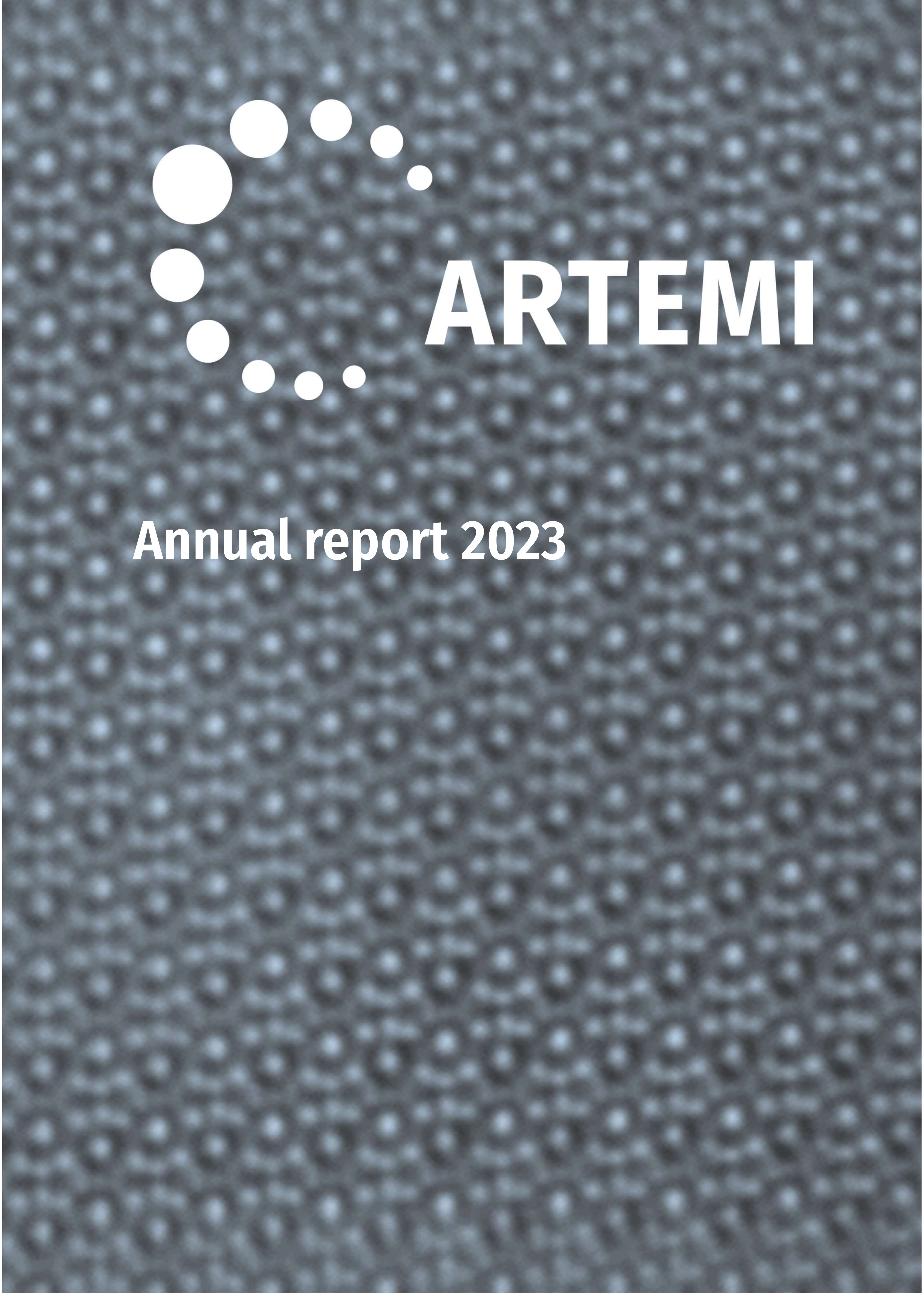

ARTEMI

Annual report 2023

Popular summary, ARTEMI

Since late 2022, ARTEMI is the Swedish national infrastructure in advanced electron microscopy. With nodes that are distributed across Sweden, ARTEMI caters to researchers in materials science, inorganic chemistry, physics, crossover- and adjacent disciplines, in Sweden and internationally.

