critical behavior at the improper ferroelectric phase transition in $Gd_2(MoO_4)_3$. Physical Review Materials **2022**, 6, 034402. https://doi.org/10.1103/PhysRevMaterials.6.034402

Nylund, I. E.; Londal, N. S.; Walker, J.; **Vullum, P. E.**; Einarsrud, M. A.; Grande, T. Cation Disorder in Ferroelectric $Ba_4M_2Nb_{10}O_{30}$ (M = Na, K, and Rb) Tetragonal Tungsten Bronzes. Inorganic Chemistry, 61, 39, 15540–15546, https://doi.org/10.1021/acs.inorgchem.2c02266

Østli, E. R.; Ebadi, M.; Tesfamhret, Y.; Mahmoodinia, M.; Lacey, M. J.; Brandell, D.; Svensson, A. M.; Selbach, S. M.; Wagner, N. P. On the Durability of Protective Titania Coatings on High-Voltage Spinel Cathodes. ChemSusChem 2022, 15, 12. https://doi.org/10.1002/cssc.202200324

Pacakova, B.; **Vullum, P. E.**; Kirch, A.; Breu, J.; Miranda, C. R.; Fossum, J. O. Large bandgap insulating superior clay nanosheets. Mrs Bulletin, October **2022**, https://doi.org/10.1557/s43577-022-00349-8

Qian, F.; Jin, S.; Wan, D.; Li, W.; Cheng, X.; Sha, G.; Li, Y. Synergistic effects of Cd, Si and Cr additions on precipitation strengthening and thermal stability of dispersoids in AA3003 alloy. Materials Science and Engineering A 2022, 832, https://doi.org/10.1016/j.msea.2021.142422

** Roede, E. D.; Shapovalov, K.; Moran, T. J.; **Mosberg, A. B.**; Yan, Z.; Bourret, E.; Cano, A.; Huey, B. D.; **van Helvoort, A. T.J.**; Meier, D. The third dimension of ferroelectric domain walls. Advanced Materials **2022**, 34. http://dx.doi.org10.1002/adma.202202614

Sarasini, F.; Tirillò, J.; Lilli, M.; Bracciale, M. P.; **Vullum, P. E.**; Berto, F.; De Bellis, G.; Tamburrano, A.; Cavoto, G.; Pandolfi, F. Highly aligned growth of carbon nanotube forests with in-situ catalyst generation: A route to multifunctional basalt fibres. Composites Part B: Engineering **2022**, 243, 110136. https://doi.org/10.1016/j.compositesb.2022.110136

Shah, S.; Gopal, A.; **Thronsen, E.**; Hatzoglou, C.; Holmedal, B. Precipitation, mechanical

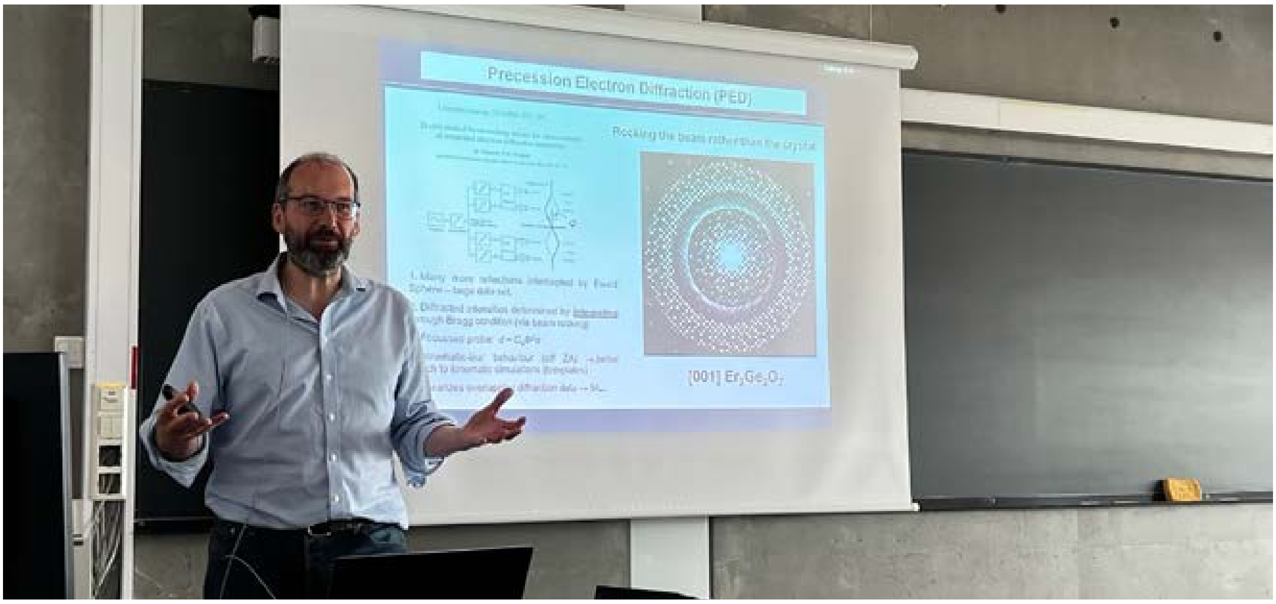


Image: *Prof. Paul Midgley explaining origins and fine details of precession electron diffraction during the ESTEEM3 workshop in Trondheim.*

properties and early slant ductile fracture in cyclic and naturally aged Al-Zn-Mg (-Cu) alloys. Materials & Design **2022**, 222, 111026. https://doi.org/10.1016/j.matdes.2022.111026

Shah, S.; **Thronsen, E.**; Hatzoglou, C.; **Wenner, S.**; **Marioara, C. D.**; **Holmestad, R.**; Holmedal, B. Effect of cyclic ageing on the early-stage clustering in Al-Zn-Mg(-Cu) alloys. Materials Science and Engineering A, **2022**, 846. https://doi.org/10.1016/j.msea.2022.143280

Sharma, A.; Raghunathan, K.; Solhaug, H.; Antony, J.; Stenvik, J.; Nilsen, A. M.; Einarsrud, M. A.; Bandyopadhyay, S. Modulating acrylic acid content of nanogels for drug delivery & biocompatibility studies. Journal of Colloid and Interface Science 2022, 607, 76-88. https://doi.org/10.1016/j.jcis.2021.07.139

** Shetty, S.; Gayen, M.; Agarwal, S.; **Chatterjee, D.**; Singh, A.; Ravishankar, N. Tuning Catalytic Activity in Ultrathin Bimetallic Nanowires via Surface Segregation: Some Insights. Journal of Physical Chemistry Letters **2022**, 13, 770-776. https://doi.org/10.1021/acs.jpclett.1c03852

Shishido, H.; Aruga, Y.; Murata, Y.; **Marioara, C.**; Engler, O. Evaluation of precipitates and clusters during artificial aging of two model Al-Mg-Si alloys with different Mg/Si ratios. Journal of Alloys and Compounds **2022**, 927, 10. https://doi.org/10.1016/j.jallcom.2022.166978

** Singh, R.; Scheinecker, D.; **Ludacka, U.**; Kotakoski, J. Corrugations in Free-Standing Graphene. Nanomaterials **2022**, 12, 8. https://doi.org/10.3390/nano12203562

Solem, C. K. W.; **Vullum, P. E.**; Ebadi, M.; Tranell, G.; Aune, R. E. The Role of CO_2 in the Oxidation-Protection of Mg-Containing Aluminum Alloys. Metallurgical and Materials Transactions B, **2022**, 53, 1967–1972. https://doi.org/10.1007/s11663-022-02524-3

Solem, C. K. W.; **Vullum, P. E.**; Tranell, G.; Aune, R. E. Heat Treatment of Mg-Containing Aluminum Alloys 5182 and 6016 in an Oxidizing Atmosphere with 4% CO_2. In Light Metals Symposia at the The-Minerals-Metals-Materials-Society (TMS) Annual Meeting, Anaheim, CA, **2022**, 587-593. https://doi.org/10.1007/978-3-030-92529-1_77

