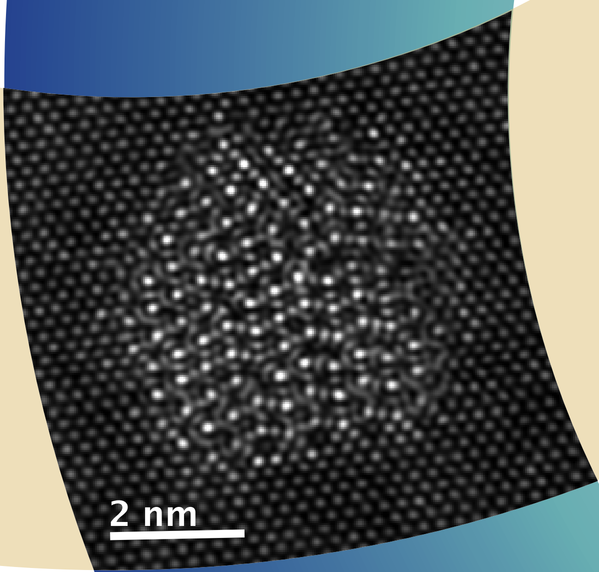
2021 Annual report

TEM GEMINI CENTRE

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

Images on front and back cover: *image collage showing magnetic materials, diffraction patterns, biological specimens, Al precipitates, and joint interfaces. By G. Nordahl, J. Bekkevold, I.-E. Nylund, J. Sørhaug, S. Wenner, E. Thronsen and T. Bergh.*

Image below and on next page*: high-resolution STEM images of Al precipitates and clusters, by E. Thronsen and C. Hell.*



Graphic design:
Jørgen A. Sørhaug & Gregory Nordahl.

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

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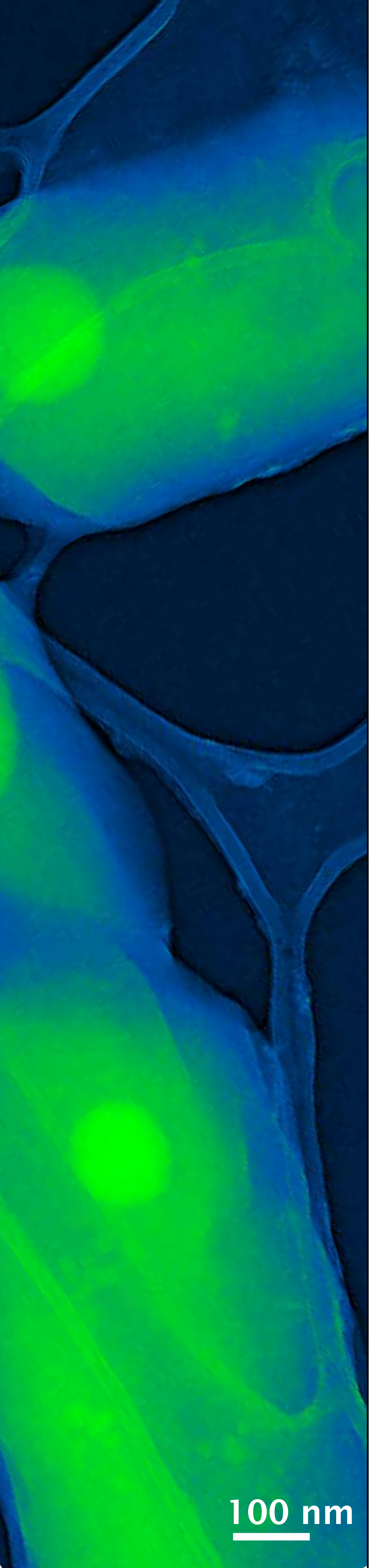


***Image to left:** E.coli, by S. Wenner.*

INTRODUCTION

2021 has been a very productive and successful year for the TEM Gemini Centre despite the ongoing pandemic. We show an 'all-time-high' in both income and output! :) Again, we see that the important 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. An all-time high of new users have been trained on the microscopes, the number of research papers are the highest ever, and we have secured funding for new infrastructure. The most important point being that 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, paving the way for continued excellence in years to come. A re-investment on this scale 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. In addition, through collaboration within the TEM Gemini Centre, a proposal for new detectors on the ARM was granted by NTNU. Both of these grants are the result of a tremendous effort from key members in the TEM Gemini Centre at NTNU and the NORTEM consortium of UiO, NTNU, and SINTEF. Now, the NORTEM consortium is facing a daunting, but exciting, time ahead where TEM possibilities in Norway for the next decade will be decided. The TEM Gemini Centre collaboration will no doubt be crucial in this important task.

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 which is important for a systematic management of the infrastructure. Managing this important infrastructure is always a team effort and 2021 was no exception. With the use of these instruments increasing every year, and the resulting high scientific throughput, it is important to recognize the importance of strategic collaboration. While NTNU owns and runs the TEM infrastructure in Trondheim, SINTEF is an important scientific user and collaborator that provides a crucial stability. Through this relationship, advanced TEM research is made available to both national and international industry and partners. Although the pandemic has made travel difficult, the Centre still facilitates transnational access to TEM through its participation in the ESTEEM3 EU Horizon 2020 network with several other world-leading TEM laboratories. This increases the visibility in Europe in a time where conferences and workshops have been cancelled or changed to less interactive formats. Several new projects were granted to staff within the TEM Gemini Centre last year, and the TEM group has grown as a result. We now have several new projects running, and the activity is high.

The Gemini Centre still 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 for a competitive and sustainable process industry (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. Due to the pandemic, the INT-PART project with Japanese aluminium industry and academia has been at a standstill, but we hope to allow some exchange of students between Norway and Japan in 2022. In spite (or because) of corona we organized a very successful online workshop in June 2021 with more than 100 participants from all over the world. This is described elsewhere in this report. We also managed to carry out a strategy seminar in between corona lockdowns. As documented in this report, the Centre had 41 active hands-on users/operators, 23 users through operators and served 104 different projects, whose results have contributed to 50 journal publications (plus 7 in press) in 2021. 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, 1 PhD student and 5 MSc candidates were educated with TEM as a substantial part of their theses in 2021. Due to the pandemic, the facility has been used less hours in physical teaching the last two year, however more courses (4 per year) have used TEM in lab exercises. The annual TEM introduction course has continued in its digital form and is followed by several (20-30) people. Through the weekly group meetings with presentations and about 20 participants, we have managed to stay in touch through yet another year of pandemic, even if conditions sometimes forced the meetings to be digital.

The new direct electron detector on the 2100F is being used extensively and is producing excellent results and offers new and interesting avenues within electron diffraction. In relation to the NORTEM II grant, we are continuing discussions regarding rebuilding and the Campus project at Gløshaugen. The complexity of the situation requires constant and diligent work from the centre and is of great concern to many. Currently, expanding the infrastructure around Chemistry Block 1 close to the original infrastructure is one of the clearest routes, but will require attention concerning both the Campus Development and the SINTEF Horizon projects.

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 2021. For more details, see our home page: ntnu.edu/geminicentre/tem

TEM Gemini Centre management, February 2022.

Prof. Randi Holmestad

Prof. Antonius T. J. van Helvoort

Prof. Yanjun Li

Assoc. prof. Per Erik Vullum

Ragnar Fagerberg

Ruben Bjørge

Inga Ringdalen

Bjørn Gunnar Soleim

Emil Frang Christiansen

BOARD AND MANAGEMENT OF TEM GEMINI CENTRE

TEM Gemini Centre board:

- Erik Wahlström, Department head, Department of Physics, NTNU
- Ragnar Fagerberg (until July), Inga G. Ringdalen (from August), Research manager, Materials Physics Trondheim, SINTEF Industry
- Einar Magne Hjorthol, Department head, Department of Materials Science and Engineering

Centre management:

- Randi Holmestad, Physics, NTNU, Leader
- Ragnar Fagerberg, Materials Physics Trondheim, SINTEF Industry
- Inga Ringdalen, Materials Physics Trondheim, SINTEF Industry
- Ton van Helvoort, Physics, NTNU
- Bjørn Soleim, Physics, NTNU
- Emil Christiansen, Physics, NTNU
- Yanjun Li, Materials Science and Engineering, NTNU
- Per Erik Vullum, Materials Physics Trondheim, SINTEF Industry
- Ruben Bjørge, Materials Physics Trondheim, SINTEF Industry

***Image above**: group picture of some of the people in the TEM Gemini Centre at Teveltunet, November 2021.*

PEOPLE IN THE TEM GEMINI CENTRE IN 2021

- Sigmund J. Andersen (Senior research scientist, SINTEF)
- Rajit Aravinth (Master student, DP, NTNU)
- Julie Marie Bekkevold (Master student, DP, NTNU)
- Tina Bergh (Postdoc, department of chemistry, NTNU / PhD student, DP, NTNU)
- Ruben Bjørge (Research scientist, SINTEF)
- Torbjørn Bogen-Storø (Master student, DP, NTNU)
- Joseph Vincent Broussard (Master student, DP, NTNU)
- Idun Bækken (Master student, DP, NTNU)
- Dipanwita Chatterjee (Postdoc, DP, NTNU)
- Emil Christiansen (Senior Engineer, DP, NTNU)
- Sivert Johan Vartdal Dagenborg (Master/ PhD student, DP, NTNU)
- Jonas Frafjord (Postdoc, DP, NTNU)
- Jesper Friis (Senior research scientist, SINTEF and Assoc. Prof. II, 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)
- Petter Lervik (Master student, DP, NTNU)
- Martin Bakken Lesjø (Master student, DMSE, NTNU)
- Yanjun Li (Prof., DMSE, NTNU)
- Marthe Linnerud (Master student, DP, NTNU)
- Ursula Ludacka (Postdoc, DMSE, NTNU)
- Hogne Lysne (PhD student, DP, NTNU)
- Calin Marioara (Senior research scientist, SINTEF)
- Knut Marthinsen (Prof., DMSE, NTNU)
- Magnus Nord (Assoc. Prof. DP, NTNU)
- Gregory Nordahl (PhD student, DP, NTNU)
- Inger-Emma Nylund (PhD student, DMSE, NTNU)
- Ding Peng (Postdoc, DP, NTNU)
- Ingeborg Nævra Prestholdt (Master student, DP, NTNU
- Knut Håvard Raen (Master student, DP, NTNU)
- Andreas Rosnes (Master student, DP, NTNU)
- Oskar Ryggetangen (Master/PhD student, DP, NTNU)
- Armand Sepehri (Master student, DP, NTNU)
- Mari Sofie Skomedal (Master student, DP, NTNU)
- Bjørn Gunnar Soleim (Senior engineer, 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, DP, NTNU)
- Haakon Tvedt (Summer student, DP, NTNU)
- Per Erik Vullum (Senior research scientist, SINTEF and Assoc. Prof. II, DP, NTNU)
- Sigurd Wenner (Research scientist, SINTEF and Assoc. Prof. II, DP, NTNU)
- Yingda Yu (Senior engineer, DMSE, NTNU)
- Hedda Øye (Master student, DP, NTNU)
- Håkon Wiik Ånes (PhD student, DMSE, NTNU)

THE NORTEM PROJECT

NORTEM (Norwegian Centre for Transmission Electron Microscopy) is a nationally coordinated largescale infrastructure project (2011-2020) with three partners - SINTEF, NTNU and UiO, funded by the Research Council of Norway and the three partners. The budget for new equipment and the rebuilding in the project was about 75 MNOK in total the two geographical nodes, Trondheim and Oslo. We have now been running the facility for eight 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 fell through.

In December 2021 we received word from the Research council of Norway 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 DEDs, advanced probe-forming systems with more flexibility in illumination, higher voltage (300 kV), improved mechanical and thermal stability and increased automation, focused on structure determination, diffraction and electric/magnetic field imaging.

The vision of NORTEM is to be "A world-class TEM facility providing access to expertise and state-of-the-art infrastructure for fundamental and applied research within the physical sciences in Norway". Besides being a top research TEM lab, the infrastructure provides access to TEM for a broader user environment, addressing fundamental and applied research topics in physics, chemistry, materials science and geology. The combination of a research lab and a user facility requires a clear and sustainable running model, and the TEM Gemini Centre has spent considerable effort during the last years to establish a sound running model for the infrastructure. This has been established and have been running well for some years now. 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 attention has to be given to getting the best out of the huge and complex investment ahead.

The Trondheim node NORTEM facility has two senior engineers, Bjørn Gunnar Soleim and Emil Christiansen supporting maintenance, training, competence and techniques. We have a high uptime and ca. 16 % of the users are based outside the host institutions. Per Erik Vullum and Sigurd Wenner have been working as adjunct (affiliated) professors (20 %) at NTNU, which particularly contributes to developing interaction between NTNU and SINTEF. For more information on NORTEM see the webpages: nortem.no.

Key numbers for NORTEM:

- 3 partners - SINTEF, NTNU and UiO;
- 2 nodes - Trondheim and Oslo;
- 58 MNOK from INFRA in 2011;
- 5 microscopes - 2 top level from 2012/2013;
- 151 projects served in 2020;
- 46 000 h used since 2012;
- ~ 60 users annualy;
- ~ 60 papers annualy (2/3 international co-authors);
- ~ 15 permanent staff in core research groups;

***Image above**: St. Hans evening trip to Theisendammen for some grilling and chatting before hiking to Våttakammen.*

Image above: *Norway/Sweden border trip during the stay at Teveltunet 2021.*

THE TEM GEMINI CENTRE

The TEM (Transmission Electron Microscopy) Gemini Centre was established in 2006, and consisted of professors, postdocs, students, and engineers from the Department of Physics (DP), NTNU and researchers from the Material Physics, Trondheim research group in SINTEF Industry. In 2009 the Department of Materials Science and Engineering (DMSE) at NTNU was included in the Centre. The same constellation was last renominated in November 2018 for a new period of 4 years. The Centre's research groups work within materials physics and materials science, studying a broad range of materials down to the nanometer and atomic level, where the main tool is the transmission electron microscope (TEM).