In the exploration of materials, researchers typically employ X-ray photons, (e.g. MaxLab), neutrons, (e.g. ESS) or electrons (ARTEMI). These particles interact differently with materials and all exhibit complementary information. Electrons benefit from their ability to be focused by electromagnetic fields and for their strong interaction with material. Therefore, as an example, spectroscopy and imaging can be performed with superior spatial resolution, enabling investigations of single atoms, which is beneficiary for studies of e.g. interfaces, low-dimensional and nanostructured materials.

With materials and devices that are increasingly being tailored at the nanoscale, access to direct observation and investigation at this scale is becoming more important. Operation of an advanced electron microscope requires years to learn and master, accordingly the general researcher needs an experienced operator to fully take advantage of all the features on the microscope. The main purpose of ARTEMI is therefore to enable researchers to access expertise, advanced microscopes and to have the ability to perform advanced measurements in a cost-efficient and scientifically rewarding manner. -All through a single entry point: **artemi.se**.

Beyond enabling access to advanced measurements, ARTEMI further develops advanced methods in imaging, diffraction, spectroscopy and *in situ* / *in operando* methods that are made available to the researcher community.

Through continuous and coordinated investments in complementary methods and state of the art equipment, ARTEMI strives for swift access to today's state of the art equipment, with an option for future development.

Front cover: ZIA phase (Nb_3SiNi_2). Collaborative result between LiU and KULeuven.

This is ARTEMI

ARTEMI is a Swedish National Research Infrastructure in Advanced Electron Microscopy, by appointment of the Swedish Research Council (VR).

Constitutional meeting 2022 09 19

Annual Funding:

Swedish Research Council:	6.38	MSEK*
University co-funding	6.38	MSEK*
Swedish Foundation for Strategic Research	3	MSEK

*of which 4 MSEK (total 8 MSEK) towards a new instrument at LiU

Staff

5 Full Time Equivalents, distributed among the nodes.

Mandate period 2022-2026

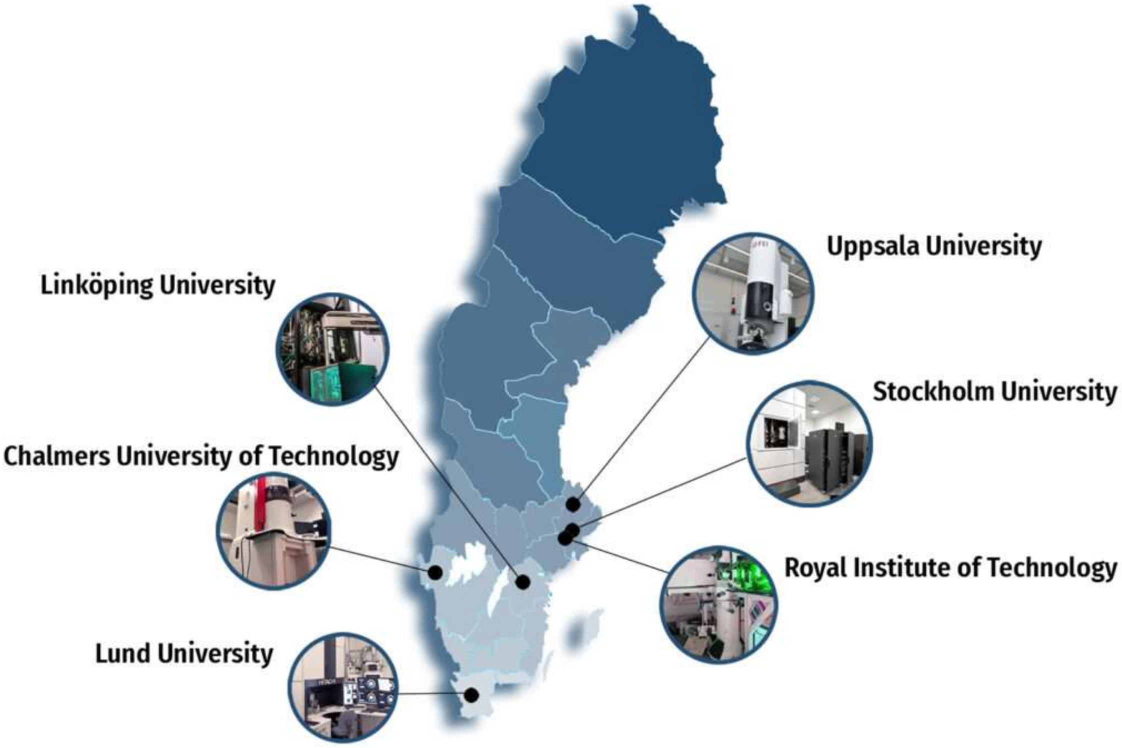

Figure 1 Nodes at Chalmers University of Technology, Linköping University, Lund University, KTH Royal Institute of Technology, Stockholm University and Uppsala University.