** Stefanek, M.; **Wenner, S.**; Borges, V.; Pinto, M.; Gomes, J. P.; Rodrigues, J.; Faria, I.; Pessanha, M. A.; Martins, F.; Sabino, R.; et al. Antimicrobial Resistance and Biofilms Underlying Catheter-Related Bloodstream Coinfection by Enterobacter cloacae Complex and Candida parapsilosis. Antibiotics-Basel **2022**, 11, 18. https://doi.org/10.3390/antibiotics11091245

Tafjord, J.; Regli, S. K.; Dugulan, A. I.; Ronning, M.; Rytter, E.; Holmen, A.; Myrstad, R.; Yang, J. Influence of temperature during pyrolysis of Fe-alginate: Unraveling the pathway towards highly active Fe/C catalysts. Applied Catalysis a-General 2022, 644, 12. https://doi.org/10.1016/j.apcata.2022.118834

Thronsen, E.; **Bergh, T.**; **Christiansen, E.**; **Thorsen, T. I.**; **van Helvoort, A.**; **Holmestad, R.** Scanning Precession Electron Diffraction and What We Get Out of Such Data in Studies of Aluminium Alloys. Microscopy and Microanalysis **2022**, 28, 2754-2755. https://doi.org/10.1017/S1431927622010364

Thronsen, E.; **Frafjord, J.**; **Friis, J.**; **Marioara, C. D.**; **Wenner, S.**; **Andersen, S. J.**; **Holmestad, R.** Studying GPI zones in Al-Zn-Mg alloys by 4D-STEM. Materials Characterization **2022**, 185. https://doi.org/10.1016/j.matchar.2021.111675

Thronsen, E.; Mørkeseth, H.; **Marioara, C. D.**; Minakuchi, K.; Katsumi, T.; **Marthinsen, K.**; Matsuda, K.; **Holmestad, R.** The Effect of Small Additions of Fe and Heavy Deformation on the Precipitation in an Al-1.1Mg-0.5Cu-0.3Si At. Pct Alloy. Metallurgical and Materials Transactions A Science **2022**, 53, 3296–3310. https://doi.org/10.1007/s11661-022-06744-9

Tomovic-Petrovic, S.; Gulbrandsen-Dahl, S.; **Wenner, S.** Corrosion susceptibility of the $CuZn_{35}Pb_{1,5}AlAs$ (CW625N) brass alloy in various environments. In Copper Alloys 2022 - Conference Proceedings, Deutsches Kupferinstitut Berufsverband e. V., **2022**, 52-55.

Tvedt, H.; **Marioara, C. D.**; **Thronsen, E.**; **Hell, C.**; **Andersen, S. J.**; **Holmestad, R.** AutomAl 6000: Semi-automatic structural labelling of HAADF-STEM images of precipitates in Al-Mg-Si(-Cu) alloys. Ultramicroscopy **2022**, 236, 113493. https://doi.org/10.1016/j.ultramic.2022.113493

Viola, G.; Musumeci, G.; Mazzarini, F.; Tavazzani, L.; Torgersen, E.; van der Lelij, R.; Aldega, L. Structural characterization and K-Ar illite dating of reactivated, complex and heterogeneous fault zones: Lessons from the Zuccale Fault, Northern Apennines. EGUsphere 2022, 2022, 1-35. DOI: 10.5194/egusphere-2022-229.

Wimmer, A.; Hammerle, A.; **Marioara, C. D.** Precipitation Hardening in Innovative Processing of 6xxx Aluminum Alloys. In Light Metals Symposia at the The-Minerals-Metals-Materials-Society (TMS) Annual Meeting, Anaheim, CA, **2022**; 245-250. https://doi.org/10.1007/978-3-030-92529-1_34

Wu, S.; Søreide, H. S.; Chen, B.; Bian, J.; Yang, C.; Li, C.; Zhang, P.; Cheng, P.; Zhang, J.; Peng, Y.; et al. Freezing solute atoms in nanograined aluminum alloys via high-density vacancies. Nature Communications 2022, 13. https://doi.org/10.1038/s41467-022-31222-6

** **Ånes, H. W.**; **van Helvoort, A. T. J.**; **Marthinsen, K.** Correlated subgrain and particle analysis of a recovered Al-Mn alloy by directly combining EBSD and backscatter electron imaging. Materials Characterization **2022**, 193, 9. https://doi.org/10.1016/j.matchar.2022.112228

Schultheiss, J.; Xue, F.; Roede, E.; **Ånes, H. W.; Danmo, F. H.; Selbach, S. M.; Chen, L.-Q.; Meier, D. Confinement-Driven Inverse Domain Scaling in Polycrystalline $ErMnO_3$. Adv. Mater. **2022**, 34, 2203449. https://doi.org/10.1002/adma.202203449

** Seniz, U. Formation of Calcium Phosphate Minerals in the Presence of Fetuin-A. Celal Bayar University Journal of Science, 2022, 18, 233-238. https://doi.org/10.18466/cbayarfbe.1038019

ACCEPTED/PUBLISHED ONLINE/IN PRESS

Frafjord, J.; **Ringdalen, I. G.**; **Holmestad, R.**; **Friis, J.** On the atomic structure of the β” precipitate by density functional theory. Computational Materials Science **2023**, 217, 111871. https://doi.org/10.1016/j.commatsci.2022.111871

Gazizov, M.; Belyakov, A.; **Holmestad, R**.; Gazizova, M. Y.; Krasnikov, V.; Bezborodova, P.; Kaibyshev, R. The deformation behavior of the {111} Al plates in an Al-Cu-Mg-Ag alloy. Acta Materialia **2023**, 243, 118534. https://doi.org/10.1016/j.actamat.2022.118534

Hunnestad, K. Schultheiss, J. Mathisen, A. Hatzoglou, C., **van Helvoort, A. T. J.**, and Meier, D. Quantitative 3D mapping of chemical defects at charged grain boundaries in a ferroelectric oxide, arXiv preprint arXiv:2212.07924. https://doi.org/10.48550/arXiv.2212.07924

Ma, H., Zheng, X., Zhang, H., Ma, G., Zhang, W., Jiang, Z., Chen, D., Atomic Cu-N-P-C Active Complex with Integrated Oxidation and Chlorination for Improved Ethylene Oxychlorination. Adv. Sci. 2023, 2205635. https://doi.org/10.1002/advs.202205635

Nordahl, G.; Jones, L.; **Christiansen, E. F.; Hunnestad, K. A.; Nord, M.** Correcting for probe wandering by precession path segmentation. arXiv preprint arXiv:2211.11413 **2022**.

Nordahl G. and **Nord M.**, Improving magnetic STEM-differential phase contrast imaging using precession, Microscopy and Microanalysis, **2023**, published online, https://doi.org/10.1093/micmic/ozad001

Nylund, I.-E.; Zeiger, C. R.; **Peng, D.**; **Vullum, P. E.**; Walker, J.; Einarsrud, M.-A.; Grande, T. Centrosymmetric Tetragonal Tungsten Bronzes $A_4Bi_2Nb_{10}O_{30}$ (A = Na, K, Rb) with a Bi 6s Lone Pair. Chemistry of Materials **2023**, 35, 1, 17. https://doi.org/10.1021/acs.chemmater.2c01944

Reisecker, V.; Flatscher, F.; Porz, L.; Fincher, C.; Hanghofer, I.; Hennige, V.; Linares-Moreau, M.; Falcaro, P.; Ganschow, S.; **Wenner, S.** The impact of pulsed current waveforms on Li dendrite initiation and propagation in solid-state Li batteries. ChemRxiv, **2022**. https://doi.org/10.26434/chemrxiv-2022-6dpzs

** **Ånes, H. W.**; **van Helvoort, A. T.**; **Marthinsen, K**. Orientation dependent pinning of (sub) grains by dispersoids during recovery and recrystallization in an Al-Mn alloy, arXiv, https://doi.org/10.48550/arXiv.2212.03527

POPULAR SCIENCE

Christiansen, Emil Frang; **Holmestad, Randi**. Nano - en reise inn i et usynlig univers. Hvem vant Kavliprisen i nanovitenskap og hvorfor? NTNU kveld; September 2022.