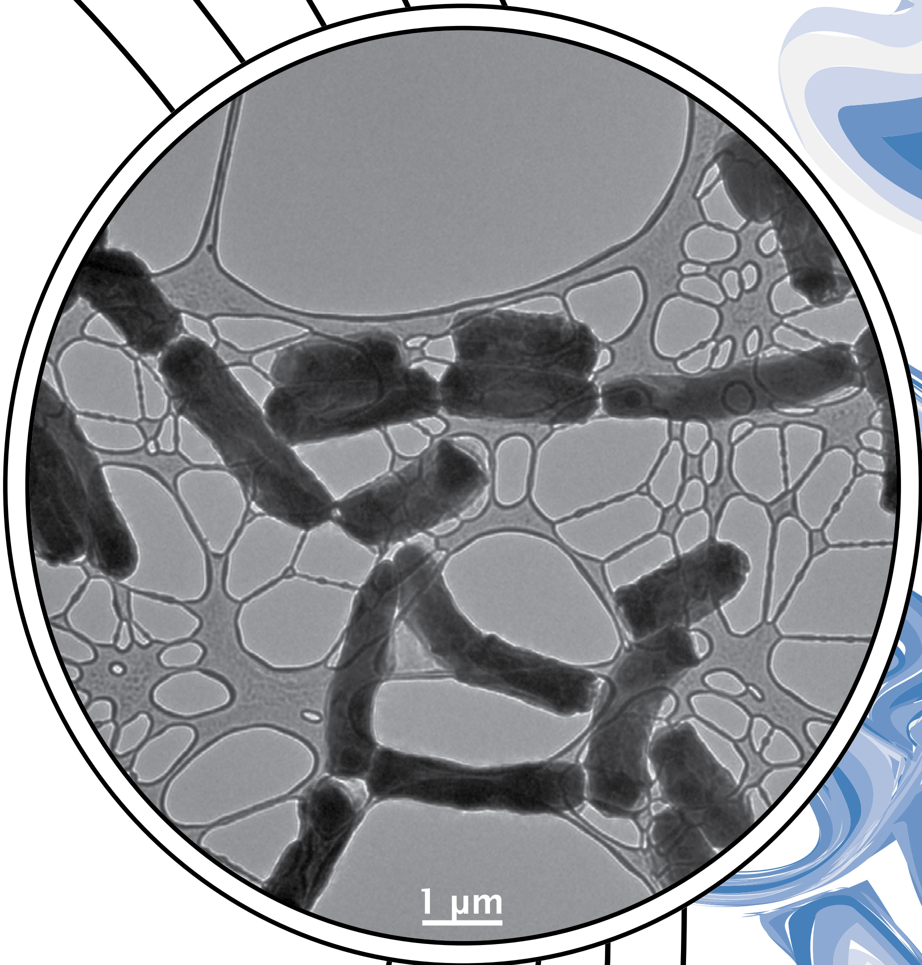
The overall objective of the TEM Gemini Centre is to build and secure a robust scientific environment within TEM with high international profile as a sound basis for growth, not only for the Centre itself, but also for other parts of NTNU and SINTEF and academic and industrial partners. Parallel to and together with this, the large nationally coordinated infrastructure project, NORTEM, has given a broader identity to the Centre's TEM infrastructure.

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 will enable collaborating groups to grasp new opportunities and bring them to fruition in the form of better value generation and profitability. High-quality technical centers are in great demand internationally from both commercial clients and students. The shared vision of Gemini Centers is:

"Global excellence together".

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

***Image at top**: grabbing a bite at Edoramen, by T. Bergh.*
***Image to the bottom left**: dinner at Randi's place after group trip in Bymarka, sept. 2021.*
***Image to the bottom right**: Group hike to Våttakammen, june 2021.*
***Image on next page**: E. coli, by S. Wenner.*



Instrumentation

JEOL double corrected JEM-ARM200F (cold FEG)

This is currently the top instrument in the TEM Gemini centre. The stable cold FEG with both probe and image spherical aberration correction and the most advanced EDX and EELS systems allow unique studies at the atomic scale. The microscope is placed in a custom designed room with water cooled walls and field cancellation.

JEOL JEM-2100F

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

JEOL JEM-2100

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

THE TEM INSTRUMENTS IN TRONDHEIM

The TEM Gemini Centre has three TEMs installed as part of the NORTEM project in 2012/2013 - a JEM-2100 LaB_6, a JEM-2100F and a double corrected JEM-ARM200F.

The ARM features:

- Cold field emission gun with energy spread of 0.3 eV
- Cs-probe corrector
- Cs-image corrector
- Centurio SDD EDX (solid angle 0.98 sr)
- Quantum GIF with DualEELS
- 2k Orius CCD (side-mounted) and 2k UltraScan CCD (bottom mounted)
- Stable 5-axis gonio with piezo control in x, y and z-directions
- Detectors for BF, ABF, ADF and HAADF STEM
- Aligned at 80 kV and 200 kV

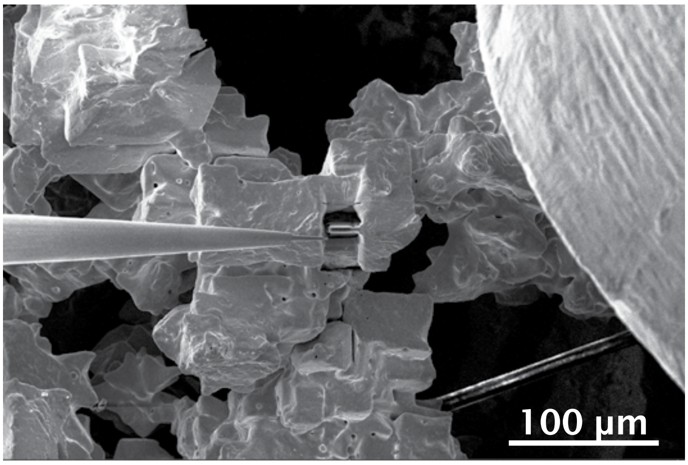
The 2100F features:

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

The 2100 features:

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

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



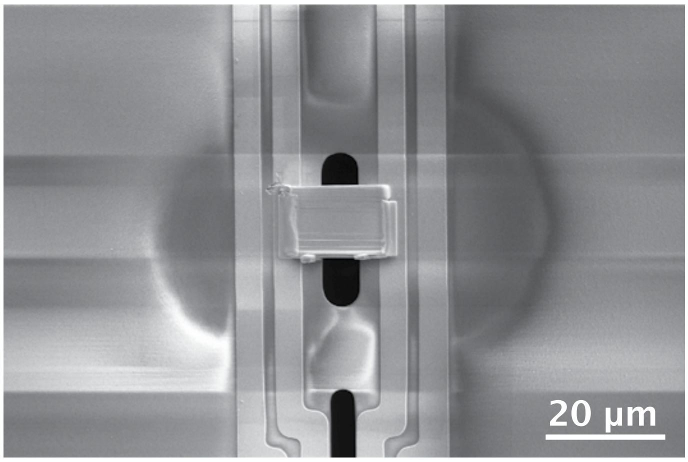


Image above to left*: SE SEM image taken during FIB preparation of a TEM lamella from a silver particle produced by K.A. Rasmussen, taken by T. Bergh.* ***Image above to right****: DENS biasing chip with $Gd_2(MoO_4)_3$ fastened to electrodes, by I.-E. Nylund.*

SPECIMEN PREPARATION

Given the high resolution of the TEM instruments, specimen quality is often the limiting factor. Also, special holders require a dedicated specimen shape for optimal performance. The Gemini Centre has well equipped specimen preparation facilities at both DP and DMSE, reflecting the broad range of materials studied. The Centre has different types of 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 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 interest application area is advanced characterization of battery materials. 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.

Image to left from left*: T. I. Thorsen, L. Sandnes, Ø. Grong, T. Austigard, J. Sørhaug and T. Bergh visiting Ø. Grong and the engineering workshop of HyBond at the Dep. of mechanical and industrial engineering to see the well known HYB machine performing Al/Al metal plates consolidation.*

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 noteworthy 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

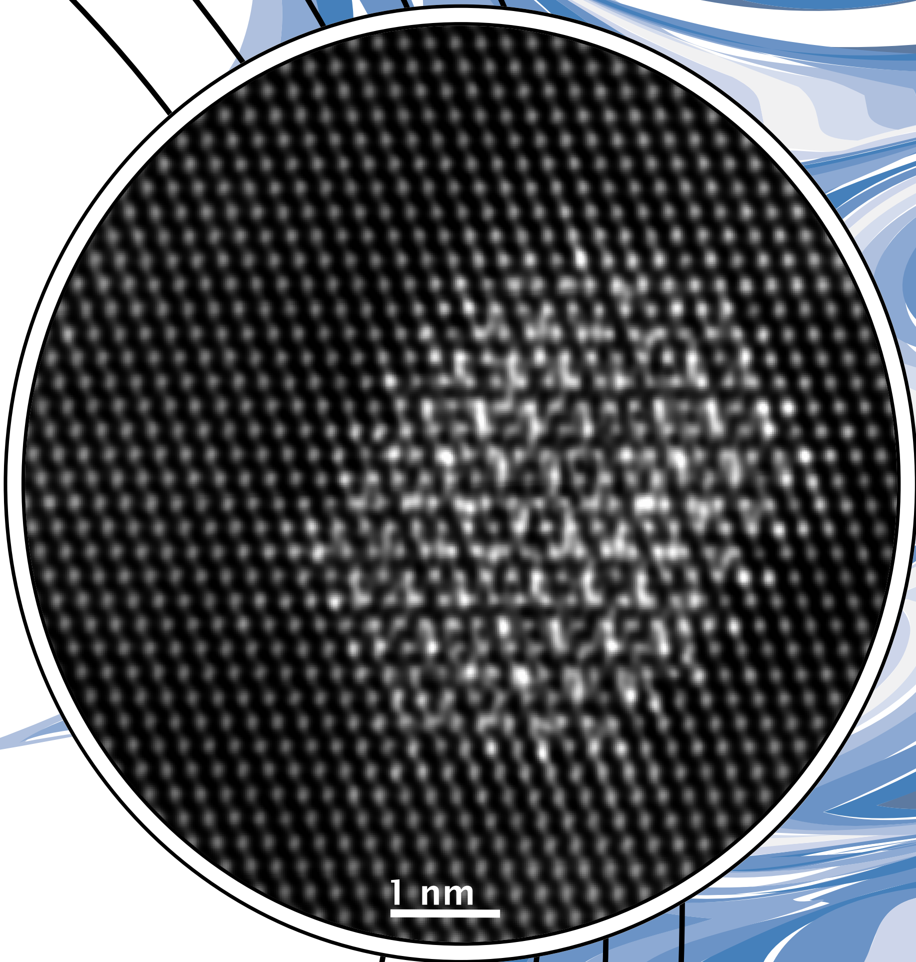
With the double aberration corrected microscope, the cleanliness requirements of the specimen and the holders are high. We have a dedicated room close to the microscopes with general equipment, such as a plasma cleaner, ozone cleaner, a stereomicroscope, user specimen storage and special holders that are used on all three TEMs. In addition, there is a data transfer room with additional facilities as a printer and a support PC with the most crucial software packages. The room has a sofa and tea/coffee machine for socializing and efficient breaks during long running sessions.
The dedicated computer room for TEM data analysis has five machines, for postprocessing and simulating TEM results, some of which can be remotely accessed. It also includes more powerful workstations, dedicated to more demanding data processing. 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 2021

The total registered used time for the three instruments in 2021 was 4345 hours, the highest number in the lifetime of the facility including 130 nonpaid hours used for testing, competence development, demonstrations, and guided tours. Of the 4215 paid hours, the use by NTNU corresponds to 77 %, externals (with NTNU operator) 5 %, and SINTEF 18 %. NTNUs use is divided over five departments, where the main use is from Department of Physics (78 % of NTNUs paid hours). 104 different projects used TEM in 2021. The infrastructure had in total 64 users, of which 41 were hands-on operators. 4 of the users were based at SINTEF, 19 were PhD candidates and 18 were master students.

Microscope use (hours)	ARM-200F	2100F	2100	Sum
SINTEF	466	99	212	777
NTNU - Physics	590	964	752	2306
NTNU - Other departments	234	290	158	682
NTNU - Visitors from abroad	0	68	49	117
NTNU - Teaching lab	7	92	16	115
External	99	112	7	218
NTNU - Setup/testing/training/ demonstrations	17	110	3	130
Total use	**1413**	**1735**	**1197**	**4345**

Image on next page: *HAADF STEM image of a precipitate in an Al-Zn-Mg alloy, by E. Thronsen.*



Activities

Research and events

FOCUS AREAS

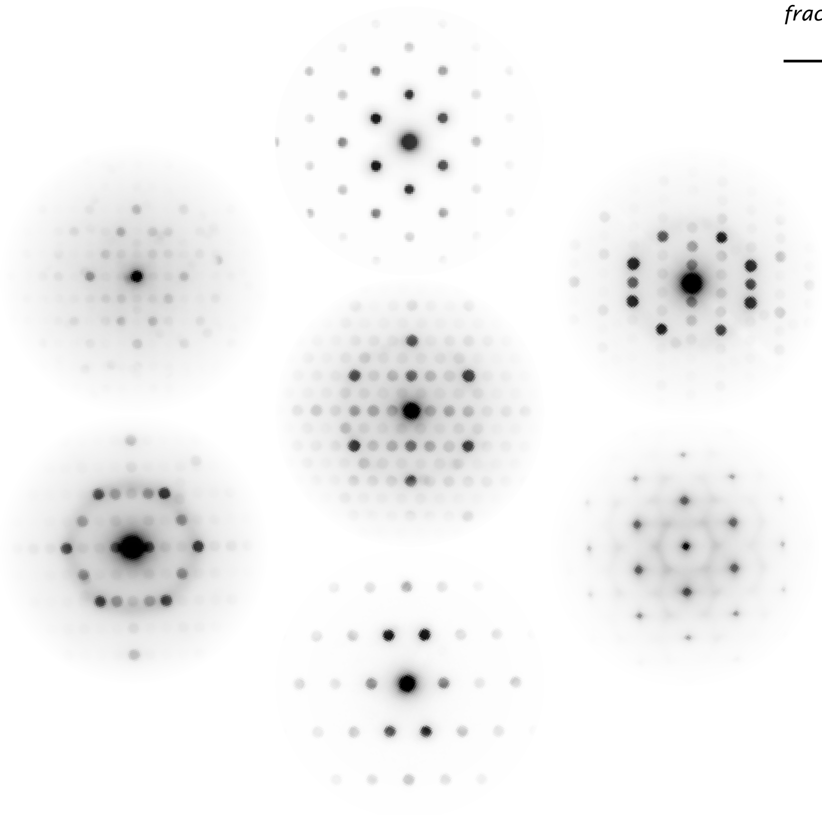
TEM is a powerful technique for fundamental and applied research in the physical sciences, in different fields from geology, metallurgy and semiconductor industry to fundamental chemistry and physics. NORTEM has identified four focus areas, which have been important for the TEM Gemini Centre activities since the Centre was formed. Within these areas we see potential for further growth and tackling unsolved issues. The focus areas are light metals, catalysis, energy materials and nanotechnology. TEM plays an important role in these research areas, which will be strategically important for Norway also in the future. The TEM Gemini Centre had activities in all these four areas in 2021. The next sections describe these activities. Activities in aluminium alloy research are the largest. In all areas the use of advanced data processing has gained significance.