Economy

The ARTEMI result for 2023 in view of the budget is described by the following table:

Outcome per category, kSEK	Result	Budget	*% of Budget*
INCOME			
Funds from VR-RFI	6 378	6 377	*100%*
Co funding (same as from VR)	6 430	6 377	*101%*
Funds from SSF-RIF	3 000	3 000	*100%*
Income	15 808	15 754	*100%*
COSTS			
Labor costs	4 284	4 452	*96%*
Travel costs	58	0	
Consumables	247	728	*34%*
Other direct costs including dissemination & conferences	70	276	*26%*
Indirect costs including premises	1 861	2 296	*81%*
Infrastructure investment	0	8 000	*0%*
Total costs	6 521	15 753	*41%*
Total costs excluding infrastructure investment	6 521	7 753	*84%*

The outcome is close to budget for labor costs. Some of the funds were not consumed as two nodes were still starting their activities during 2023. Other costs have been lower than expected, e.g. consumables are lower because activities within method development are still picking up speed. Similarly, costs for the board have been lower than expected although travel costs for the board (and the management team) are listed in the unbudgeted category of travel costs. Unused funds from 2023 are brought to the budget for 2024.

Note the outstanding infrastructure investment in a new STEM to LiU.

Annual report

While most efforts during 2022 was to get agreements in place and to establish the organization by a constituent meeting in September that year, 2023 was characterized by efforts to consolidate the infrastructure and increase the interactions between the distributed nodes. These efforts include a range of fundamental activities such as regular meetings among the management team, regular visits by the coordinator to the nodes, and a kick-off meeting organized at LiU. The main objective for these activities was to establish unity and belonging among the ARTEMI members, and these efforts will continue during 2024. A second objective has been to explore the different administrative prerequisites for the nodes, as background for priorities and strategies for ARTEMI.

The board met three times during the year; at LiU in conjunction with the kick-off meeting, at Chalmers and at Stockholm University (hybrid), with the intention to visit all nodes and meet all staff on a regular basis. In conjunction with these meetings ARTEMI has initiated *Dialogues* with local universities, and while in Stockholm also with The Swedish Research Council (VR) and with the Swedish Foundation for Strategic Research (SSF) – for the purpose of identifying expectations on ARTEMI.

Apart from deciding on a strategy for 2024, the year has included many other projects within ARTEMI, the main ones described below.

Design Language Among the prioritized items for 2023 was to develop a Design Language for ARTEMI, including a logotype, to help ARTEMI in our communications. As can be understood from this document, this was completed and the ARTEMI Design Language has been adopted since. One of the main goals for 2024 is to inform more researchers about our activities and for that a unified language is required to coherently promote the infrastructure.

JIRA Reporting System To keep track of activities within ARTEMI to make the instruments available to the researcher community, we have adopted JIRA, a project management system. This software is web based and enables us to log our efforts within each individual project regardless of at which node the project is pursued. The system was initially tested at LiU, after which it was implemented at other nodes.

New STEM Among the requirements that came with the infrastructure mandate from VR, was to procure a new electron microscope to LiU. The budget associated with the intended state-of-the-art instrument at the time for application was around 40 MSEK. As a result of the pandemic, costs for electronics increased and a weak Swedish currency resulted in insufficient funds available for the procurement. At present, an equivalent instrument comes at a price above 50 MSEK. Accordingly, efforts to raise additional funding and to arrive at a procurement were prioritized during 2022 and 2023. An application to the WISE initiative to fund technology platforms in sustainable materials research bridged the insufficient funds and an invitation for tenders was published late 2023, towards an expected installation before summer 2025.

It should be noted that apart from LiU the nodes at SU and LU were also able to secure funding through the WISE technology platform initiative for new instruments in the years to come.

Access and publications 2023

- **28 publications**
    - LU-1, KTH-1 and LiU-26
- **54 applications for access**
    - UU-2, SU-10, LU-3, CHALMERS-5, KTH-7, LiU-27
- **1356 hours of access to external users registered**
    - UU-95, SU-46, LU-24, CHALMERS-344, KTH-351, LiU-496

The numbers reflect differences in how the nodes operate and in readiness. E.g. at KTH, experiments typically run for an extended period of time, such that a single experiment can last for a week, while a single experiment at LiU may require only 1 hour. Regarding differences in publications and submitted projects, LiU has the advantage, both because this node is leading the work and has therefore been the first adopter of the procedures, which were exported to the other nodes during the year. Particularly the publications reflect this difference, as publications typically are published long after access was granted. In comparison, the differences between LiU and other nodes in terms of access and applications are lower, reflecting the accelerating activity at the other nodes.