Bazilchuk, Nancy Reney; Frøland, Hans Otto; **Holmestad, Randi**; Brandtzæg, Svein Richard. Hermann Göring's Luftwaffe and the $6 billion deal. https://play.acast.com/s/63-degrees-north/hermann-gorings-lu [Internett] March 2022.

CONFERENCE CONTRIBUTIONS (A SELECTION)

Asheim, Karina; Vullum, Per Erik; Holsen, Ingrid; Wagner, Nils Peter; Renman, Viktor; Blanco, Maria Valeria; Mæhlen, Jan Petter; Svensson, Ann Mari. Performance of micron-sized silicon anodes and NMC cathodes in full cells with LiFSI-based electrolytes. International Society of Electrochemistry, 32nd topical meeting; June 2022.

Bergh, Tina; Fyhn, Hursanay; Sandnes, Lise; Blindheim, Jørgen; Grong, Øystein; Holmestad, Randi; Berto, Filippo; Vullum, Per Erik. Hybrid metal extrusion & bonding for multi-material welding of aluminium alloys to copper, steel, and titanium. ICAA 18; Toyama, Japan, September 2022.

Bergh, Tina; Thronsen, Elisabeth; Christiansen, Emil Frang; Frafjord, Jonas; Thorsen, Tor Inge; Nordahl, Gregory; Sørhaug, Jørgen A; Holmestad, Randi; Van Helvoort, Antonius., Scanning precession electron diffraction for phase mapping. Electron diffraction for solving engineering problems; June 2022.

Celotto, Ambra; Grong, Øystein; Holmestad, Randi; Sørhaug, Jørgen A; Vullum, Per Erik; Wan, Di; Berto, Filippo. Unconventional use of Ga+ FIB and Xe^{+} Plasma-FIB for performing and investigating microscale cold-welding of dissimilar aluminium alloys. 1st National Junior Scientist Research Conference in Nanoscience and Nanotechnology; November 2022.

Crout, Phillip; Francis, Carter; Madsen, Jacob; MacArthur, Katherine; Nord, Magnus; de la Peña, Francisco; Slater, Thomas. ePSIC HyperSpy workshop 2022. May 2022.

Holmestad, Randi. Crash course in TEM (with examples from microstructure and precipitates in Al alloys). Global Engineering lectures; (online) Toyama, Japan. October 2022.

Holmestad, Randi; Thronsen, Elisabeth; Bergh, Tina; Christiansen, Emil; Thorsen, Thor Inge; van Helvoort, Antonius. Scanning Precession Electron Diffraction and What We Get Out of Such Data in Studies of Aluminium Alloys. M&M, Portland, USA, August 2022.

Holmestad, Randi; van Helvoort, Antonius; Thronsen, Elisabeth; Bergh, Tina; Chatterjee, Dipanwita; Christiansen, Emil Frang. Scanning Precession Electron Diffraction – experimental setup, data analysis and results obtained in Trondheim. Imaging, Diffraction and Crystallography – where John Spence's Legacy Leads Us; Phoenix, USA; October 2022.

Hunnestad, Kasper Aas; Das, Hena; Hatzoglou, Constantinos; Holtz, Megan E.; Brooks, Charles M.; van Helvoort, Antonius T. J.; Schlom, Darrell G.; Mundy, Julia A.; Meier, Dennis Gerhard. Quantifying interfacial defect accumulations in oxide superlattices. Atom Probe Tomography & Microscopy; October 2022.

Hunnestad, Kasper Aas; Das, Hena; Hatzoglou, Constantinos; Holtz, Megan, E.; Brooks, Charles M.; van Helvoort, Antonius; Schlom, Darrel G.; Mundy, Julia A.; Meier, Dennis. Quantitative 3D imaging of oxygen defects in multiferroic $(LuFeO_3)_9/(LuFe_2O_4)1$ superlattices. MRS Fall Meeting; November 2022.

Linnerud, Marthe; Høvik, Jens; Nord, Magnus. Towards customizable TEM membranes for in-situ and in-operando STEM studies of epitaxial thin films and nanostructures. The 1st TNNN Conference 2022; November 2022.

Marthinsen, Knut; Ånes, Håkon Wiik; Huang, Ke; van Helvoort, Antonius. Particle effects during recrystallization of aluminium alloys and their influence on the texture evolution. The 8th International Conference on Solid -> Solid Phase Transformations in Inorganic Materials (PTM); June 2022.

Marthinsen, Knut; Ånes, Håkon Wiik; van Helvoort, Antonius. Correlated sub-grain and particle analysis during recovery and recrystallization of an AlMn-alloy. The 6th International Symposium on Advanced Structural Materials 111-program symposium, Chongqing University;. November 2022.

Marthinsen, Knut; Ånes, Håkon Wiik; van Helvoort, Antonius. Orientation dependent Zener-pinning of sub-grains during recovery and recrystallization of an Al-Mn alloy. The 16th International Conference on the Physical Properties and Application of Advanced Materials (ICPMAT); November 2022.

Mukherjee, Anjan; Ren, Dingding; Vullum, Per Erik; Huh, Junghwan; Fimland, Bjørn Ove Myking; Weman, Helge. Axial GaAs/AlGaAs Nanowire Solar Cell on Si with Ultra-High Power-per-Weight Ratio. III-V Semiconductors for Energy Conversion Technologies; May 2022.

Nordahl, Gregory; Jones, Lewys; Christiansen, Emil; Hunnestad, Kasper Aas; Nord, Magnus Kristofer. Precession segmentation to increase SPED image resolution. PICO2022; Belgium; May 2022.

Nord, Magnus. Analysing very large electron microscopy datasets using Python and JupyterLab. TNNN Conference 2022; November 2022.

Nord, Magnus. Scanning Transmission Electron Microscopy: studying magnetic fields in materials. Stockholm University; May 2022.

Nord, Magnus. Writing and sustaining analysis tools for large and complex microscopy data. 16th Multinational Congress on Microscopy; Germany, September 2022.

Nordahl, Gregory; Dagenborg, Sivert Johan Vartdal; Nord, Magnus Kristofer. Utilizing neural networks for nanoscale magnetic field mapping with STEM-DPC. TNNN; November 2022.

Pacáková, Barbara; Vullum, Per Erik; Breu, Josef; Fossum, Jon Otto. Large band gap of insulator clay nanosheets. Virtual MRS Spring meeting; May 2022.

Pacáková, Barbara; Vullum, Per Erik; Breu, Josef; Fossum, Jon Otto. Large band gap of insulator clay nanosheets. APS March Meeting 2022 Chicago; March 2022.

Ren, Dingding; Nilsen, Tron Arne; Nilsen, Julie Stene; Ahtapodov, Lyubomir; Mukherjee, Anjan; Li, Yang; van Helvoort, Antonius; Weman, Helge; Fimland, Bjørn Ove Myking. MBE Growth of GaAsSb/GaAs Multiple Quantum Disk Nanowires on Few-layer Graphene for Photonic Applications. 22nd International Conference on Molecular Beam Epitaxy; September 2022.

Rosnes, Andreas; Lysne, Hogne; Christiansen, Emil Frang; Reenaas, Turid; Holmestad, Randi. Differentiating Polyamorphous SiO_2 by SPED, PDF and Blind Source Separation Algorithm (online), SCANDEM 2022; June 2022.

Ryggetangen, Oskar; Marioara, Calin Daniel; Andersen, Sigmund Jarle; Wenner, Sigurd; Holmestad, Randi; Mihara, Mami. SPED studies of quasicrystalline phases in Al-Mg-Cu-Ag alloys. ICAA 18 – Toyama, Japan, September 2022.

Sørhaug, Jørgen A; Celotto, Ambra; Grong, Øystein; Vullum, Per Erik; Holmestad, Randi. Scanning transmission electron microscopy investigations on an Al-Al joint interface made using FIB-assisted cold welding. 1st National Junior Scientist Research Conference in Nanoscience and Nanotechnology; November 2022

Thronsen, Elisabeth; Marioara, Calin Daniel; Wenner, Sigurd; Frafjord, Jonas; Friis, Jesper; Andersen, Sigmund Jarle; Holmestad, Randi. The evolution of precipitates in an Al-Zn-Mg alloy. ICAA18; Toyama, Japan, September 2022.