ALUMINIUM - LIGHT METALS

The study of aluminium alloys using TEM has been a pillar in the Trondheim TEM environment for many years, and there have been many successful projects. All these projects have been jointly between NTNU and SINTEF and supported by the Research Council of Norway. In addition, many of them were supported by Norwegian light metal industry, in particular Hydro Aluminium. In 2021 we have been involved in 3 SFI Centers, one competence project (KPN) and one Digitalization project in aluminium research, in addition to the INT-PART project with Japanese universities and aluminium industry. In SFI CASA, headed by Prof. Magnus Langseth at the Structural Engineering department in the NTNU Engineering Faculty, we are involved in the “lowest scale” of the multiscale activities, including TEM and atomistic calculations of precipitates, grain boundaries, precipitation free zones and interactions between them and dislocations in deformed, mostly industrial, Al alloys. There is no PhD or Postdoc hired on this scale at the moment, but there is some SINTEF activity. The project leader of the SINTEF part of CASA Lower scale is Inga Ringdalen. SFI CASA has made a promotion video. where TEM on aluminium has a central part. and In SFI Manufacturing, headed by Sverre Guldbrandsen-Dahl from SINTEF Manufacturing, joining of aluminium with other materials in multi-material products is a central topic. Tina Bergh characterized the microstructure of the interfacial region in aluminium-steel joints made by various joining techniques, including joints made by the start-up company HyBond. Tina used conventional and advanced TEM techniques and also worked on electron diffraction data analysis (pyXem). She defended her thesis in May 2021. Ding Peng works as a postdoc on this SFI in the TEM group. Ding studies 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 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 was hired as a PhD student here in August 2021 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

Images below: *image collage showing precession electron diffraction patterns, by T. Bergh and J. Sørhaug.*



of alloying elements, nanoparticles and heat treatments will be studied.

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 Ruben Bjørge as a project leader and with one PhD at NTNU. Oskar Ryggetangen started his PhD in August 2021 and will focus on acquiring and analysing electron diffraction data from quasi-crystalline phases, aided by advanced high resolution analysing techniques, in particular transmission electron microscopy.

The project 'In-Sane' - In-situ studies of highly conductive bonded interfaces between aluminium and copper at the nanoscale – started in 2020 and is a Nano2021 project in collaboration with Department of Mechanical and Industrial Engineering where Randi Holmestad is the project leader. The idea is to perform nanoscale joining in the FIB at the nanoscale, in order to understand and develop the HYB (Hybrid Metal Extrusion & Bonding) method. The motivation for In-Sane is to produce dissimilar and highly conductive Cu/Al 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 the Department of Mechanical and Industrial Engineering and focuses mostly on making the joints in the FIB. Per Erik Vullum is central in this work.

We have a competence project on aluminium - SumAl (Solute cluster manipulation for optimized properties in Al-Mg-Si based Al alloys) with industry partners from Norway (Speira, Benteler and Neuman), Austria (Neuman), Sweden (Hydro) and Germany (Hydro). The primary objective of SumAl is to establish an in-depth understanding of early-stage solute ordering and atomic clustering by advanced experiments and modelling, and how these structures relate to the development of hardening precipitates and materials properties. Randi Holmestad is project leader, and 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 with Calin Marioara and Sigmund Andersen as central participants. 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.

During the last years, we have had several aluminium alloy related collaborations abroad. The largest is the Japanese collaboration with academia and industry, where the INTPART project was renewed for 4 new years in 2018 and extended with new partners. This is further presented elsewhere in this report. Another aluminium project is the NTNU financed Digitalization project AllDesign with Jaakko Akola as project leader. AllDesign provides fundamental insight on solid-state precipitation in aluminium alloys based on synergistic multiscale modelling, and its impact on macroscopic properties and manufacturing processes. The concept is data-driven and utilizes new trends in materials research. Elisabeth Thronsen is a PhD student on this project and works on TEM of early-stage clustering and precipitation in close collaboration with the SumAl project. 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!) – MRM in Japan, TherMec in Austria, M&M in USA, PICO in Netherlands, which shows that our work on aluminium is internationally recognized.

ENERGY MATERIALS – SOLAR CELLS

TEM has proven to be a crucial characterization tool to understand and improve the efficiency of both conventional and novel types of solar cells. The TEM Gemini Centre activities within solar cells include both types and a large range of materials. The Gemini Centre is participating in the FME SUSOLTECH (The Norwegian Research Centre for Sustainable Solar Cell Technology) on solar cells and project students, PhD students and SINTEF researchers within TEM are actively taking part in subprojects related to both conventional as well as third generation solar cells. Hogne Lysne is a PhD student in the FME on solar cells working on the growth and characterisation of Cr and N codoped TiO_2 with Turid Reenaas as main supervisor. In addition, MSc student Andreas Rosnes works on characterization of thin films in this project. SINTEF has worked together with ELKEM and IFE in three consecutive IPN projects within production of tailored Si powders for use in Li-ion batteries. The present project, “Silicon-based anodes towards market penetration (SiBanode)” started in 2019. The aim is to develop Si/graphite-based composites as anodes in commercial Li-ion batteries. TEM has been one of the primary tools to characterize and understand the behavior of the anode composites as a

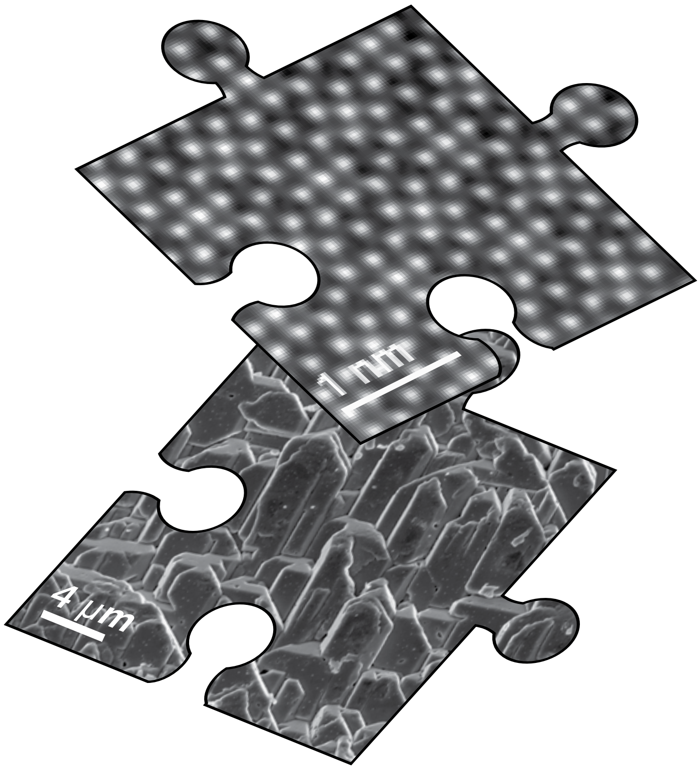
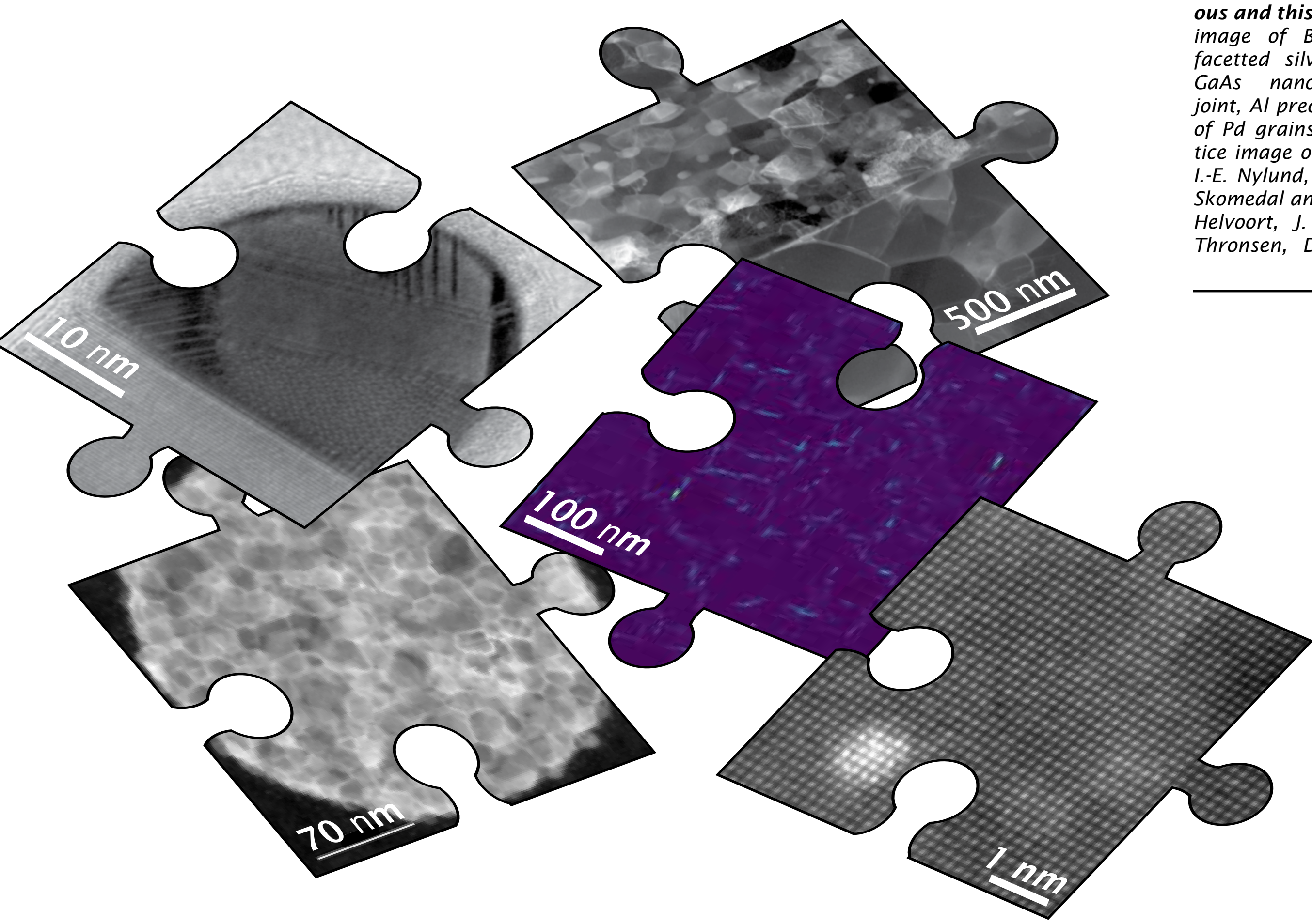


***Image collage on previous and this page**: lattice image of $Ba_4Rb_2Nb_{10}O_{30}$, facetted silver particles, GaAs nanowire, Al/Al joint, Al precipitates, disc of Pd grains, and Al lattice image of clusters, by I.-E. Nylund, T. Bergh, M. Skomedal and A. T. J. van Helvoort, J. Sørhaug, E. Thronsen, D. Chatterjee and C. Hell.*



function of structure, morphology and cycling conditions. Another IPN project, "HAST", is led by the company CENATE, a spin-off company to Dynatec. This project also aims to develop Si-based materials optimized for anodes in commercial Li-ion batteries. TEM is here a central characterization tool to study and understand the behavior of the initial and cycled Si-based electrodes. At the end of 2019 the IPN project, "Surface treatment of Artificial Graphite for Anodes in Lithium-ion Batteries (SAGA)", was funded by RCN. The project owner, 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. TEM is also used in several other projects related to development of Li-ion battery technologies. In the KMB project "Silicon on the Road", with SINTEF, IFE and UiO as academic partners and Cenate, Equinor, Borregaard and Beyonder as industry partners, TEM is used to characterize battery electrodes as a function of syntheses and cycling conditions. In the KPN project "SiBEC" TEM is one of the important characterization tools to understand the behavior of the cathode material as a function of synthesis and cycling conditions. In the FME MoZEES TEM is also used to characterize and understand the fundamental behavior of the battery electrodes as a function of electrode and electrolyte compositions, synthesis and cycling conditions.

NANOTECHNOLOGY

As TEM can analyze size, crystal structure and composition it is an important tool at the nm-scale in nanotechnology and nano-sciences. TEM is important because it can do all these characterizations on the same small volume and thereby relate structure to properties. The TEM Gemini Centre activities cover nanoparticles, 1D-nanostructures (i.e. nanowires, carbon nanotubes), thin films and 2D-materials such as graphene. These activities are part of NANO@NTNU. NTNU NanoLab is our direct neighbour and many of the TEM operators also use equipment in the cleanroom. Especially, the FIBs are important for the TEM Gemini Centre. In 2021 a plasma FIB is installed, in a room which had once the centre's first FEGTEM. This new Xe-based FIB could become important for TEM preparation of materials like Al which is difficult to prepare with a Ga FIB. We developed together with collaborators in Trondheim methods for correlated studies where TEM is directly combined with SEM, EBSD, Cathodeluminescence, Photoluminescence and Scanning Probe Microscopy. Hereby, more all-round characterization of nanomaterials is realized. NorFab is an important partner for the TEM Gemini Centre. Many of our master students follow the Nanotechnology study program, a further demonstration that TEM is an essential tool for practical nanotechnology. These student projects include both practical as well as theoretical/computational focus. Several core TEM Gemini people are actively involved in nanotechnology projects, for example as co-supervisors. In the TEM Gemini Centre, both NTNU and SINTEF have worked with the start-up company CrayoNano. As can be seen from the publication list, many TEM studies on nanomaterials resulted in journal publications in 2021. With the atom probe tomography (APT) instrument recently installed at NTNU, the TEM group is participating in building up competence on APT and work on correlative techniques between TEM and APT.

MAGNETIC MATERIALS: IMAGING ELECTROMAGNETIC FIELDS

While there has been much work done studying magnetic materials at the TEM Gemini Centre, there has been very little work on directly imaging the magnetic fields themselves. Thanks to recent advances in fast pixelated direct electron detectors this has become easier, making techniques such as scanning TEM-differential phase contrast (STEM-DPC) much more accessible. One of the focuses of newly hired associate professor Magnus Nord is to improve the STEM-DPC technique, utilizing the recently installed MerlinEM fast pixelated direct electron detector, together with PhD student Gregory Nordahl. This activity increased in 2021, since Magnus was granted a Young Research Talents "In-situ correlated nanoscale imaging of magnetic fields in functional materials" (InCoMa) project from the Research Council of Norway. PhD student Sivert Dagenborg was hired on this project from August 2021, and a postdoc is starting in 2022. Another upgrade that will take place in 2022 will be the new 4D-STEM detector for the ARM, which will give us 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, Chemistry Oil and Gas Process Technology Departments. The SFI Innovation for a Competitive and Sustainable process Industry (iCSI), headed by Profess -or Hilde Johnsen Venvik, hired in 2021 Tina Bergh as a postdoc to provide a platform for further applications of TEM in both academic and industrial catalysis research. Membrane research has contributed to a study in the BIGCCS carbon capture FME project. In the H2020 project eForFuel, Ir-based catalyst nanoparticles were investigated for their potential use in producing formic acid from carbon dioxide and water.