The Coming Year

Internal and external meetings together have resulted in an internal strategy document to guide ARTEMI among prioritized activities for 2024. Our main emphasis lies on outreach and interactions with the ambient researcher community, in order to increase our user base and activities in providing access to the state of the art equipment and expertise within ARTEMI.

Per Persson, Coordinator ARTEMI

Research example - Nanoparticle composition

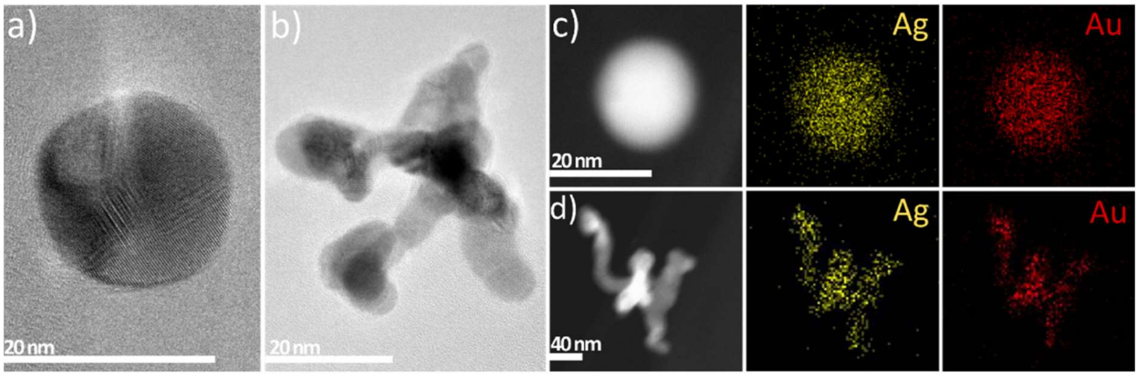

Figure 2 a) A sintered AuAg particle, b) an unsintered agglomerated AuAg particle and c,d) XEDS maps of the elements, showing even alloying down to atomic level.

What is the composition of nanoparticles produced by spark ablation (SA)? This is a long-standing question for the community that use SA to produce bimetallic particles. The particles are formed from creating a high-voltage spark between two electrodes in a flowing inert gas. The electrodes can consist of one element each, or pre-formed alloy electrodes can be used.

Analytical high-resolution transmission electron microscopy is one of the few methods that can give simultaneous information about particle size, degree of agglomeration and chemical composition of *individual* nanoparticles. There are, however, many other methods employed by e.g. the Aerosol community using collective properties of a large number of particles, and the results from these are compared to TEM-XEDS, to reveal any systematic discrepancies. The result show that TEM-XEDS is a reliable, reproducible method with high precision. The starting system was alloyed electrodes of two miscible metals; Ag and Au.

This work has been performed by Linnea Jönsson/Maria Messing at Solid State Physics, in collaboration with ARTEMI in Lund, Ergonomics and Aerosol Technology, Occupational and Environmental Medicine, Chemical Engineering, Mathematical Statistics and in Hungary, Optics and Quantum electronics.