Thronsen, Elisabeth; Marioara, Calin Daniel; Wenner, Sigurd; Frafjord, Jonas; Friis, Jesper; Andersen, Sigmund Jarle; Holmestad, Randi. 4D-STEM used to study GPI zones in Al-Zn-Mg alloys. ICAA18, International conference of aluminium alloys; September 2022.

Sikorski, Pawel; van Helvoort, Antonius; Christiansen, Emil; Vorwald, Charlotte E; Gonzalez-Fernandez, Tomas; Reseland, Janne Elin; Solberg, Lene Bergendal; Leach, J. Kent. Spheroid-based model system to study bone mineralization in 3D. EMBL IBEC Conference on Engineering Multicellular Systems, June 2022.

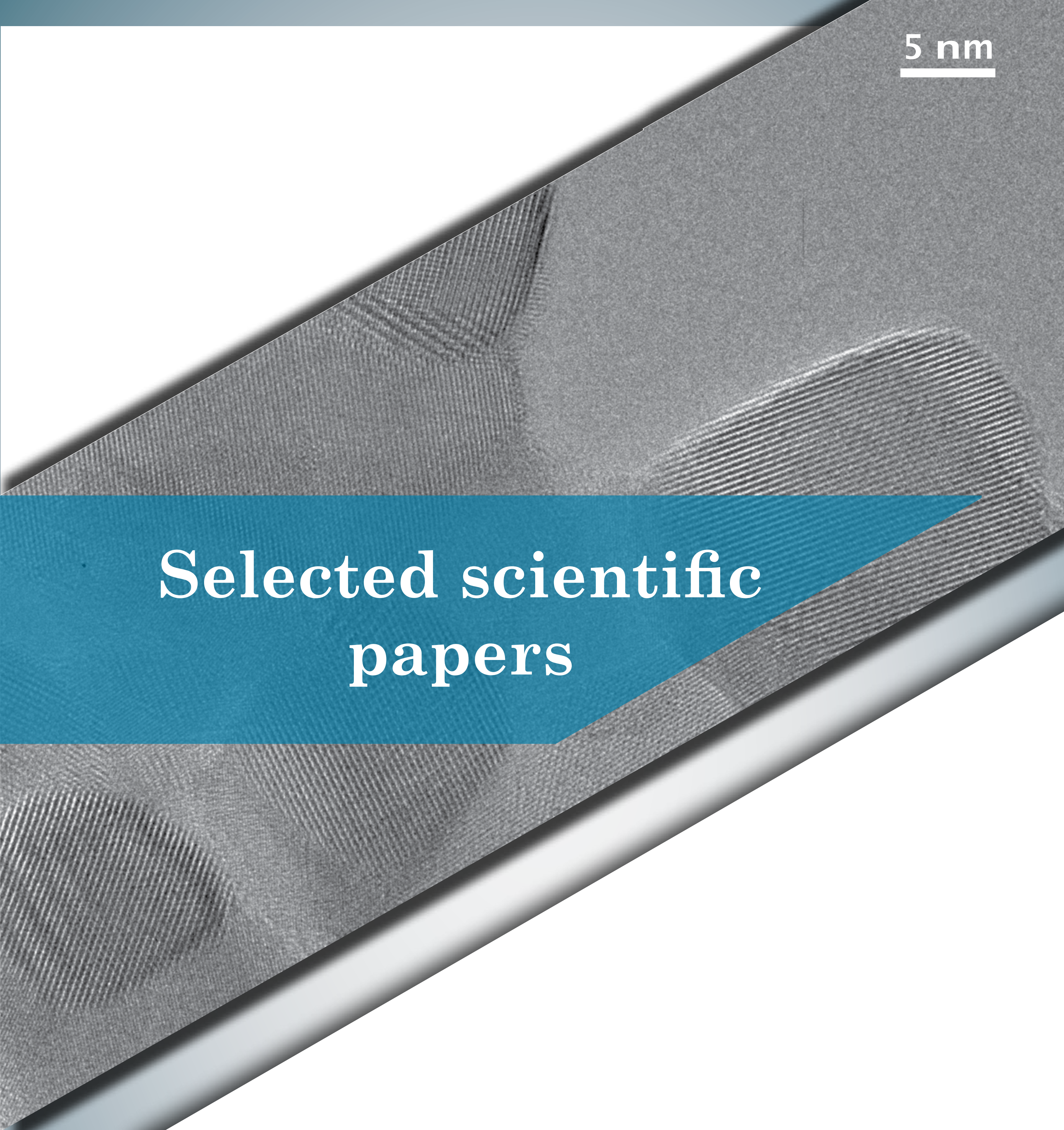
Zeiger, Caren Regine; Nylund, Inger-Emma; Peng, Ding; Vullum, Per Erik; Walker, Julian; Einarsrud, Mari-Ann; Grande, Tor. Exploring the Role of Bismuth 6s Lone Pair in Tetragonal Tungsten Bronzes $A_4Bi_2Nb_{10}O_{30}$ (A=Na,K,Rb). ISAF 2022 - International Symposium on Applications of Ferroelectrics; June 2022.

MASTER'S THESES

- **Rajith Aravinth**, Fresnel imaging of ferromagnetic domain wall motion using fast pixelated electron detector (Supervisor Magnus Nord)
- **Julie Marie Bekkevold**, Improving in-situ dynamic magnetic characterisation of artificial spin ice ensembles using STEM, (Supervisor Magnus Nord)
- **Joseph Vincent Broussard**, Analyzing and improving open-source template matching for orientation mapping based on SPED data (Supervisor Ton van Helvoort)
- **Petter Lervik**, Effect of natural ageing temperature and time on precipitation in 6xxx aluminium alloys (Supervisor Randi Holmestad)
- **Martin Lesjø**, The effect of cooling rate from solution heat treatment on ductility in Al-Mg-Si crash box alloys (Supervisor Knut Marthinsen)
- **Marthe Linnerud**, Building a framework for domain characterisation of $K_3Nb_3B_2O_{12}$ by electron diffraction and transmission electron microscopy, (Supervisor Ton van Helvoort)
- **Andreas Rosnes**, EM study of Cr, N Co-doped TiO_2 thin film (Supervisor Randi Holmestad)
- **Armand Sepehri**, Transmission Electron Microscopy Characterization of Degradation Mechanisms in Lithium-ion Batteries (Supervisor Per Erik Vullum)
- **Mari Sofie Skomedal**, Improving quantitative EDS of III-V heterostructure semiconductors in low voltage STEM (Supervisor Ton van Helvoort)
- **Elisabeth S. Thrane**, Effect of natural ageing on Al-Cu-Mg-Si alloy for zippers: a transmission electron microscopy study (Supervisor Randi Holmestad)
- **Hedda Øye**, Investigation of the effects of additions of vanadium and titanium to 6005 and 6008 aluminium alloys (Supervisor Randi Holmestad)

PROJECT THESES

- **Susanne Boucher**, A comparative characterization of LSMO/mica and LSMO/STO(111) using TEM, (S)TEM, EELS and EDS (Supervisor Magnus Nord)
- **Kristin Frøystein**, SPED Data analysis of precipitates in aluminium alloys (Supervisor Randi Holmestad)
- **Erik Gaupseth**, Structural characterization of Cr,N-doped TiO_2 thin films (Supervisor Randi Holmestad)
- **Håkon Longva Korsvold**, TEM-studies of the microstructure in a hybrid metal extrusion welded Al-Mg-Si-Cu alloy (Supervisor Randi Holmestad)
- **Anders Christian Mathisen**, Investigations of Polycrystalline Ferroelectric $ErMnO_3$ by TEM", (Supervisor Ton van Helvoort)
- **Brynjar Morka Mæhlum**, Improving qualitative and quantitative SEM EDS analysis with open-source data processing (Supervisor Ton van Helvoort)
- **Hedda Christine Soland**, Exploring Magnetic Domain Formation in Permalloy Nanoelements using ADF STEM-DPC (Supervisor Magnus Nord)
- **Mats Topstad**, TEM characterization of ferroelectric $BaMgF_4$ (Supervisor Ton van Helvoort)
- **Kristian Tveitstøl**, Using EELS to measure local conductivity (Supervisor Randi Holmestad)



Selected scientific papers

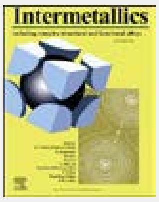
Image: HRTEM/lattice image of SnO_2 for battery anodes, by S. Wenner.