ADVANCED DATA PROCESSING

The ongoing revolution within TEM research is digital and data-driven and recent investments take this into account. "Big data" processing, machine learning, open-source development and increasingly automated data handling are some of the current "hot topics". In 2021 we worked further on developing transparent procedures to handle larger TEM data sets and automation for more robust TEM studies. There were MSc, PhD and SINTEF projects dedicated on improving data handling. The TEM Gemini Centre has bought a share in the NTNU

HyperSpy
multi-dimensional data analysis

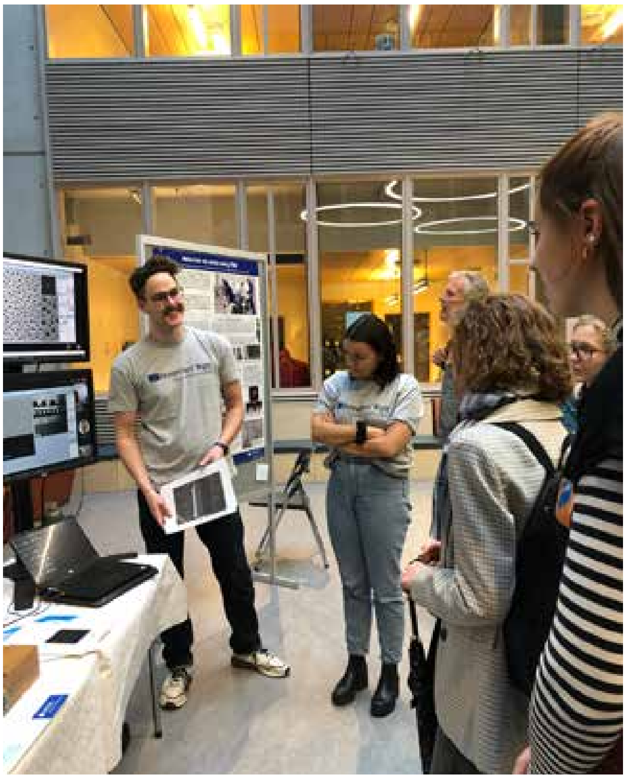


Image above *from left: E. Christiansen og E. Thronsen telling about TEM and research during Researchers night.*

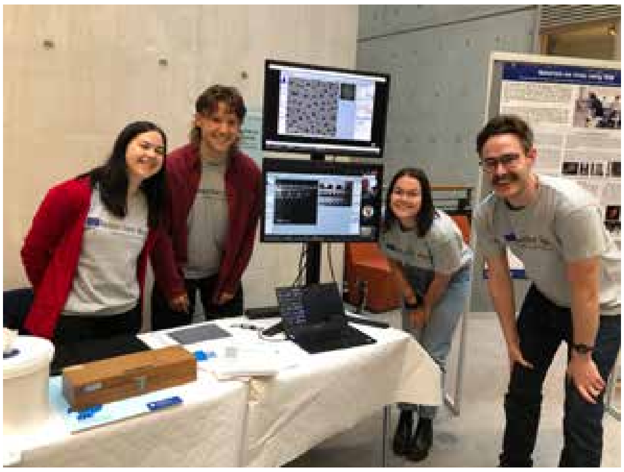


Image above*, from left: E. Thrane, A. Rosnes, E. Thronsen, E. Christiansen during Researcher's night.*

cluster IDUN, and is utilizing this, in particular for SPED data analysis. The common effort in establishing cluster-based TEM data handling will be continued and we had an internal workshop for the use of IDUN. We have recently invested in two powerful local workstations for all TEM users to be able to analyze the large data volumes produced. In September 2020 Norway's first direct detection TEM detector (DED) was mounted and in 2022 we will install the second. As this detector can easily generate giga- or terabyte size data during one TEM session, file conversion, data transfer and storage have quickly become a bottleneck. We are confident that a recent update on the labs data infrastructure will allow us to make full use of the new possibilities this detectors give our TEM facility. In the application of advanced TEM, especially multidimensional data set acquisition and handling, data processing transparency and dynamic in-situ studies are further developed. This is also in line with the open science policy pushed by EU and other authorities. Students within the group have used and contributed over many years to the open-source software, especially the Python library HyperSpy (hyperspy.org) and for electron diffraction pyXem (github.com/pyxem). Image, diffraction, EDX and EELS data from all our TEMs can be handled in the same user interface. After Magnus Nord started in the group in 2020, these topics have been further developed and used. This has created a more robust software project for working on scanning electron diffraction data and has enabled increased speed and big data handling capacities of several processing categories, making it possible to analyze the very large datasets generated by the DED detector on desktop or laptop computers. Also, within SINTEF advanced data handling and incorporation of data TEM activities were further developed. Most of our MSc and PhD students are familiar with data handling in a Python environment. We have students who are dedicated towards modelling (e.g. DFT) or for whom scattering simulations are an essential part of their work.

RESEARCHERS' NIGHT, SCHOOL VISITS AND OUTREACH

The TEM Gemini Centre has contributed to many high school visits and Researchers Night. Because of the pandemic, there has been less activity on this arena also in 2021, but we managed to participate in the Researchers Night in September 2021.

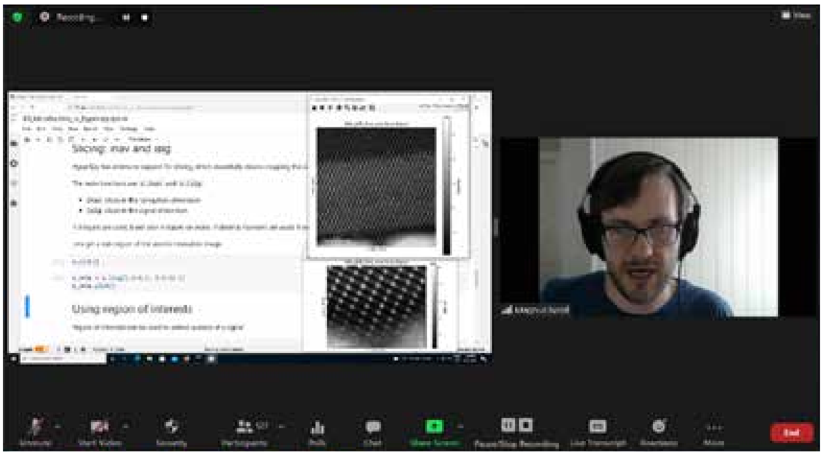


Image above: *M. Nord teaching how to use HyperSpy on TEM data during the NordTEMhub workshop on digital electron diffraction.*

STRATEGY SEMINAR AT TEVELTUNET

The TEM Gemini Centre organized a strategy seminar at Teveltunet in Meråker 11.-12. November 2021. After more than a year of corona and very limited social activities, we did some teambuilding activities and social get together, including an outdoor group competition, beer tasting, hot tub, a three-course dinner and quiz. Friday we had some standpoint analysis from people in the group about strategy and the way forward, and also an online talk by the well-known Cambridge professor Colin Humphreys 'My new life in graphene: can graphene really change the world?" Some went for a walk, and others drove to Sweden for shopping. The group work gave some new ideas and long-awaited physical discussions.

NORDTEMHUB WORKSHOP ON DIGITAL ELECTRON DIFFRACTION

21.-23. June 2021 NTNU in Trondheim organised the first NordTEMHub workshop on 'Open-source analysis of TEM data' and 200 participants attended online. We had lectures and practicals given by Katherine MacArthur (Forschungszentrum Jülich, Germany) Colin Ophus (Berkeley Lab, USA) Philip Crout (University of Cambridge, UK) and Magnus Nord (NTNU, Trondheim, Norway). Participants had to install the software (Hyperspy and py4D-STEM) and did analysis themselves on their own computers during the practicals. The workshop was very well received, and the participants said they had learned a lot. In 2022 there will be more workshops, organised by University in Oslo and DTU.

NORDTEMHUB NETWORK

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.

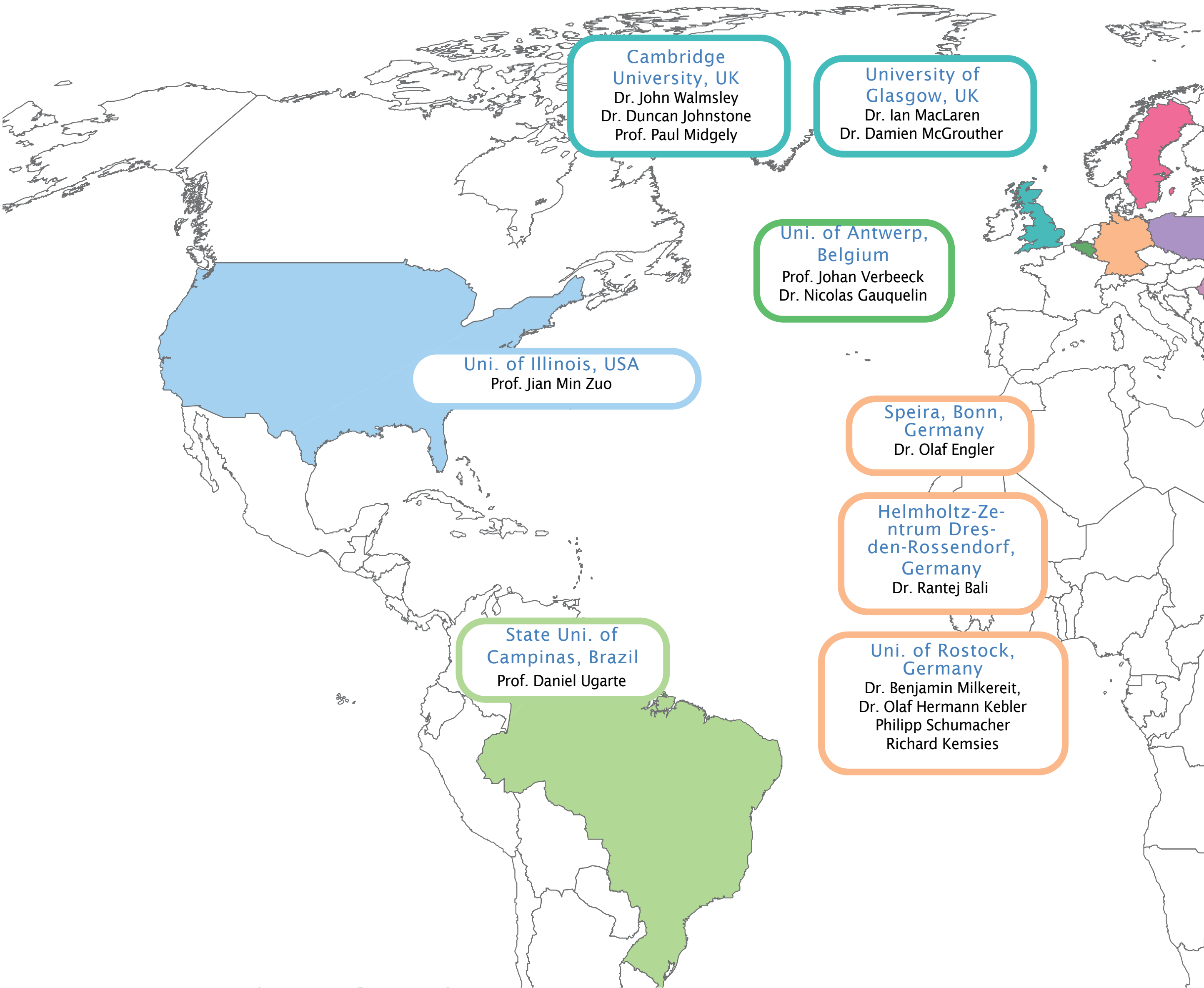
The aim is to establish collaborations, provide access, optimize instrument use and build and utilize Nordic competence on advanced microscopy. Common to all the nodes are recent and significant investments in state-of-the-art transmission electron microscopes. This initiative adds value to academia 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, but we organised an online kick-off meeting with 75 participants Friday 18th June 2021. Here we had presentations of the labs and group work across nodes to get to know each other, plan and discuss how we can collaborate.

ACTIVE PROJECTS IN 2021

The table below shows the larger projects connected to TEM within the Gemini Centre. They are listed by funding type, title, duration and research partners. Smaller projects (not listed), both academic and with direct industrial support, run in parallel. In total the Centre had about 104 different projects using the facilities in 2021.

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, Gass-co, 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	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			
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
IPN/ENERGIX	Nanowire UV LEDs on graphene	SINTEF	2019-21
Partners: CrayoNano, SINTEF, NTNU			
IPN/ENERGIX	Silicon-based anodes towards market penetration – SiBanode	SINTEF	2019-21
Partners: Elkem Technology AS, IFE, SINTEF			
IPN/ENERGIX	Surface treatment of Artificial Graphite for Anodes in Lithium-Ion Batteries.	SINTEF	2020-2022
Partners: SINTEF, Vianode, IFE			
IPN/ENERGIX	HAST.	SINTEF	2020-2021
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	2017-22
INTPART	Norwegian-Japanese Aluminium alloy Research and Education Collaboration (NJALC) – II.	NTNU, SINTEF, Travel, exchange students	2019-2023
Partners: NTNU, SINTEF, Hydro, University of Toyama, Tokyo Institute of Technology			
EU	ESTEEM3 - https://www.esteem3.eu/	1 postdoc NTNU, prof IIs	2019-24
IPN/PETROMAKS2	AMRREX	SINTEF	2018-21
IPN/BIA	HIPTEC	SINTEF	2018-20
NFR/FRIPRO	HEATER	SINTEF	2018-20
NFR/ENERGIX	ANSWER	SINTEF	2018-20
SINTEF/SEP	Molecular structure of organic nanomaterials (MOSON)	SINTEF	2021-22
IPN	Novel Failure Monitoring System for Marine Applications by including Acoustic Emission (AEMON)	SINTEF	2019-22
IPN	Catch & Kill. Partners: SINTEF, Standard Bio, USN, Uni. New South Wales	SINTEF	2020-23
EU/H2020	SAFE-N-MEDTECH	SINTEF	2019-23