Publication: *L. Jönsson et al. Journal of Aerosol Science 177 (2024) 106333*

Figure 3 Linnea Jönsson & Maria Messing

Research example – Bi_2GeTe_4 Crystal structure

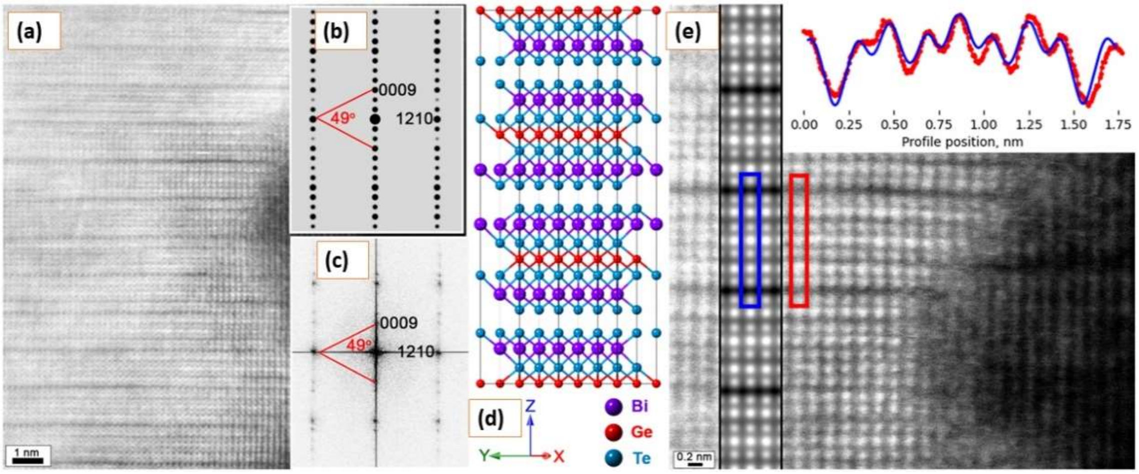

Figure 4 (a) Cross-sectional STEM image, (b) simulated electron diffraction and (c) corresponding FFT pattern of the (d) Bi2GeTe4 crystal viewed in [210] direction. (e) HR-STEM image acquired at higher magnification. The overlay shows the simulated HR- STEM image of the Bi_2GeTe_4 crystal viewed in [210] direction while the inset presents averaged intensity line profiles obtained from the experimental (red rectangle) and simulated (blue rectangle) HR-STEM images.

The next generation of computing will be driven by the quantum phenomena and associated technologies, where interactions of light with matter and understanding of electronic spin-charge in solids will become primary control parameters. Quantum materials will offer a robust platform for exploring and harnessing these control parameters in advanced hardware technologies.

Chalcogenides are a class of fundamental materials which shows several interesting quantum phenomena including superconductivity, charge density wave and quantum topological nature for spintronics and quantum qubit applications. However, to observe and probe the quantum phenomena, we require extremely high quality crystalline materials for substrate as well as components of electronic device fabrications. In general, the crystalline defects such as vacancies, dislocations, interfaces and grain boundaries can influence the overall performance of the materials. Thus, to get a better understanding of the atomic arrangements in the material system, advanced electron microscopy provides direct observation of the structure at the atomic scale, providing insights into their formation mechanisms and effects on electronic transport.

The layered Bi_2GeTe_4 is one of the topological quantum materials with an intriguing crystal structure made up of periodic repetition of seven layered atomic slabs separated by van der Waals gaps. Bi_2GeTe_4 exhibits a hierarchical layered crystalline structure with distinct chemical bonding arrangements (Figure 3).

The tremendous expertise within ARTEMI was instrumental in visualizing the atomic arrangement within Bi_2GeTe_4, a task made challenging due to the delicate nature of the samples and the unique crystal structure of the material. Through low dose microscopy studies, we gained deeper insights into the atomic environments of Bi_2GeTe_4. Figure 3e shows the actual and the simulated crystal structure. This joint effort focused on low-temperature magneto-transport studies and revealed a pronounced increase in conductivity near zero magnetic field, indicative of weak anti-localization effects, which underscores the significance of topological surface states for spintronic applications. Preparing such materials demands a unique skill set and is challenging to achieve in nature. Even subtle alterations in the crystal structure can profoundly impact electronic and magnetic properties. Conventional characterization techniques often fall short in precisely determining the position of germanium within Bi_2GeTe_4, as germanium occupies a central position and remains inert in lattice dynamics, making identification of structural and morphological characteristics difficult.

Figure 5 Dr. Ajay Soni group at Indian Institute of Technology.

Method development -Time resolved 3D electron diffraction – Structures at high speed

3-dimensional electron diffraction (3DED) is an established technique that allows for ab-initio three-dimensional crystal structure determination. Advantages of 3DED include that the technique can extract reliable information from very small samples, inhomogeneous transitions, and can be conducted using instrumentation available at university labs, with no need for large scale infrastructures. Over the last few years, automation has made 3DED widely accessible and increasingly user friendly, rendering 3DED competitive with other three-dimensional techniques such as X-ray diffraction or neutron diffraction.

However, 3DED has been restricted to static structures and lacks information of ultrafast dynamics highly desired for applications in materials science, chemistry, and biology. Extending 3DED to the time domain would facilitate the recording of movies of samples as they undergo a change in state, at material relevant spatial (atomic) and temporal (femtosecond) resolutions.