On intermetallic phases formed during interdiffusion between aluminium alloys and stainless steel

Tina Bergh, Siri Marthe Arbo, Anette Brocks Hagen, Jørgen Blindheim, Jesper Friis, Muhammad Zeeshan Khalid, Inga Gudem Ringdalen, Randi Holmestad, Ida Westermann, Per Erik Vullum

Abstract

One of the major challenges in welding of aluminium (Al) alloys and steels is the growth of brittle intermetallic phases, which depends on the thermomechanical processing history and the alloying elements. This work focuses on the intermetallic phase layers formed in roll bonded composites of Al alloy 6082 and stainless steel 316L after interdiffusion at temperatures in the range of 400 – 550 ºC. Scanning and transmission electron microscopy characterisation showed that during interdiffusion, an α_c-$Al_{15}(Fe,Cr,Mn)_3Si_2$ phase layer formed first, before a discontinuous layer of τ_1-$FeNiAl_9$ formed at the Al-α_c interface. Subsequently, a layer of θ-Fe_4Al_{13} and τ_{11}- Al_5Fe_2Si formed at the α_c-steel interface, followed by a layer of η-Fe_2Al_5 with precipitates rich in Cr and Si or Ni. Nanoindentation and density functional theory calculations were performed to assess the mechanical properties of the formed phases. Miniature tensile testing confirmed that the bond strength decreased as the thicknesses of brittle phase layers increased. Further, it was found that the growth rate of the total intermetallic phase layer was significantly reduced for the high alloyed 6082-316L composites compared to unalloyed reference composites of 1080-S355. Altogether, this work provides insight into the combined effects of the alloying elements Si, Mn, Cr and Ni on the formation, growth and mechanical properties of the interfacial intermetallic phases in Al-steel joints.



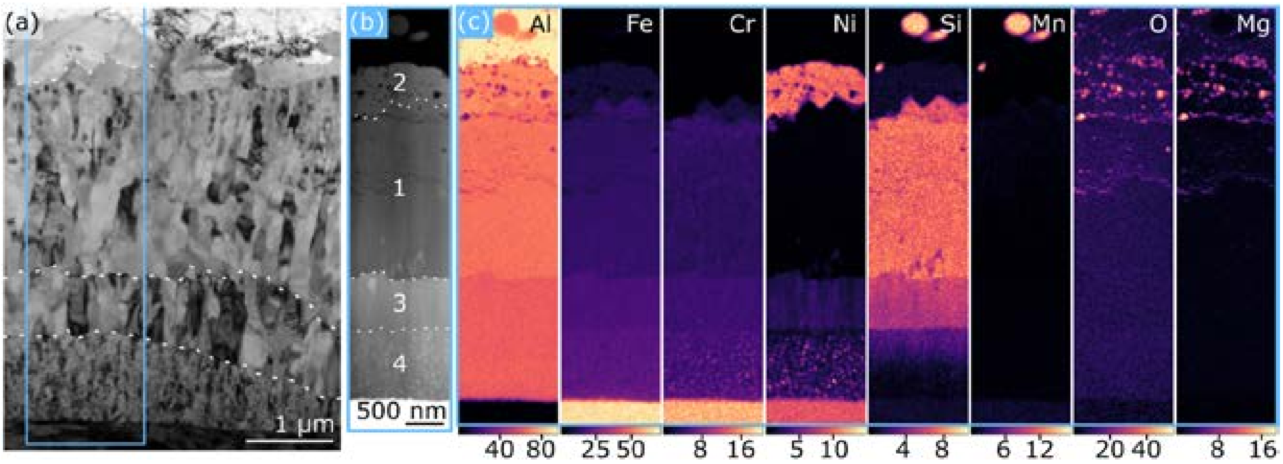


Fig. 6. The interfacial IMP layers after 60 min at 550 ºC. (a) Overview BF-TEM image and (b) HAADF-STEM image of the region outlined in blue in (a), where the dashed white lines mark borders between each IMP layer (Layers 1 – 4). (c) Element maps (showing at.%) based on STEM EDS of the region shown in (b).

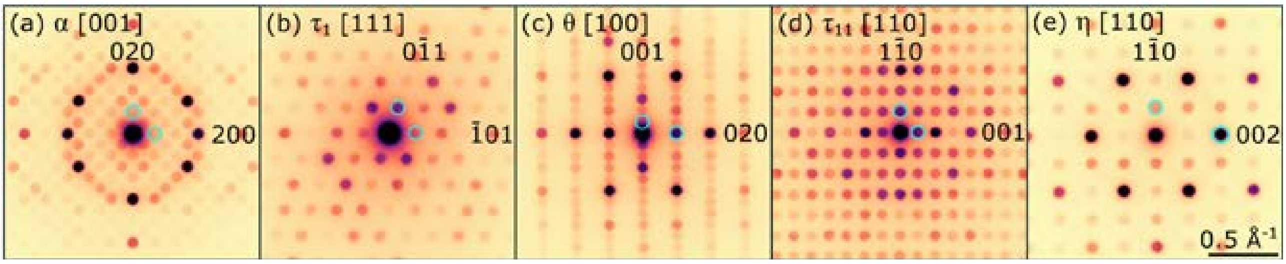


Fig. 7. Precession electron diffraction (PED) patterns from the identified IMPs. (a) α_c-$Al_{15}(Fe,Cr,Mn)_3Si_2$ at zone axis [001], (b) τ_1-$FeNiAl_9$ at zone axis [111], (c) θ-Fe_4Al_{13} at zone axis [100], (d) τ_{11}-Al_5Fe_2Si at zone axis [110] and (e) η-Fe_2Al_5 at zone axis [110]. In each pattern, hkl indices are given for the two spots marked by cyan circles. All patterns share the scale bar shown in (e).

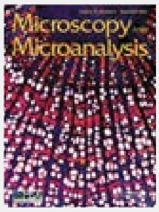
Bergh, T., et al., On intermetallic phases formed during interdiffusion between aluminium alloys and stainless steel. Intermetallics, **2022**. 142. https://doi.org/10.1016/j.intermet.2021.107443

Composition Analysis by STEM-EDX of Ternary Semiconductors by Internal References

Julie Stene Nilsen and Antonius T. J. van Helvoort

Abstract

A practical method to determine the composition within ternary heterostructured semiconductor compounds using energy-dispersive X-ray spectroscopy in scanning transmission electron microscopy is presented. The method requires minimal external input factors such as userdetermined or calculated sensitivity factors by incorporating a known compositional relationship, here a fixed stoichiometric ratio in III-V compound semiconductors. The method is demonstrated for three different systems; AlGaAs/GaAs, GaAsSb/GaAs, and InGaN/GaN with three different specimen geometries and compared to conventional quantification approaches. The method incorporates absorption effects influencing the composition analysis without the need to know the thickness of the specimen. Large variations in absorption conditions and assumptions regarding the reference area limit the accuracy of the developed method.