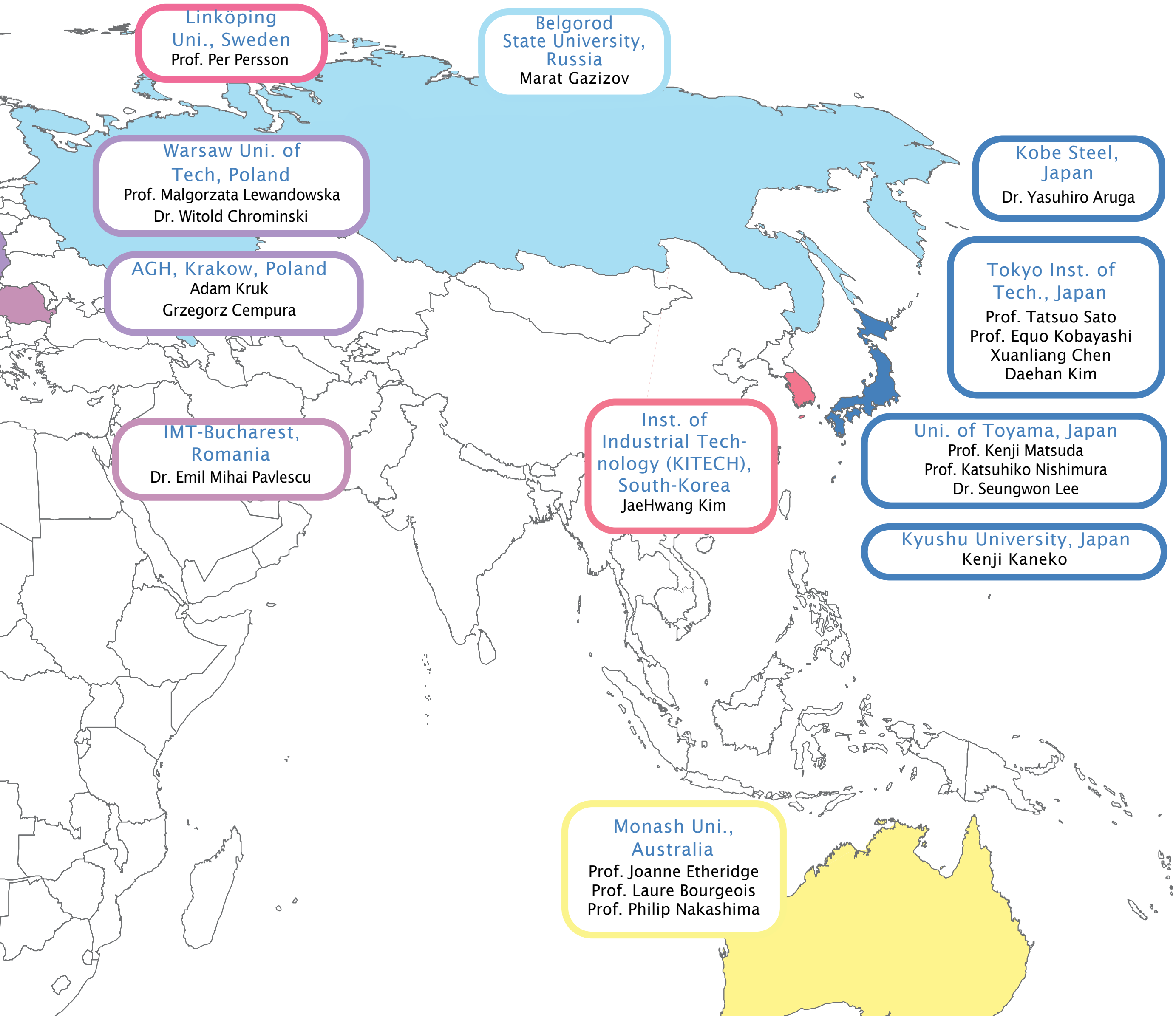


INTERNATIONAL COLLABORATION

As can be seen from the map and the publication list, the TEM Gemini Centre has productive relations to many research institutions and researchers across the world. Some are long term collaborators; others are new initiatives. However, because of the corona pandemic, there have been very few visits in 2021. Witold Chromiński from Warsaw University of Technology, Poland, visited the TEM Gemini Centre from August to November. He did TEM on plastically deformed Al-Mg-Si alloys. Through the ESTEEM3 project, the facility has been used by several renowned researchers across Europe – we also had a few 'physical' visitors to Trondheim in the fall from Cambridge, UK and Stockholm, Sweden. The map illustrates the direct scientific collaborations. In addition to the ESTEEM3 project, the INT-PART project ensures international collaborations, in this case with Japan. We thank all our international collaborators for the productive and stimulating digital interaction and hope we can be able to continue the cooperation and meet physically in the coming years!

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 package Diffraction and Data analysis. The main part of ESTEEM3 is that we can welcome researchers for transnational access, 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. The Trondheim node is going to organize a workshop through ESTEEM3 in June 2022 on '[Electron diffraction for solving engineering problems]', and we hope that this will be with physical attendance.

Image above from front: *R. Holmestad, A. T. J. van Helvoort and Witold Chromiński on topp7 trip in Bymarka in nice weather, Sept. 2021.*

INTPART PROJECT WITH JAPAN

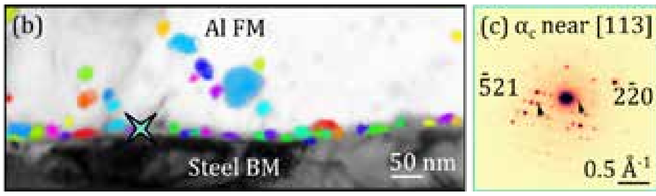
A 3-year International Partnership (INTPART) project funded by the Norwegian Research Council and the Norwegian Centre for International Cooperation in Education (SIU) called "The Norwegian-Japanese Aluminium alloy Research and Education Collaboration" ended in 2019. In addition to NTNU and SINTEF, Hydro Aluminium, University of Toyama and Tokyo Institute of Technology were partners. A phase II of this was granted in 2019, with the same partners, except one additional university, Kyushu University in Fukuoka. The objective of this project has been to continue the fruitful partnership we obtained through earlier projects, and include and formalize educational issues, such as guest lecturers, workshops, joint courses and internships. Furthermore, exchange of MSc and PhD students on internships in Japanese and Norwegian aluminium industry and universities have been a prioritized activity. During 2021 there has been extremely low activity in the INTPART project due to the pandemic. However, we see from the publication list that earlier collaboration starts to show results, as we had many common publications in 2021. In October, we participated in the CAMRIC conference in Toyama by Zoom. Knut and Randi also gave lectures online for Japanese students. The project has been extended for 6 months and will probably be prolonged further. We really hope for more physical meetings in 2022 -The trip to Japan in May 2022 is still 'tentative', and most of the group have submitted abstracts to the ICAA conference in Toyama, Japan in September!

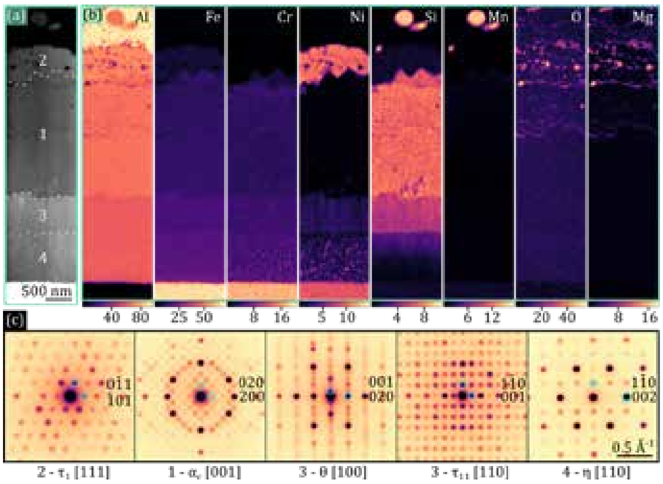
PHD DEFENSES

IN THE TEM GEMINI CENTRE 2021

TINA BERGH, 28. MAY 2021

Tina Bergh worked in SFI Manufacturing and wrote a thesis on 'Electron microscopy of intermetallic phases in aluminium-steel joints'. The overall goal of her work was to characterise the interface microstructure in selected Al-steel joints, and in turn to contribute to better understanding of the underlying bonding mechanisms and the performance of the joints. Intermetallic phases were studied in a more fundamental sense to gain insight into the influence of alloying elements. The joints studied were produced by the solid-state welding methods roll bonding and hybrid metal extrusion & bonding (Hybond), and by the fusion welding method cold metal transfer. The main focus was placed on the interfacial intermetallic phase layers, which typically have thicknesses on the nanometre or micrometre scale in sound joints. Electron microscopy provides the spatial resolution to study such layers. The main tool was transmission electron microscopy, which enables acquisition of a broad range of complementary signals that can be used to characterize the specimen both in terms of morphology, chemical composition, and crystal structure, with a spatial resolution down to atomic scale. Tinas supervisor was Per Erik Vullum, co-supervisors were Randi Holmestad and Ida Westermann. Tina is still associated with the TEM Gemini Centre, as she is hired as a postdoc in the SFI Industrial Catalysis Science and Innovation (iCSI) (50%), at the Chemical Engineering Department (25%), and in the project AluBridge (25%) at the Mechanical and Industrial Engineering Department.





Images at the top: *schematic figure of an Al/Fe HYB joint, VBF image with overlayed coloured VDF image segments of intermetallic phases, STEM-EDS element maps from composites, and precession electron diffraction pattern of intermetallic phases.*
Image at the bottom: *the two opponents are shown on the screen behind - they were Prof. Dr.-Ing. habil. Hauke Springer, RWTH Aachen University, Germany and Dr. Eva Mørtsell, Hydro Sunndal. In front from left: Per Erik Vullum (supervisor), Knut Marthinsen (administrator) and the new PhD Tina Bergh.*

Snapshots from the trip to Teveltunet 2021

Gathered for dinner

R. Holmestad

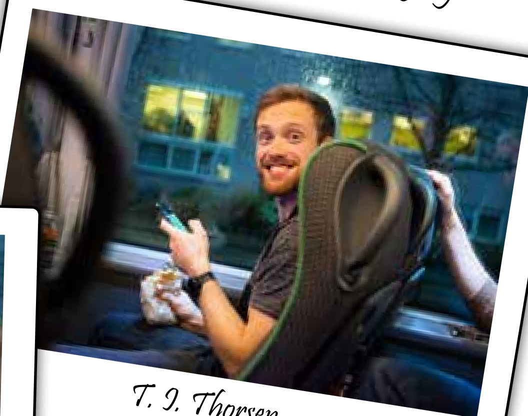
U. Ludacka and P. E. Vullum

E. F. Christiansen

M. Nord enjoying the peace and quiet

R. Bjørge

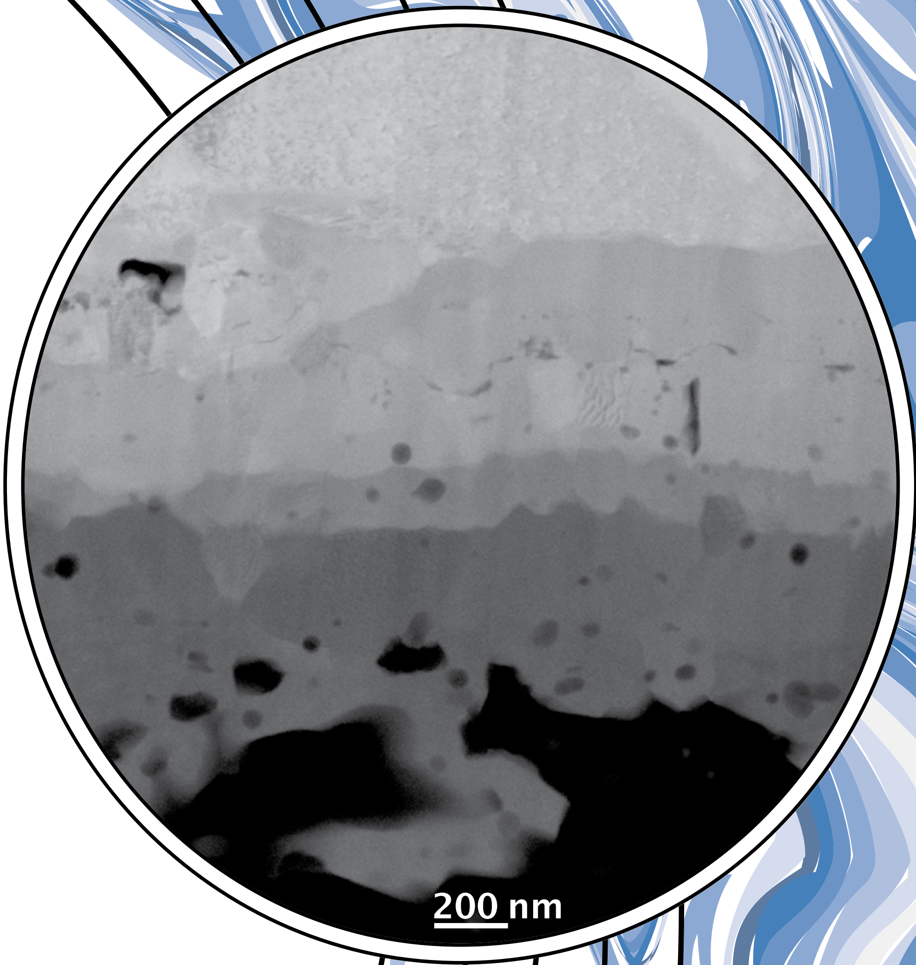
H. Lysne



T. J. Thorsen

O. Ryggetangen

Images above: *image collage of people from the TGS at Teveltunet, Nov. 2021, by I.-E. Nylund.* ***Image on next page****: Al/Cu HYB joint interface after long time exposure heat treatment, by J. Sørhaug.*



Publications 2021

People in the TEM gemini centre are highlighted

TEM GEMINI CENTRE PUBLICATIONS 2021

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

JOURNAL PUBLICATIONS

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POPULAR SCIENCE

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CONFERENCE CONTRIBUTIONS (A SELECTION)

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Ånes, Håkon Wiik; Hjelen, Jarle; Van Helvoort, Antonius; Marthinsen, Knut. Processing and analysis of EBSD patterns with the Python package kikuchipy. Krakow EBSD meeting 2021; 2021-12-13 - 2021-12-14