In this joint development effort between KTH and SU, we push 3DED beyond *state-of-the-art* and demonstrate its extension to the ultrafast domain. With approximately 100 femtosecond time resolution we can follow phase transitions at their intrinsic time scale. Compared to conventional operation of electron microscopes, which exhibits excellent coherence and brightness, an ultrafast TEM is characterized by pulsed operation with low electron flux. We show that despite these limitations, 3DED is feasible in the ultrafast regime and offers immense advantages over conventional 2D diffraction.

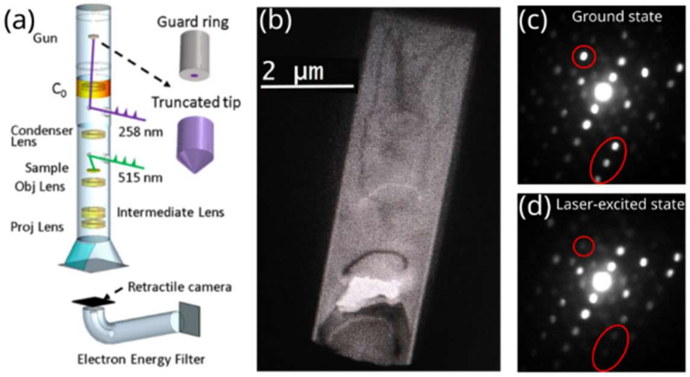

Figure 6 Time-resolved imaging of the structural phase transition in VO2. (a) the Ultrafast transmission electron microscope (UTEM) at KTH. (b) High-quality single-crystalline VO2 lamella prepared using FIB at KTH. (c-d) Electron diffraction in VO_2 prior and after laser excitation measured as part of the 3DED measurements. The diffraction measurements are repeated over many sample tilts.

Our drosophila material system is vanadium dioxide (VO_2), which exhibits a concomitant structural and electronic phase transition near 68°C. This phase transition can be optically driven at below 100 fs and is consequently of great interest for the development of advanced electronic devices, such as high-speed switches and optical detectors. The ultrafast dynamics of this transition are not fully understood, which has hindered its application in devices.

Here, by using time-resolved 3-dimensional electron diffraction (tr-3DED), we show that the light-induced phase transition behaves differently from the thermally driven transition. We interpret the results that laser excitation induces acoustic strain waves in the sample, exerting stress on the order of GPa, which leads to a different phase transition trajectory than expected. Given that some of the known crystal structures in VO_2 have remarkable similarities, we found that tr-3DED is crucial for unambiguously distinguishing between them.

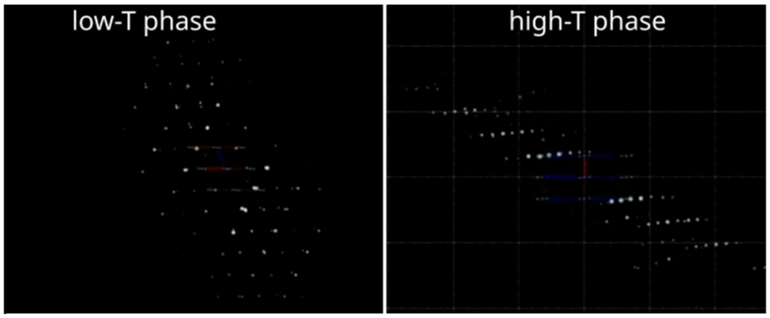

Figure 7 Reconstructed crystal structure using tr-3DED. Surprisingly, we find that the laser-induced structural transition follows a different path compared to the heat-induced phase transition, tentatively influenced by stress in the lamella sample.

In summary, we have demonstrated the feasibility of 3DED in the ultrafast regime, which allowed us to unequivocally determine the changes in crystal structure taking place *during* the transition of VO_2. We plan to extend our study by collecting more data sets at later time delays with respect to the laser excitation and hope to observe another structural phase transition once the stress in the lamella has decayed. We believe that ultrafast 3DED will become indispensable for transient structural determination.