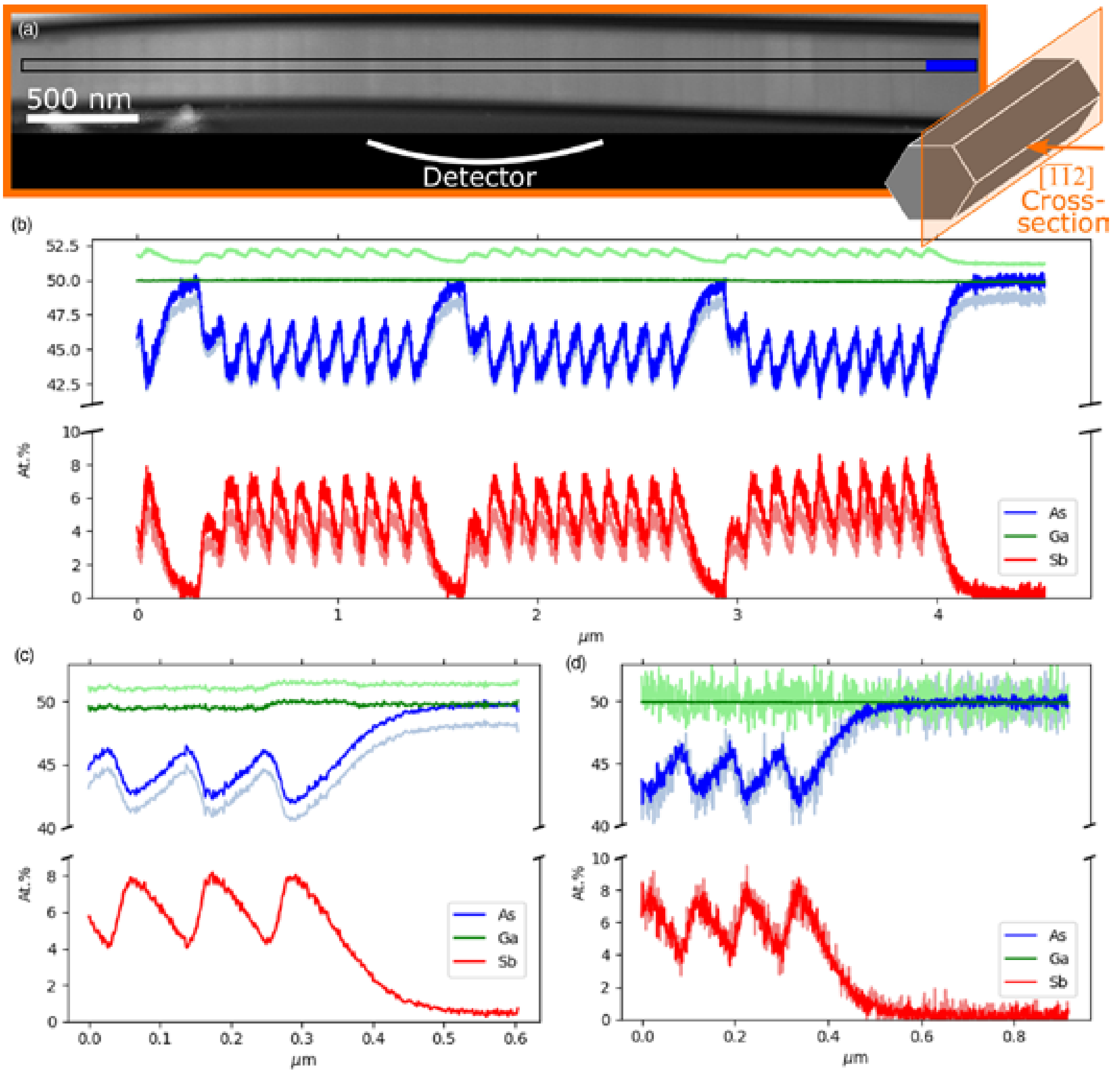


Fig. 3. (a) HAADF STEM image of parts of the GaAs/GaAs$_{1-x}$Sb$_x$ nanowire showing the top three superlattices. The scan area and the reference area (blue) are indicated. The inset on the right depicts the specimen geometry. (b) Internal determination of the composition of three superlattices, compared to quantification using calculated k-factors (lighter color) at a tiltx = -14.2° away from the detector. (c) Internal determination of the composition of the top three GaAsSb inserts of the top superlattice compared to calculated k-factors at a tiltx = +15° towards the detector. (d) The same data as in (b), cropped to about the same area as in (c), and compared to the nondenoised data (lighter color).

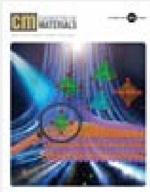
Nilsen, J.S. and A.T.J. van Helvoort, Composition Analysis by STEM-EDX of Ternary Semiconductors by Internal References. Microscopy and Microanalysis, **2022**. 28(1): p. 61-69. https://doi.org/10.1017/S1431927621013672

Centrosymmetric Tetragonal Tungsten Bronzes $A_4Bi_2Nb_{10}O_{30}$ (A = Na, K, Rb) with a Bi 6s Lone Pair

Inger-Emma Nylund, Caren Regine Zeiger, Ding Peng, Per Erik Vullum, Julian Walker, Mari-Ann Einarsrud, and Tor Grande

Abstract

A first-principles study of the tetragonal tungsten bronze (TTB) $K_4Bi_2Nb_{10}O_{30}$ has suggested that the Bi 6s lone pair causes in-plane polarization (within the a-b plane), corresponding to the one found in $Pb_5Nb_{10}O_{30}$ (PN), in contrast to the out-of-plane polarization (along c) found in most TTBs. Replacing PN with KBN potentially opens for a leadfree analogue to morphotropic phase boundaries known in TTBs based on PN. Here, we report on the synthesis and properties of $A_4Bi_2Nb_{10}O_{30}$ (ABN, A = Na, K, Rb) with the objective to determine the structure and electrical properties, paying particular attention to the role of the Bi 6s lone pair. The ABN materials were synthesized via conventional solid-state synthesis in a two-step process. Convergent-beam electron diffraction demonstrated a centrosymmetric tetragonal space group for the two compounds KBN and RBN, and ferroelectric polarization-electric field measurements confirmed the lack of hysteretic behavior in line with the observed centrosymmetric symmetry. Non-ambient powder X-ray diffraction demonstrated the signature of a phase transition for KBN and RBN, as several weak satellite reflections vanished during heating and reappeared upon cooling. Dielectric spectroscopy supported the observation of an anomaly due to the presence of a weak maximum in the electrical permittivity at temperatures corresponding to the disappearance of the satellite reflections. Possible explanations for the absence of polarization in ABN TTBs are discussed with particular attention to the suppression of the 6s2 lone pair effect of Bi and the size of A-site cations in the TTB crystal structure.



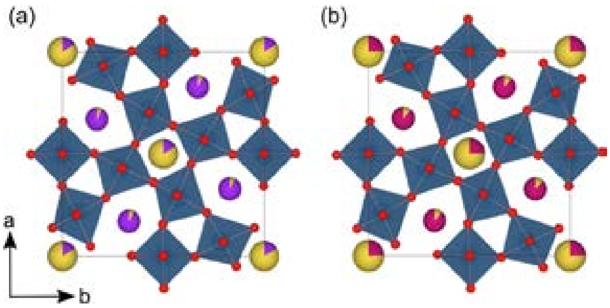


Figure 2. Ambient crystal structure of (a) KBN and (b) RBN obtained by Rietveld refinement using space group P4/mbm. Nb is blue, O is red, Bi is yellow, and the alkali metals K and Rb are purple and pink, respectively. Cake diagrams of the A-site cations indicate the degree of cation disorder.