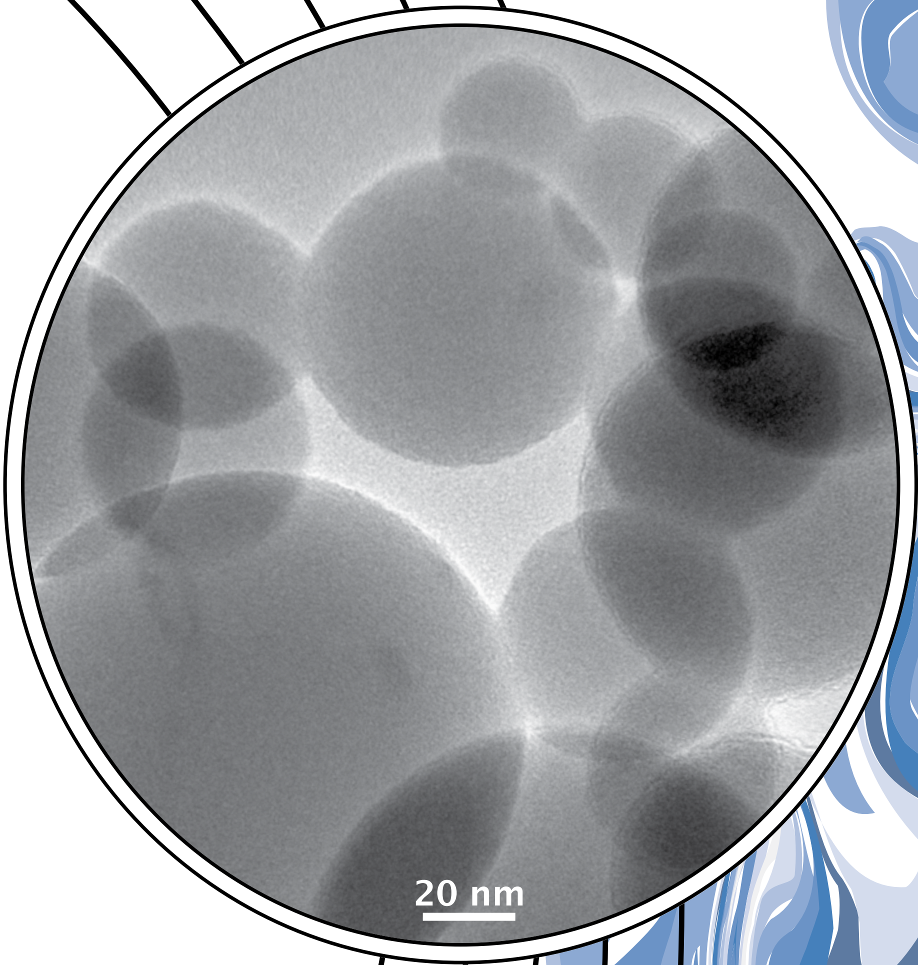
MASTER'S THESES

- Idun Bækken, Characterizing the structural and optical properties of AlGaN nanostructures for ultraviolet LED applications using correlated electron microscopies (Supervisor Ton van Helvoort)
- Sivert Dagenborg, Structural analysis of the uniformity and crystallinity of TiO2-covering on Al-flakes (Supervisor Randi Holmestad)
- Ingeborg Nævra Prestholdt, Strain mapping based on scanning (precession) electron diffraction of heterostructured semiconductor nanowires: set-up and analysis (Supervisor Ton van Helvoort)
- Oskar Ryggetangen, Domain imaging of multiferroic K3Nb3B2O12 by transmission electron microscopy (Supervisor Ton van Helvoort)

PROJECT THESES

- Rajith Aravinth, Characterizing ferromagnetic domains in ring structures using in-situ magnetic Fresnel imaging. (Supervisor Magnus Nord)
- Julie Marie Bekkevold, Magnetic characterization of artificial spin ice structures using (S)TEM (supervisor Magnus Nord)
- Torbjørn Bogen-Storø, Crystal phase mapping based on scanning precession electron diffraction/Extracting meaningful information from Electron Diffraction data (supervisor Ton van Helvoort)
- Martin Lesjø, The effect of cooling from solution heat treatment on ductility in a 6063 crashbox aluminium alloy (Supervisor Knut Marthinsen)
- Marthe Linnerud, Correlated Transmission Electron Microscopy Studies for High Spatial and Lattice Defect Structure Analysis in Single Crystal K3Nb3B2O12 and its Domain Wall Structures (supervisor Ton van Helvoort)
- Knut Håvard Raen, Microprobing of nanowire-based LEDs down to single nanowire level inside a FIB-SEM (supervisor Ton van Helvoort)
- Armand Sepehri Transmission electron microscopy characterization of Fe–Sn thin films deposited with molecular beam epitaxy supervisor Sigurd Wenner)
- Mari Sofie Skomedal, Correlated S(T)EM-TEM-EDX study of core-shell GaAs/AlGaAs Nanowires, (supervisor Ton van Helvoort)
- Hedda Øye, Investigation of aluminium alloys containing Vanadium and Titanium (Supervisor Randi Holmestad)

***Image on next page**: balls of silica (SiO_2), by S. Wenner.*



Selected scientific papers

Microstructural and mechanical characterisation of a second generation hybrid metal extrusion & bonding aluminium-steel butt joint

Tina Bergh, Lise Sandnes, Duncan Neil Johnstone, Øystein Grong, Filippo Berto, **Randi Holmestad**, Paul Anthony Midgley, **Per Erik Vullum**

A B S T R A C T

Hybrid metal extrusion & bonding (HYB) is a joining method that enables solid-state bonding by combining addition of aluminium filler material through continuous extrusion with pressure exerted by a rotating steel tool. This work presents mechanical and microstructural characterisation of a second generation HYB butt joint of aluminium alloy 6082 and structural steel S355. The ultimate tensile strength was measured to be in the range of 184–220 MPa, which corresponds to 60–72% joint efficiency. Digital image correlation analysis of the strain development during tensile testing revealed that root cracks formed, before the final fracture ran close to the aluminium-steel interface. A significant amount of residual aluminium was found on the steel fracture surface, especially in regions that experienced higher pressure during joining. Scanning and transmission electron mi-croscopy revealed that the bond strength could be attributed to a combination of microscale mechanical inter-locking and a discontinuous nanoscale interfacial Al-Fe-Si intermetallic phase layer. Analysis of scanning electron diffraction data acquired in a tilt series, indicated that the polycrystalline intermetallic phase layer contained the cubic α_c phase. The results give insight into the bonding mechanisms of aluminium-steel joints and into the performance of HYB joints, which may be used to better understand and further develop aluminium-steel joining processes.

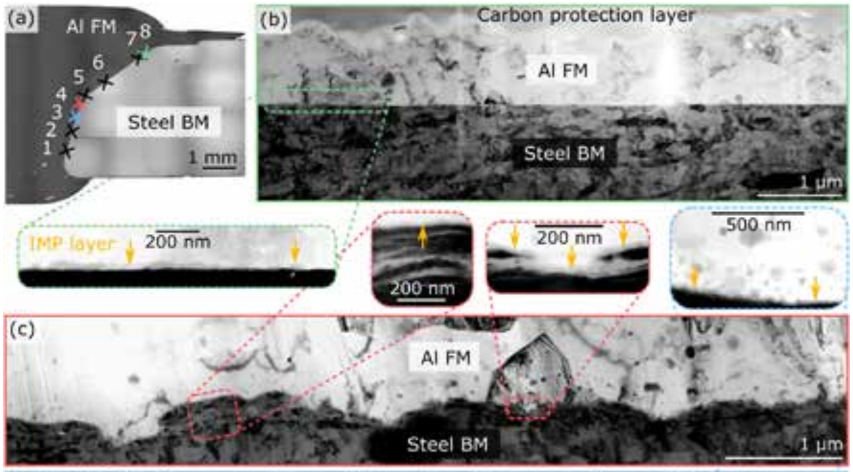


Fig. 7. TEM characterisation of the Al-steel interface region. (a) BSE SEM image showing the locations from where TEM lamellae were lifted out by FIB. (b) BF-TEM image of Lamella 8 showing a straight interface. The inset shows a BF-STEM image of a ~0.9 µm wide IMP layer segment. (c) BF-TEM image of Lamella 4 showing a rough interface region. The left-hand inset shows a BF-STEM image of intermixed Al and steel, while the right-hand inset shows an IMP layer growing around deformed steel fragments.

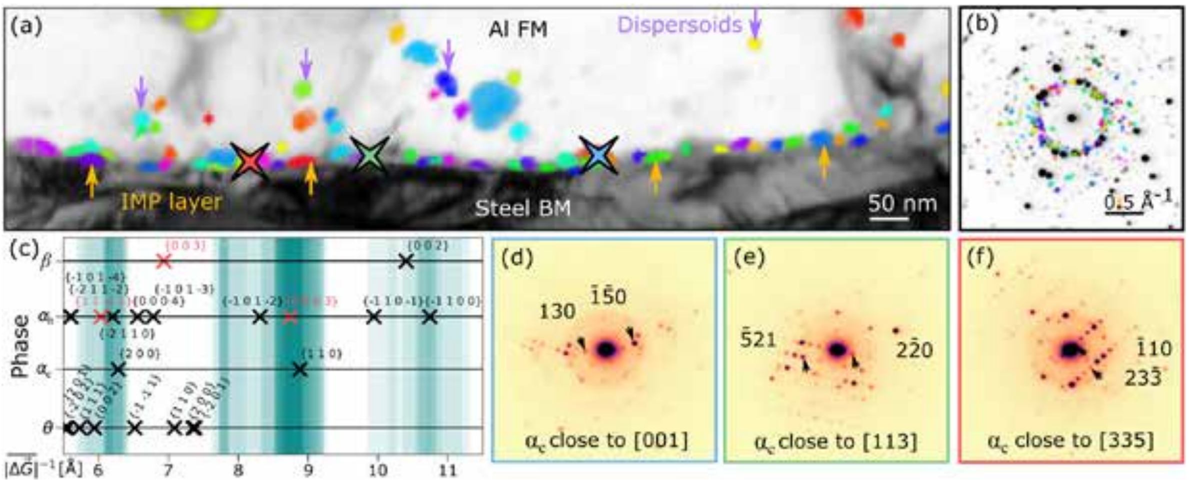


Fig. 10. Scanning electron diffraction of an interface region in Lamella 3 (the same as Fig. 9). (a) Virtual bright field image with overlayed coloured virtual dark field image segments from some individual crystals in the IMP layer and some dispersoids in the Al FM. (b) Coloured diffraction spots corresponding to the coloured segments in (a), superimposed on a greyscale pattern that shows the maximum intensity with respect to detector position based on all patterns from the region in (a). (c) Measured average d-spacings from selected patterns in the IMP layer plotted in partly transparent turquoise with a line width equal to one standard deviation. On the vertical axis, d-spacings of the phases θ, α_c, α_h, and β are shown. Black crosses correspond to kinematically allowed spots, while red crosses correspond to spots possibly seen due to double diffraction. Patterns indexed with respect to the α_c phase from crystals oriented close to (d) [001], (e) [113] and (f) [335] zone axis, located at the positions marked in (a). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

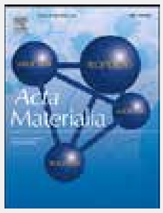
T. Bergh, L. Sandnes, D. N. Johnstone, Ø. Grong, F. Berto, R. Holmestad, P. A. Midgley, P. E. Vullum,
Mater. Charact. **173**, 110761 (2021). https://doi.org/10.1016/j.matchar.2020.110761

Atomic structure of solute clusters in Al–Zn–Mg alloys

A. Lervik, E. Thronsen, J. Friis, C.D. Marioara, S. Wenner, A. Bendo, K. Matsuda, **R. Holmestad, S.J. Andersen**

A B S T R A C T

Scanning transmission electron microscopy imaging of Al–Zn–Mg alloys has provided new information on the atomic structures of solute rich clusters forming from a supersaturated solid solution at low temperatures. A unique unit of high Zn/Mg ratio is the fundamental cluster building block. The unit is essentially a partial substitution by Mg and Zn on the cubic aluminium cell and its surrounding truncated cube octahedral shell. A simple set of principles based on Frank–Kasper structures describes how the basic units arrange with respect to each other to form larger clusters. Density functional theory calculations, atom probe tomography and simulated diffraction patterns support the proposed atomic models. The results provide new insight into the very early stages of age-hardening in aluminium alloys.



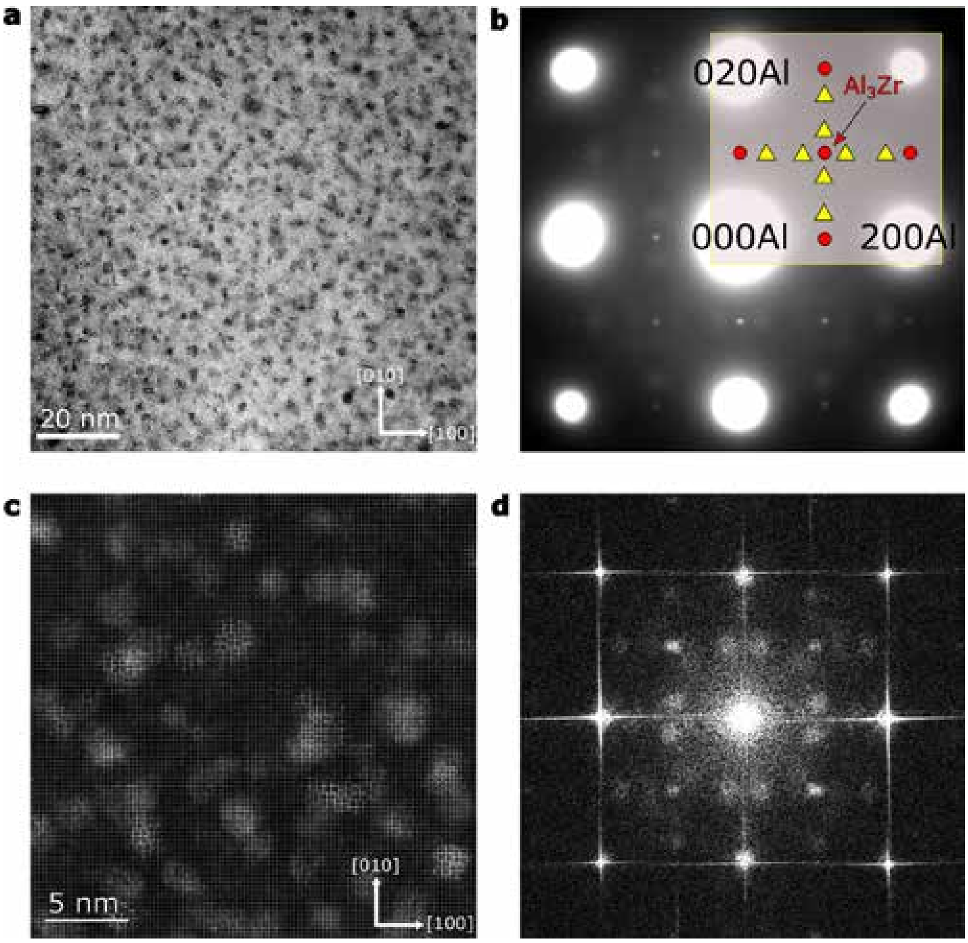


Fig. 1. Alloy #1 after 17 years of natural ageing, oriented along the [001] direction. a, BF-TEM image showing a high density of solute clusters (dark contrast). b, SADP with diffraction spots of Al_3Zr dispersoids and GP(I) zones, marked by circles and triangles, respectively. c, HAADF-STEM image clearly indicating ordering of solute on the fcc lattice. d, fast Fourier transform of c where the intensity in the forbidden {110} positions are due to a surface layer artefact.