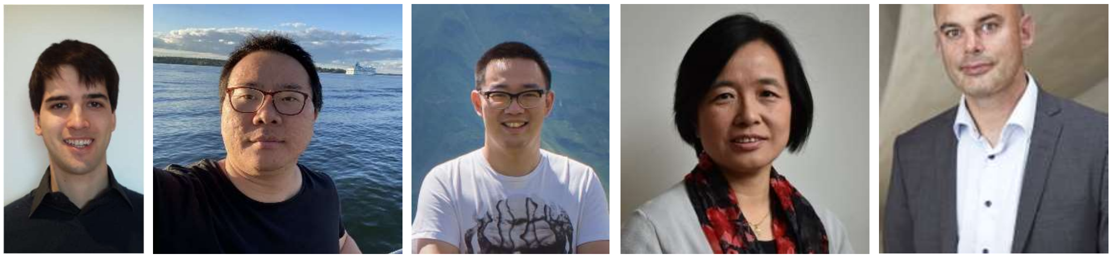

Figure 8 This method was developed in collaboration between researchers at the SU and KTH ARTEMI nodes: Arthur Niedermayr (KTH), Gaolong Cao (KTH), Hongyi Xu (SU), Xiaodong Zou (SU), and Jonas Weissenrieder (KTH).

Method development -New EELS Spectrometer at Uppsala University

Figure 9 Olivier and Sharath with the Themis at Uppsala University. The CEFID filter, mounted under the TEM is shown in the inset with its 90-degree prism.

With the increasing use of the scanning transmission electron microscopy (STEM) technique in our aberration-corrected TEM, atomic resolution spectroscopy techniques have become feasible and contribute essentially to the understanding of modern materials. To build advanced analysis techniques and to analyse novel materials, Uppsala University decided to equip its current Titan Themis aberration-corrected TEM with an energy filter. The base of this energy filter is an electron energy loss spectrometer (EELS) where the elemental edges in the spectra obtain information about the structure and electronic structure of a material. The energy filter is equipped with two excellent cameras, one CMOS camera, mainly for energy-filtered imaging, and one hybrid camera for EELS spectroscopy.

With this energy filter, it is possible to characterise with down to subatomic resolution materials properties such as chemical composition, oxidation states, the structural allotropes that are locally present, interfacial electronic structures and magnetic moments to name a few.

Furthermore, the hybrid camera can acquire up to 13.000 images per second and allows therefore for the acquisition of 4-dimensional STEM maps of diffraction patterns to analyse strain, magnetic and electrical fields, and structure with very high resolution.

This CEFID energy filter is available for the collaboration partners of the ARTEMI infrastructure and will equally be used for building novel spectroscopy techniques for ARTEMI. We believe that the filter equipped with its excellent cameras is a disruptive change as compared to the previous generation of energy filter and will make many exciting applications feasible.

Publications 2023

28 publications, Highest impact: 55 (Science), Average imapct: 6.4, Median impact 5

1. Nonequilibrium phonon dynamics and its impact on the thermal conductivity of the benchmark thermoelectric material SnSe
   ACS Nano 17, 21, 21006–21017 (2023)
   doi.org/10.1021/acsnano.3c03827

2. Structural stability under Xe-ion irradiation of TiZrNbTaV-based high-entropy alloy and nitride films
   Surface and Coatings Technology 454, 129198 (2023)
   doi.org/10.1016/j.surfcoat.2022.129198

3. Enhanced thermoelectric properties of Mg_2Sn-Mg_3Sb_2 nanocomposites by tailoring matrix/inclusion interface toward energy harvesting applications
   ACS Applied Nano Materials 6, 7, 6133–6140 (2023)
   doi.org/10.1021/acsanm.3c00439

4. Tuning composition in graded AlGaN channel HEMTs toward improved linearity for low-noise radio-frequency amplifiers
   Applied Physics Letters 122, 153501 (2023)
   doi.org/10.1063/5.0141517

5. Microstructure control and property switching in stress-free van der Waals epitaxial VO_2 films on mica
   Materials & Design 229, 111864 (2023)
   doi.org/10.1016/j.matdes.2023.111864

6. Phase formation in CrFeCoNi nitride thin films
   Physical Review Materials 7, 055002 (2023)
   doi.org/10.1103/PhysRevMaterials.7.055002

7. Correlating cathodoluminescence and scanning transmission electron microscopy for InGaN platelet nano-LEDs
   Applied Physics Letters 123 (2), 022103 (2023)
   doi.org/10.1063/5.0150863

8. Structural investigation of ultra-low resistance deeply recessed sidewall ohmic contacts for AlGaN/GaN HEMTs based on Ti/Al/Ti-metallization
   Semiconductor Science and Technology 38 (10) 105006 (2023)
   doi.org/10.1088/1361-6641/acf396

9. Upscaled synthesis protocol for phase-pure, colloidally stable MXenes with long shelf lives
   Small Methods, 2300776 (2023)
   doi.org/10.1002/smtd.202300776