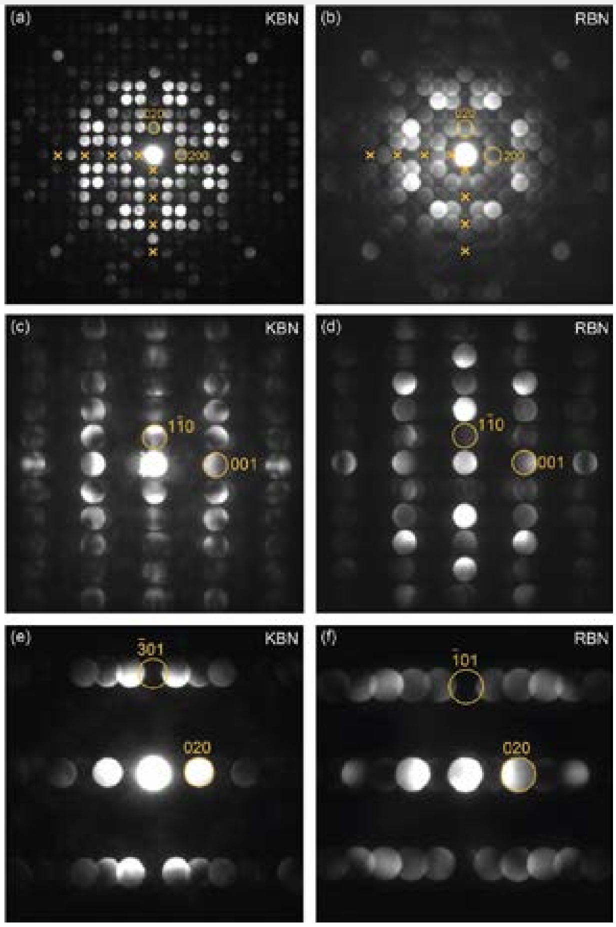


Figure 4. [001] CBED patterns from (a) KBN and (b) RBN with 4mm symmetry present in both cases. Crosses mark kinematically forbidden but dynamically allowed reflections. [110] CBED patterns from (c) KBN and (d) RBN showing that 2mm symmetry is present in both cases. (e) [103] CBED pattern from KBN and (f) [101] CBED pattern from RBN also displaying that 2mm symmetry is present in both cases.

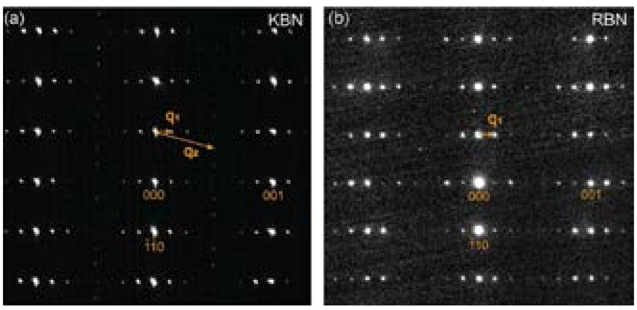


Figure 6. [110] SAD patterns from (a) KBN and (b) RBN with diffraction spots visible due to incommensurate modulations. KBN exhibits two modulations q_1 and q_2 (q_2 is most clearly visible in the upper left corner), whereas RBN only exhibits the q_1 modulation.

Nylund, I.E., et al., Centrosymmetric Tetragonal Tungsten Bronzes $A_4Bi_2Nb_{10}O_{30}$ (A = Na, K, Rb) with a Bi 6s Lone Pair. Chemistry of Materials, **2022**. https://doi.org/10.1021/acs.chemmater.2c01944

Studying GPI zones in Al-Zn-Mg alloys by 4D-STEM

E. Thronsen, J. Frafjord, J. Friis, C.D. Marioara, S. Wenner, S.J. Andersen, R. Holmestad

Abstract

A new methodology has been developed to study the fine details of GP zones in age-hardenable aluminium alloys. It is complementary to atomic resolution high-angle annular dark-field scanning transmission electron microscopy imaging, and combines scanning precession electron diffraction with diffraction simulations. To evaluate the method, data was collected from an Al-Zn-Mg alloy in a condition with a dense distribution of GPI zones. Diffraction patterns were recorded in the <001> Al orientation, capturing GPI zones in three projections: along the unique [001]GPI axis, and along the two other mutually orthogonal orientations. The GPI zones viewed along [001]GPI revealed how the truncated octahedron units of the GPI zones were connected in multi-unit GP zones, while the two orientations normal to [001]GPI highlight the internal structure. The stability of the atomic models developed based on the experimental results was verified by density functional theory calculations. Possible explanations for the absence of polarization in ABN TTBs are discussed with particular attention to the suppression of the 6s2 lone pair effect of Bi and the size of A-site cations in the TTB crystal structure.

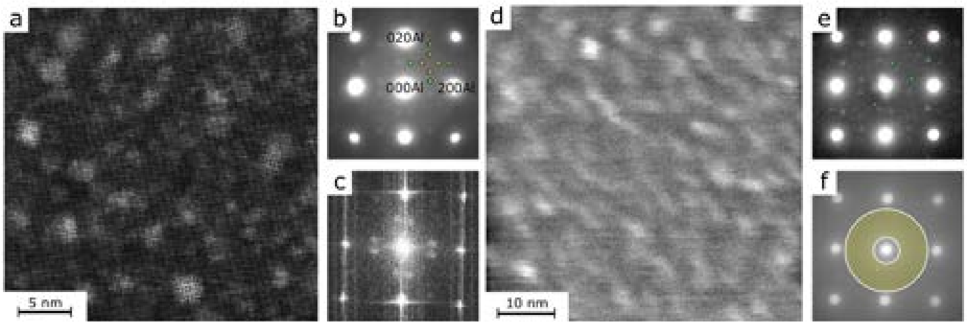


Fig. 4. a: HAADF-STEM showing a high density of GPI zones. b: SADP of a similar region as in a. c: fast Fourier transform of a. d: VDF image from a similar region as in a. e: Constructed pattern by maximum pixel values in the data stack. f: Average PED pattern illustrating the placement of the virtual aperture in the SPED data stack.

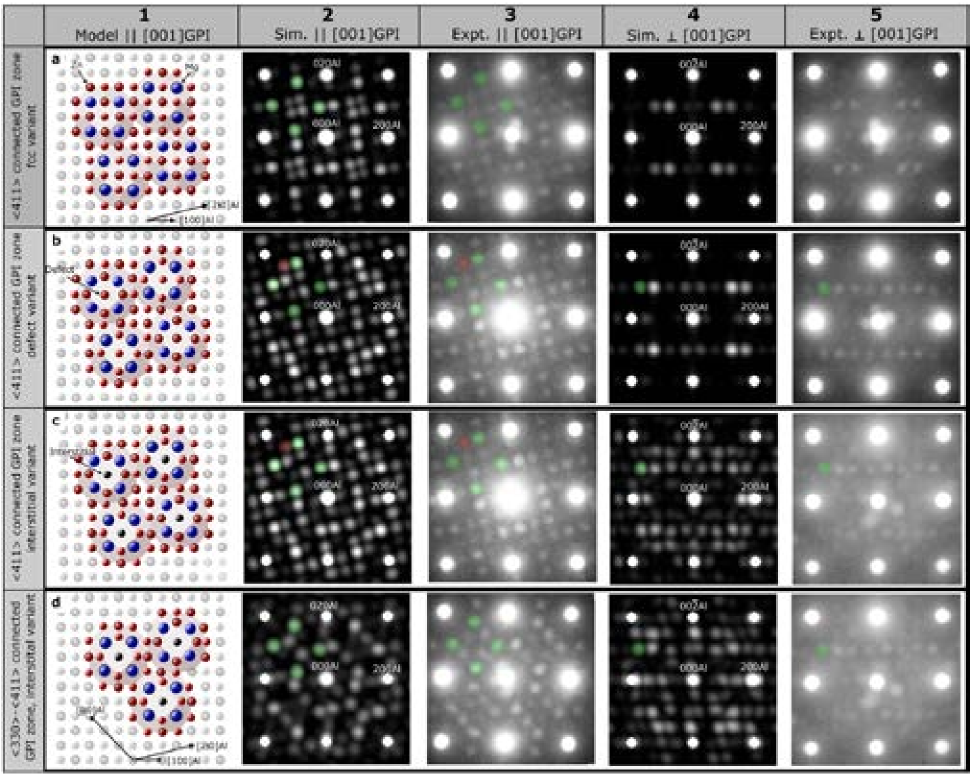


Fig. 5. GPI zone models and corresponding simulated NBD- and experimental PED patterns viewed parallel and normal to [001]GPI. Columns: (1) models along [001]GPI. (2,3) corresponding simulated patterns and selected experimental patterns, (4,5) simulated patterns and selected experimental patterns in one of the <001>Al zone axes normal to [001]GPI. Rows: (a-c) <411>connected GPI zones with all solutes on fcc, with defected central column or with 3/2 occupancy in central columns, respectively, (d) <330>-<411>connected GPI zone with 3/2 occupancy in central columns. Note that all the simulations are NBD patterns, while the experimental patterns are PED patterns.