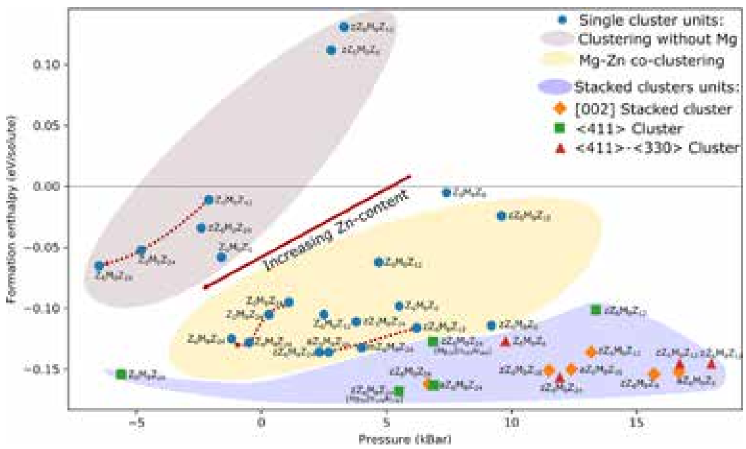


Fig. 4. DFT calculated formation enthalpy versus pressure for cluster structures with different Al, Zn, and Mg content embedded in aluminium matrix. Compositions refer to net content in single cluster units after stacking. Results demonstrate an advantage of high Zn content in the clusters. (Cf. Supplementary Tables 1 and 2 for detailed explanation of the structures.)

A. Lervik, E. Thronsen, J. Friis, C. D. Marioara, S. Wenner, A. Bendo, K. Matsuda, R. Holmestad, S. J. Andersen,
Acta Mater. **205**, 116574 (2021). https://doi.org/10.1016/j.actamat.2020.116574

Journal of Applied Physics

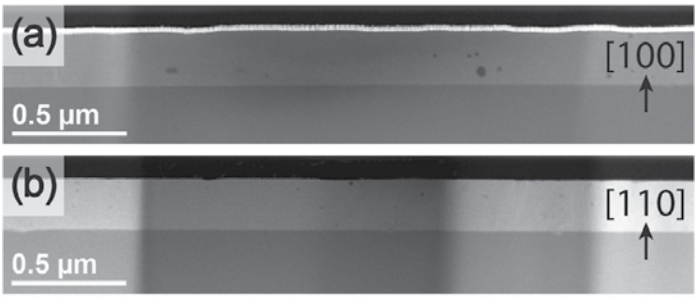
ARTICLE

Epitaxial (100), (110), and (111) $BaTiO_3$ films on $SrTiO_3$ substrates—A transmission electron microscopy study

I.-E. Nylund, T. M. Raeder, P. E. Vullum, and T. Grande

ABSTRACT

Chemical solution deposition (CSD) is a versatile method to fabricate oxide films. Here, the structure and local variations in the chemical composition of $BaTiO_3$ (BTO) films prepared by CSD on (100), (110), and (111) $SrTiO_3$ (STO) substrates were examined by transmission electron microscopy. The films were shown to be epitaxial and the relaxation of the films occurred by the formation of edge dislocations at the substrate–film interfaces. The Burgers vectors of the dislocations were determined to be $a\langle 010\rangle$, $a[1\bar{1}0]$ and $a[001]$, and $a\langle 110\rangle$ for the (100), (110), and (111) films, respectively. Due to the difference in thermal expansion between STO and BTO, the films are demonstrated to be under tensile strain. Furthermore, the boundaries between each deposited layer in the BTO films were found to be Ba-deficient in all cases. In the case of the (111) oriented film, defects like an anti-phase boundary or a thin layer with a twinned crystal structure were identi-fied at the boundary between each deposited layer. Moreover, a larger grain was observed at the film surface with a twinned crystal structure. The interdiffusion length of A-cations at the STO–BTO interface, studied by electron energy-loss spectroscopy, was found to be 3.4, 5.3, and 5.3 nm for the (100), (110), and (111) oriented films, respectively. Interdiffusion of cations across the STO–BTO interface was discussed in relation to cation diffusion in bulk BTO and STO. Despite the presence of imperfections demonstrated in this work, the films possess excel-lent ferroelectric properties, meaning that none of the imperfections are detrimental to the ferroelectric properties.



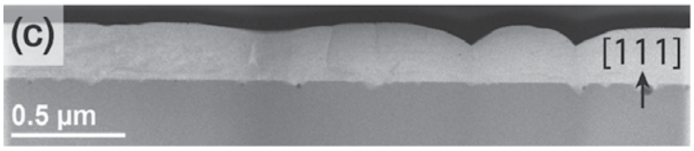


FIG. 1. HAADF-STEM images showing the cross section of the three differently oriented films (a) (100), (b) (110), and (c) (111). Vertical contrast differences occur because of varying lamella thickness, created by the FIB specimen preparation method. The (100) and (110) films show uniform thickness, whereas the thickness of the (111) oriented film varies. The thin, bright contrast layer directly above the BTO film in (a) is Pt/Pd that was sputter coated on top of the sample prior to FIB preparation in order to avoid charging.

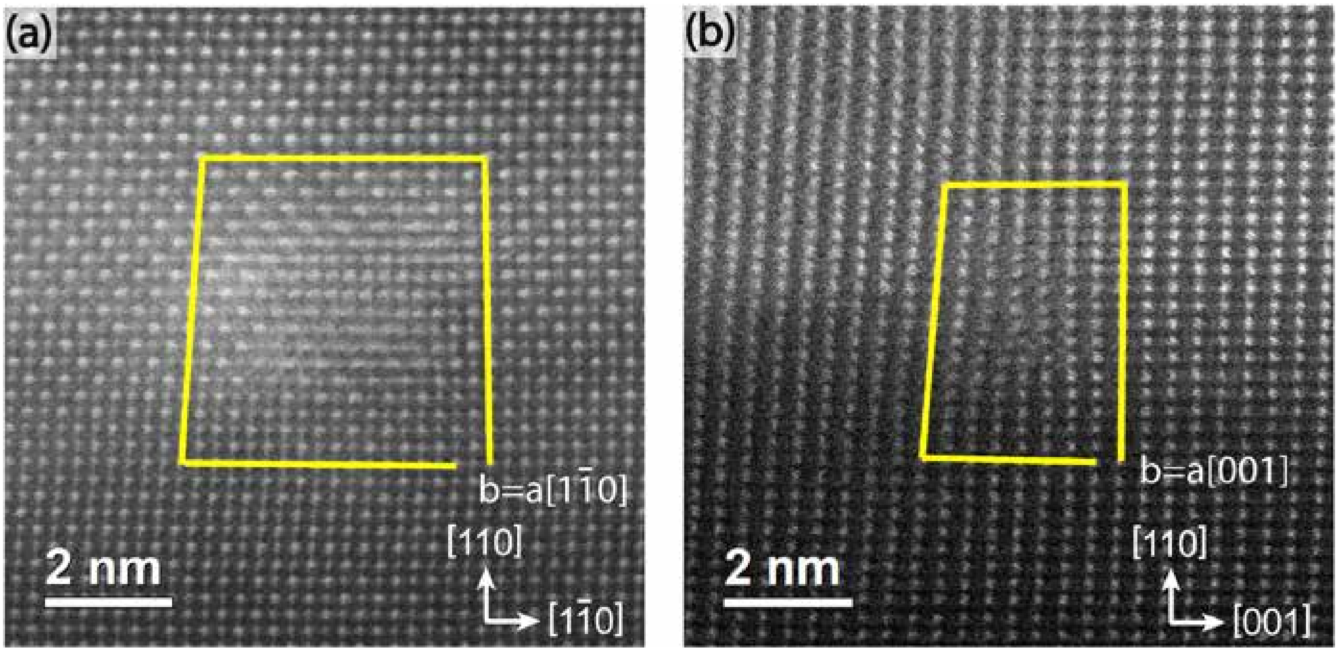


FIG. 5. High-resolution HAADF-STEM images of misfit dislocations in the (110) film imaged along the beam direction (a) [001] and (b) [110]. The Burgers closures demonstrate that the Burgers vectors are (a) a[110] and (b) a[001].

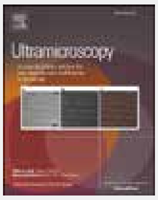
I.-E. Nylund, T. M. Raeder, P. E. Vullum, T. Grande,
J. Appl. Phys. **129**, 095304 (2021). https://doi.org/10.1063/5.0045011

Detecting minute amounts of nitrogen in GaNAs thin films using STEM and CBED

Maryam Vatanparast, Yu-Tsun Shao, Mohana Rajpalke, Bjørn-Ove Fimland, Turid Reenaas, **Randi Holmestad**, **Per Erik Vullum**, Jian Min Zuo

A B S T R A C T

Nitrogen (N) is a common element added to GaAs for band gap engineering and strain compensation. However, detection of small amounts of N is difficult for electron microscopy as well as for other chemical analysis techniques. In this work, N in GaAs is examined by using different transmission electron microscopy (TEM) techniques. While both dark-field TEM imaging using the composition sensitive (002) reflections and selected area diffraction reveal a significant difference between the doped thin-film and the GaAs substrate, spectroscopy techniques such as electron energy loss and energy dispersive X-ray spectroscopy are not able to detect N. To quantify the N content, quantitative convergent beam electron diffraction (QCBED) is used, which gives a direct evidence of N substitution and As vacancies. The measurements are enabled by the electron energy-filtered scanning CBED technique. These results demonstrate a sensitive method for composition analysis based on quantitative electron diffraction.



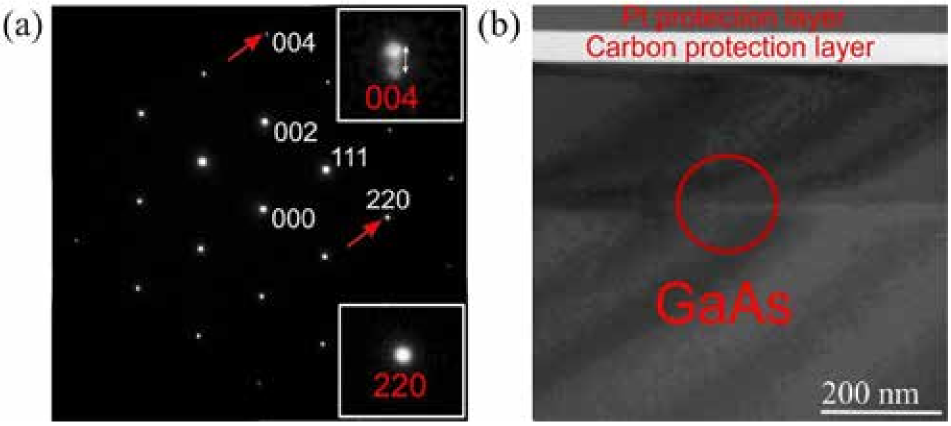


Fig. 3. Diffraction pattern (a) with corresponding BF-TEM image (b) taken from the interface between GaAs and GaN_xAs_{1-x} in the 6.4%N film. The SAED pattern is taken from an area covering the 300 nm in diameter circular region shown with a red circle in (b). The splitting is pointed out by the red arrow in (a) and corresponds to a 1.2% reduction in lattice parameter between substrate and film. The (004) and (220) reflections are pointed out by red arrows in (a) and shown in the insets. The splitting in (004) corresponds to a 1.2% reduction in lattice parameter between substrate and film. (For interpretation of the references to the color in this figure legend, the reader is reffered to the web version of this article.).

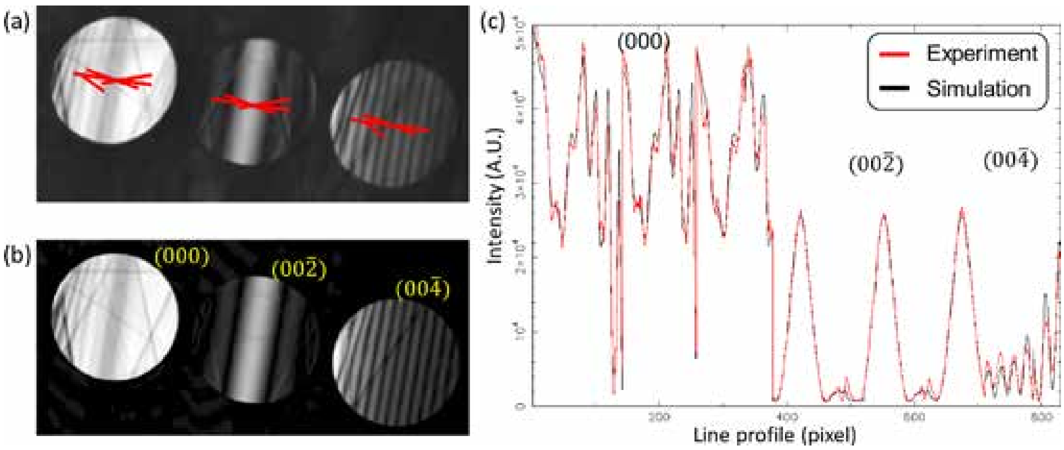


Fig. 8. CBED structure factor refinement for the 1.0%N sample. (a) An experimental energy-filtered CBED pattern acquired at an orientation of ~5° tilt away from the [110] zone axis, and (b) the corresponding simulated pattern after refinement. First, assuming a tetragonal distortion along the c-axis, the lattice parameters (a,c) were refined using the HOLZ lines as a=5.6537 Å and c=5.64745 Å, corresponding to 0.11% strain. The structure factors of U(002) and U(004)were then refined, and the intensity fitting result shown in (c). The red lines in (a) indicate the intensity line profiles used for QCBED fitting. The red and black curves correspond to intensity line profiles of the experimental and refined simulated line profiles, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.).

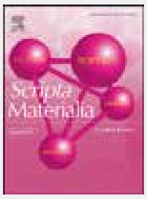
M. Vatanparast, Y.-T. Shao, M. Rajpalke, B.-O. Fimland, T. Reenaas, R. Holmestad, P. E. Vullum, J. M. Zuo,
Ultramicroscopy **231**, 113299 (2021). https://doi.org/10.1016/j.ultramic.2021.113299

The unique hybrid precipitate in a peak-aged Al-Cu-Mg-Ag alloy

M.R. Gazizov, A.O. Boev, **C.D. Marioara**, **S.J. Andersen**, **R. Holmestad**, R.O. Kaibyshev, D.A. Aksyonov, V.S. Krasnikov

A B S T R A C T

The prevalent hardening phase forming in an Al-Cu-Mg-Ag alloy after peak-aging at 150 and 190 °C has been investigated using transmission electron microscopy methods. The precipitate atomic structure was determined. It is a hybrid precipitate (HP) with plate morphology on $\{111\}_{Al}$ planes, consisting of orthorhombic and hexagonal structural fragments. Density functional theory calculations suggest that the hybridization reduces structural incompatibility of the HP plates with the Al matrix at the broad interfacial boundaries. Incorporation of Cu, Mg and Ag in the bulk HP structure reduces its formation enthalpy.