10. Room temperature two-dimensional electron gas scattering time, effective mass, and mobility parameters in $Al_xGa_{1-x}N/GaN$ heterostructures (0.07≤x≤0.42)
    Journal of Applied Physics 134, 185701 (2023)
    doi.org/10.1063/5.0163754

11. Room temperature ferromagnetism in the nanolaminated MAX phase $(Mn_{1-x}Cr_x)$2GaC
    APL Materials 11 (12), 121102 (2023)
    doi.org/10.1063/5.0176571

12. Phase separation paths in metastable $Zr_{1-x}Al_xN$ monolithic layers compared to multilayers with TiN: Growth versus annealing temperatures
    Materialia 28, 101758 (2023)
    doi.org/10.1016/j.mtla.2023.101758

13. Effect of modulation period and thickness ratio on the growth and mechanical properties of heteroepitaxial c-$Ti_{0.4}Al_{0.6}N$/h-Cr_2N multilayer films
    Surface and Coatings Technology 472, 129921 (2023)
    doi.org/10.1016/j.surfcoat.2023.129921

14. Efficient CuO/Ag_2WO_4 photoelectrodes for photoelectrochemical water splitting using solar visible radiation
    RSC advances 13 (17) 11297-11310 (2023)
    doi.org/10.1039/D3RA00867C

15. Kinetically controlled synthesis of quasi-square $CsPbI_3$ nanoplatelets with excellent stability
    Small 2306360 (2023)
    doi.org/10.1002/smll.202306360

16. High-mass metal ion irradiation enables growth of high-entropy sublattice nitride thin films from elemental targets
    Journal of Vacuum Science & Technology A 41 (6), 063108 (2023)
    doi.org/10.1116/6.0003065

17. Discovery of Guinier-Preston zone hardening in refractory nitride ceramics
    Acta Materialia 255, 119105 (2023)
    doi.org/10.1016/j.actamat.2023.119105

18. Single-phase growth, stabilization, and electrical properties of B phase VO$_2$ films grown on mica by reactive magnetron sputtering
Advanced Physics Research, 2300032 (2023)
doi.org/10.1002/apxr.202300032

19. Efficient CuO/Ag$_2$WO$_4$ photoelectrodes for photoelectrochemical water splitting using solar visible radiation
RSC Advances 13, 11297-11310 (2023)
doi.org/10.1039/d3ra00867c

20. Computationally-driven discovery of quaternary tantalum-based MAB-phases: Ta$_4$M"SiB$_2$ (M" = V, Cr, or Mo): synthesis, characterization, and elastic properties
Crystal Growth & Design 23, 6, 4442–4447 (2023)
doi.org/10.1021/acs.cgd.3c00197

21. Structural stability under Xe-ion irradiation of TiZrNbTaV-based high-entropy alloy and nitride films
Surface and Coatings Technology 45, 129198 (2023)
doi.org/10.1016/j.surfcoat.2022.129198

22. Synthesis, characterization, and modeling of a chemically ordered quaternary boride, Mo$_4$MnSiB$_2$
Crystal Growth & Design 23, 5, 3258-3263, (2023)
doi.org/10.1021/acs.cgd.2c01416

23. Tuning composition in graded AlGaN channel HEMTs toward improved linearity for low-noise radio-frequency amplifiers
Applied Physics Letters 122, 153501 (2023)
doi.org/10.1063/5.0141517

24. Enhanced thermoelectric properties of Mg$_2$Sn-Mg$_3$Sb$_2$ nanocomposites by tailoring matrix/inclusion interface toward energy harvesting applications
ACS Applied Nano Materials 6 (7), 6133-6140 (2023)
doi.org/10.1021/acsanm.3c00439

25. Effects of stoichiometry and individual layer thickness ratio on the quality of epitaxial CrB$_x$/TiB$_y$ superlattice thin films
Materials & Design 228, 111842 (2023)
doi.org/10.1016/j.matdes.2023.111842

26. Chemical-scissor-mediated structural editing of layered transition metal carbides
    Science 379 (6637), 1130-1135 (2023)
    doi.org/10.1126/science.add5901

27. Experimental and theoretical investigations of out-of-plane ordered nanolaminate transition metal borides: M_4CrSiB_2 (M = Mo, W, Nb)
    Inorganic Chemistry 62(14), 5341-5347 (2023)
    doi.org/10.1021/acs.inorgchem.2c03729

28. Mo_3Ni_2N nanoparticle generation by spark discharge
    Materials 16(3), 1113 (2023)
    doi.org/10.3390/ma16031113