Thronsen, E., et al., Studying GPI zones in Al-Zn-Mg alloys by 4D-STEM. Materials Characterization, **2022**. 185. https://doi.org/10.1016/j.matchar.2021.111675

AutomAl 6000: Semi-automatic structural labelling of HAADF-STEM images of precipitates in Al–Mg–Si(–Cu) alloys

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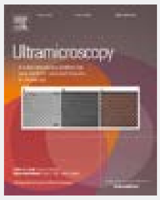
Abstract



When the Al–Mg–Si(–Cu) alloy system is subjected to age hardening, different types of precipitates nucleate depending on the composition and thermomechanical treatment. The main hardening precipitates extend as needles, laths or rods along the <100> directions in the aluminium matrix. It has been found that the structures of all metastable precipitates may be generalized as stacks of <100> columns, where most of these columns are replaced by solute elements. In the precipitates, a column relates to neighbour columns by a set of simple structural principles, which allows identification of species and relative longitudinal displacement over the (100) cross-section.

Aberration-corrected high-angle annular dark field scanning transmission electron microscopy (HAADF-STEM) is an important tool for studying such precipitates. With the goal of analysing atomic resolution HAADF-STEM images of precipitate cross-sections in the Al–Mg–Si(–Cu) system, we have developed the standalone software AutomAl 6000, which features a column characterization algorithm based on the symbiosis of a statistical model and the structural principles formulated in a digraph-like framework. The software can semiautonomously determine the 3D column positions in the image, as well as column species. In turn, AutomAl 6000 can then display, analyse and/or export the structure data.

This paper describes the methodology of AutomAl 6000 and applies it on three different HAADF-STEM images, which demonstrate the methodology. The software, as well as other resources, are available at http://automal.org. The source code is also directly available from https://github.com/Haawk666/AutomAl-6000. Possible explanations for the absence of polarization in ABN TTBs are discussed with particular attention to the suppression of the 6s2 lone pair effect of Bi and the size of A-site cations in the TTB crystal structure.

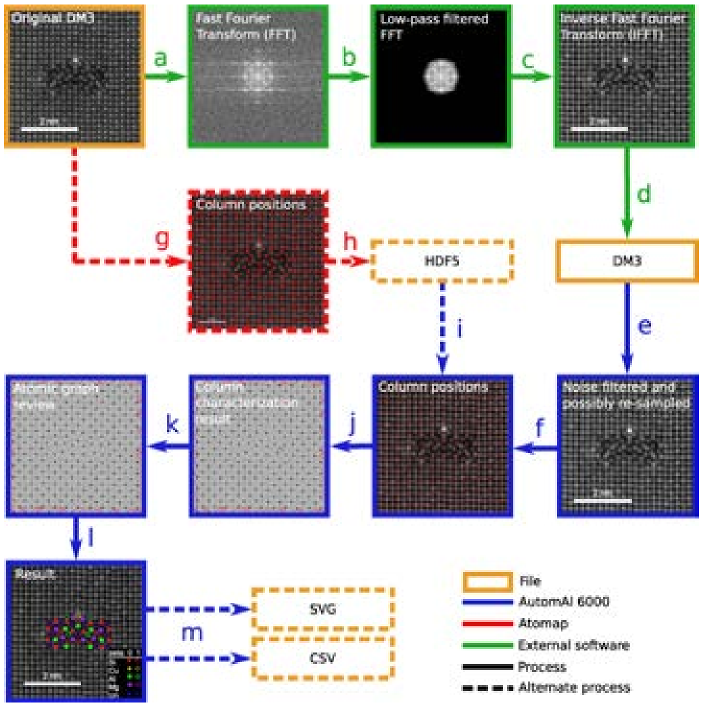


Fig. 3. Schematic of the AutomAl 6000 workflow. Column detection is performed with atomap [14], or by appropriate pre-processing, followed by AutomAl 6000's centre of mass column detection. The steps are; (a) import the image with software like for instance gatan microscopy suite (GMS) [15], and perform fast Fourier transform (FFT), (b) apply low pass mask on the FFT with a radius of approximately 6,7 nm^{-1}, (c) apply inverse FFT, (d) save results in the DM3 file format, (e) import DM3 file with AutomAl 6000, and (f) guess a threshold value and run column detection. Alternatively, (g) open image with atomap and perform column detection, (h) save result in the HDF5 file format and (i) import the HDF5 file into AutomAl 6000. Once column positions have been determined, precipitate characterization may be performed with the steps; (j) AutomAl 6000's column characterization algorithm, (k) manual corrections based on review of the atomic graph, (l) refresh statistics and finally, (m) export of the data, either as CSV for parsing the 3D positions and species into other software, or SVG for making high quality atomic overlays with any SVG software, for instance Inkscape [16].

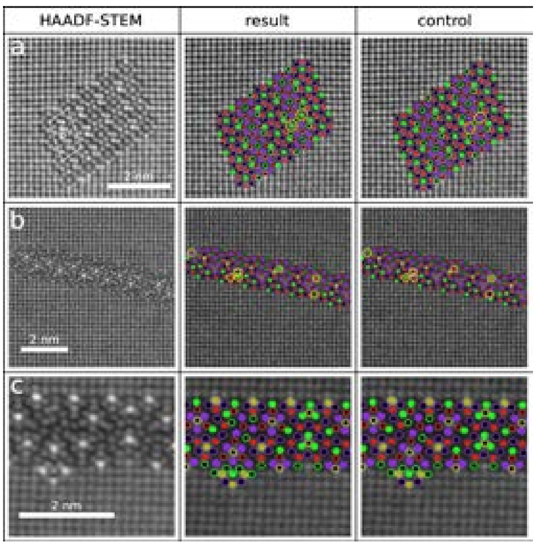


Fig. 9. Example HAADF images of 3 different precipitates in Al zone. The left column shows sections of the HAADF-STEM images chosen for this study: (a) precipitate, (b) Q' precipitate, and (c) L precipitate which also features a unit. Image details can be found in Table 3. Middle column shows the result of the column characterization algorithm, without any manual graph review, and the right column shows control overlays. The discrepancies between the result and control overlays are highlighted by yellow circles. For overlay legend, see Fig. 1. For relative discrepancies, confer Table 4.

Tvedt, H., et al., AutomAl 6000: Semi-automatic structural labelling of HAADF-STEM images of precipitates in Al-Mg-Si(-Cu) alloys. Ultramicroscopy, 2022. 236. https://doi.org/10.1016/j.ultramic.2022.113493

Snapshots from the year


Yearly summer BBQ


G. Nordahl, J. Zelenina & J. Sørhaug


S. Dagenborg

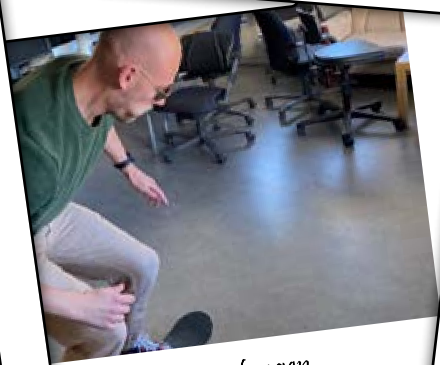

E. Thronsen & M. Nord


C. Hell

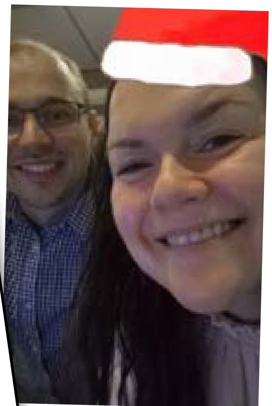

People from the TEM Gemini Center


S. Dagenborg, D. Chatterjee, J. Zelenina & J. Frafjord


J. Frafjord ...


O. Ryggetangen


J. Frafjord


C. Hell & E Thronsen


Social lunch

Images: *people from the TGC, 2022.*

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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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