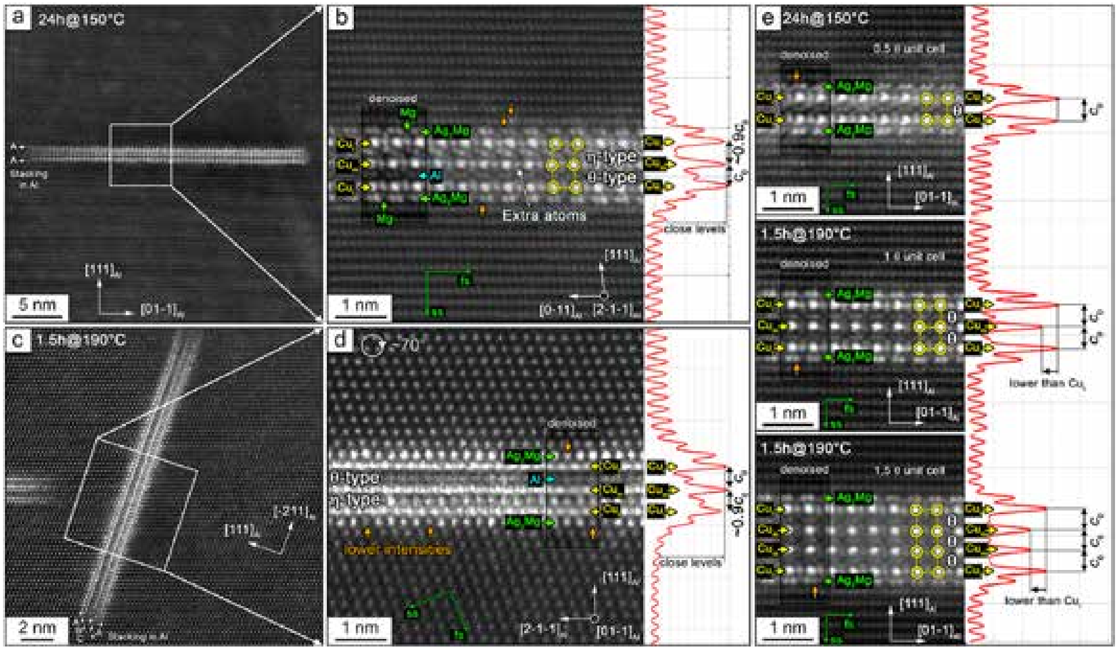


Fig. 2. STEM images showing the hybrid (a–d) and non-hybrid {111} Al plates (e). Fast Fourier transform (FFT) filtering was applied (in b, d, and e) to reduce noise with a periodicity shorter than ~0.05 nm. For each image, fast and slow scanning directions are marked as 'fs' and 'ss', respectively. Non-uniform oscillation of the atomic column intensity along the interface, as indicated by orange arrows, can be evidence of the difference in chemistry between the atom columns.

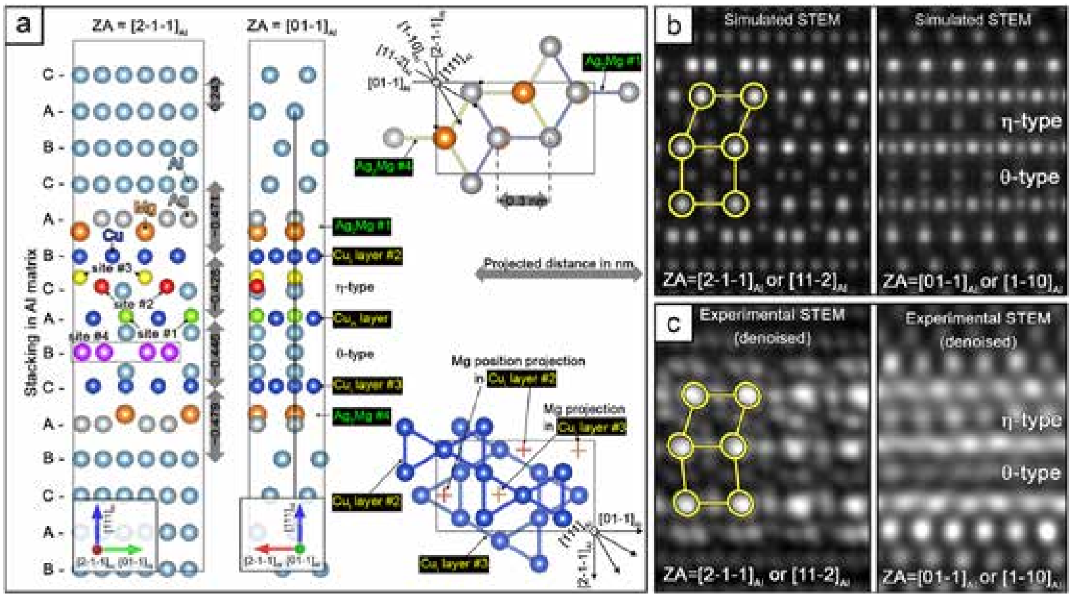


Fig. 3. The models including the HP (a); simulated (b) and experimental denoised STEM images (c). The projection scale was reduced by a factor of 0.5 in [2-1-1] Al . 12 variants of the HP with substitution of the Al atoms in sites #1 –#4 by Cu, Mg and Ag have been checked to find the energetically favorable one (Tables 2 and S4). In STEM image simulations, the elastic electron scattering factors were parameterized as in [20]. The same Debye-Waller factor of 0.5 Å^{-2} was chosen for Al, Cu, Mg and Ag. The sample thicknesses were the same as respective sizes of the DFT model (~10 Å for {110} Al and {211} Al projections). The electron probe parameters (spot size, convergence and collection semi-angles, etc.) were set in the MULTEM software as in our TEM experiments.

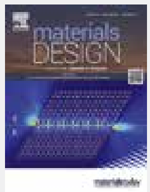
M. R. Gazizov, A. O. Boev, C. D. Marioara, S. J. Andersen, R. Holmestad, R. O. Kaibyshev, D. A. Aksyonov, V. S. Krasnikov, Scr. Mater. **194**, 113669 (2021). https://doi.org/10.1016/j.scriptamat.2020.113669

Precipitation processes and structural evolutions of various GPB zones and two types of S phases in a cold-rolled Al-Mg-Cu alloy

Xuanliang Chen, **Calin D. Marioara**, **Sigmund J. Andersen**, **Jesper Friis**, **Adrian Lervik**, **Randi Holmestad**, Equo Kobayashi

A B S T R A C T

The Guinier–Preston–Bagaryatsky (GPB) zone and the S phase are the key strengthening precipitates in Al-Cu-Mg alloys. However, how their respective structures evolve during aging has not been fully understood. In this work, the age-hardening behavior and the precipitates in an Al–3Mg–1Cu (wt.%) alloy were investigated by means of Vickers hardness measurements, differential scanning calorimetry, transmission electron microscopy, and density functional theory calculations. A series of common GPB zones and a novel type of GPB zone named "GPBX" were observed in the cold-worked samples aged at 443 K for 20 min. In the subsequent aging, two types of S phases were found to coexist, labeled S-I and S-II phases. Density functional theory calculation results indicate that GPBX zone is stable and the S-I and S-II phases have almost the same formation enthalpy. Common GPB zones transform to S-I phases, while S-II phases are formed from GPBX zones preferentially along dislocation lines. The misorientation angles and morphologies of the S phases are also discussed. GPB zones were confirmed to be structurally linked to β" and U2 precipitates reported in 6xxx (Al-Mg-Si) series Al alloys. The revealed precipitate structures and their interrelationships may provide insights into future alloy design.



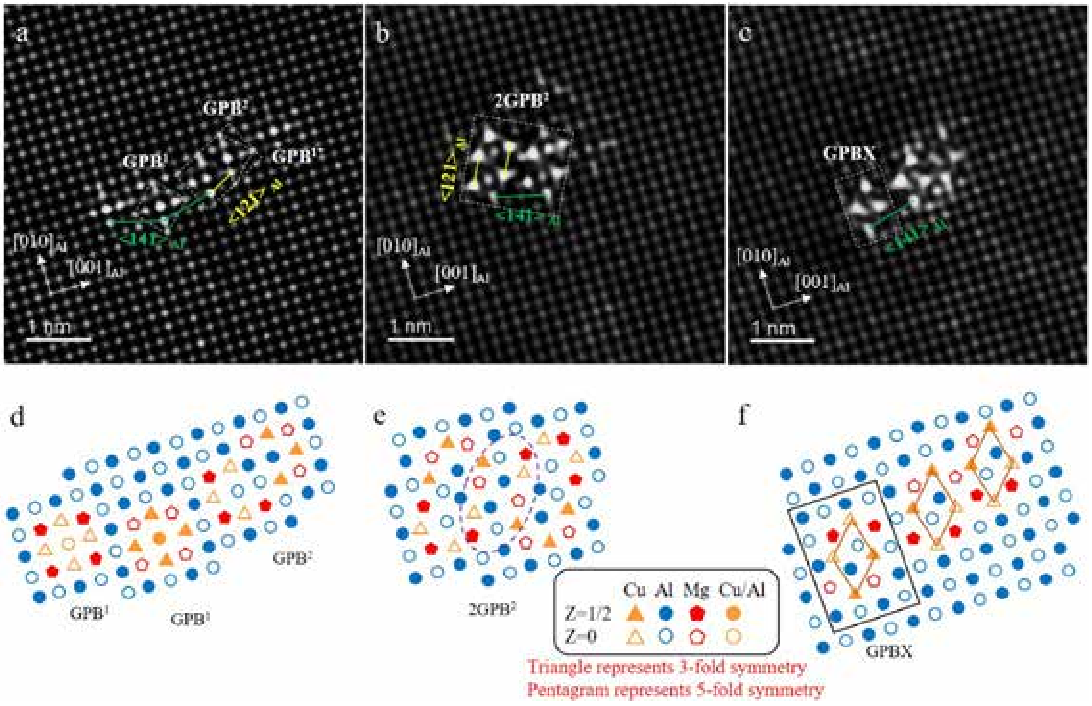


Fig. 2. HAADF-STEM images of the A.R. sample aged at 443K for 20 min (a-c) and the corresponding atomic structures of GPB zones (d-f). (a)(d) GPB1 and GPB2 zones, (b)(e) 2GPB2 zone, (c)(f) GPBX zones.

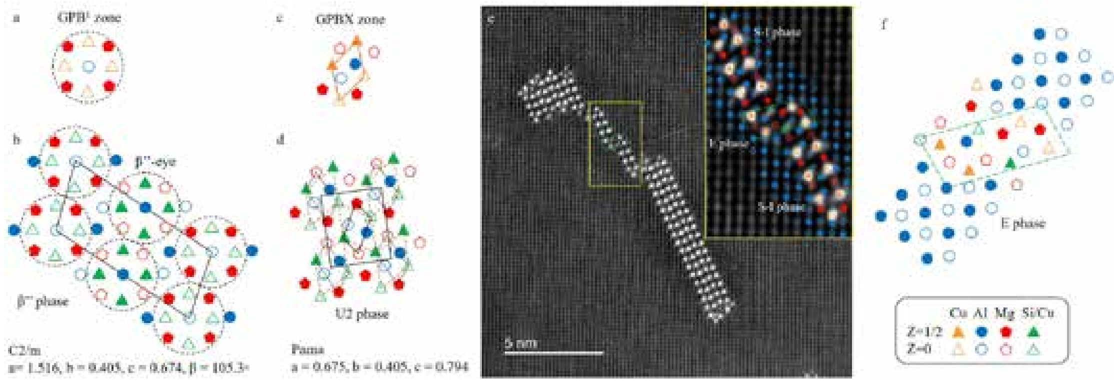
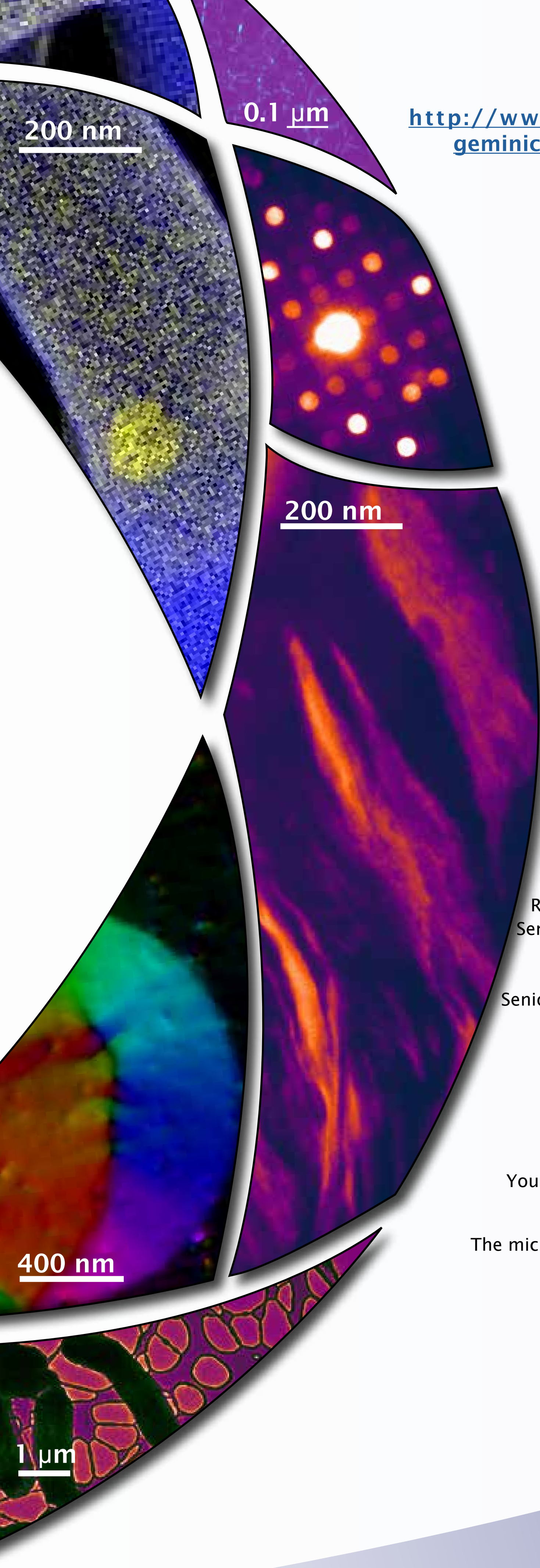


Fig. 10. Atomic models (a-d, f) and HAADF-STEM image (e) of selected precipitates. (a) GPB1 zone, (b) β" phase, (c) GPBX zone, (d) U2 phase, and (f) E phase. (e) HAADF-STEM image of S-I and E phases in the A.R. sample aged at 443 K for 1 day, with the partial atomic overlay.

X. Chen, C. D. Marioara, S. J. Andersen, J. Friis, A. Lervik, R. Holmestad, E. Kobayashi,
Mater. Des. **199**, 109425 (2021). https://doi.org/10.1016/j.matdes.2020.109425



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

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