



esteem3

**Opportunities of transnational access  
for industries in ESTEEM3**

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# Overview

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- Background
- Benefits for industry
- Application process
- Conditions for industry
- Examples from research in JRA materials sectors
- Final comments



- Projective objective
  - Provide Transnational Access (TA) for the academic and industrial research community in the physical sciences to some of the most powerful characterisation techniques available at the nanoscale.
- Application process described on web-site  
<https://www.esteem3.eu/>
  - Send open or lab proposals.
  - Companies are invited to contact industry liason officers or consortium members.
  - Applications evaluated by Transnational Access Proposal Evaluation Committee (TAPEC).



# TEM in industry

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- TEM is expensive - investment, operating costs, competence building.
- State-of-the art is under continuous development.
- Investment in TEM infrastructure challenging for larger companies and likely impossible for SMEs.
- Outstanding facilities are available in Universities and research Institutes.



# Benefits for companies



- Transnational Access in ESTEEM3 is a mechanism to provide fully funded access to TEM facilities in the consortium.
- This includes sample preparation, TEM analysis and data processing.
- TA presents an excellent opportunity to;
  - Carry out small scale studies.
  - Building platform for larger-scale study.
  - Learn about the potential of TEM in your area of interest.
  - Strengthen existing TEM competence.
  - Establish contact with expert TEM research environments.



# Confidentiality and IP



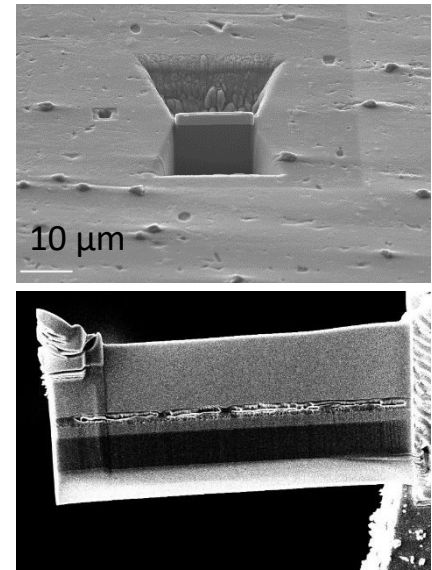
- For larger companies, the outcome of the work must be disseminated.
- SMEs can retain full IP.
- Non Disclosure Agreements can be established for discussion and protection of existing IP.
  - Project descriptions are only seen by the TAPEC committee, the institution performing the work and the company.
  - TA is administered by Euronovia.



# Scope of applications



- Sample preparation
    - E.g Focussed Ion Beam (FIB)
  - TEM analysis
  - Data processing
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- Unit of 1 day and up to 20 days in total.
  - Cannot be all TEM
    - e.g. 6 prep + 6 TEM + 8 data = 20
  - Accommodation can also be supported.







# Examples of topic areas

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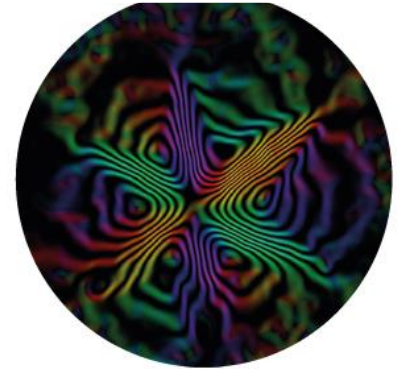
- Area of ongoing industrial TA projects include
  - Nanoscale semiconductor materials
  - Light metal alloys
  - Powder metallurgy
  - 2-D materials



# ESTEEM3 focus areas

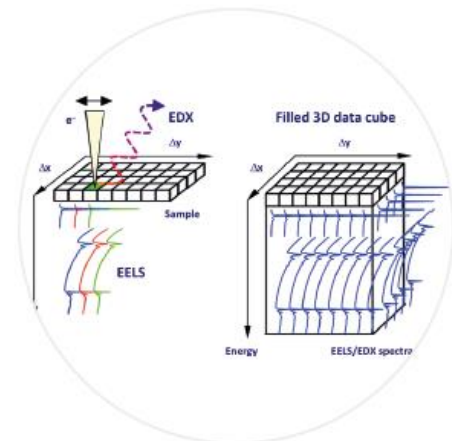


- JRA 1
  - Imaging, Diffraction and Metrology, Spectroscopy, In-situ TEM.
- JRA 2
  - See following slides.
- JRA 3
  - Data acquisition and analysis in TEM - see next slide.



Magnetic induction of a FeCo nanostar observed by off-axis electron holography

Credits picture JRA1 - L.-M. Lacroix and C. Gatel (CNRS-CEMES)



Data processing in EELS and EDS filling up a multidimensional data cube

Credits picture JRA3 - Peter van Aken (MPG-StEM)

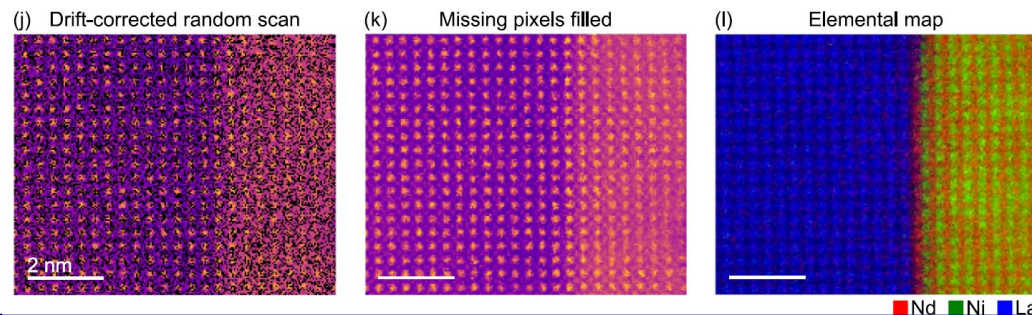


# Data acquisition and analysis



- Instrument control, data acquisition, data processing and machine learning with reduced human intervention using automated and smart workflows.
- Open software for the design and interpretation of experiments, including electron generation, propagation, scattering and detection in standard and specialized imaging modes will be investigated.
- All advances will be directly transferred to TA users.
- Data analysis platforms
  - Hyperspy (<https://hyperspy.org/>) Python library which provides tools to facilitate the interactive data analysis of multi-dimensional datasets.
  - Pyxem (HyperSpy-based): multidimensional diffraction microscopy
  - LumiSpy (HyperSpy-based): analysis of luminescence data
  - LiberTEM: Open-source platform for efficient, parallel and distributed data processing

Spatial and spectral dynamics in STEM hyperspectral imaging using random scan patterns, Zobelli et. Al. Ultramicroscopy, DOI: 10.1016/j.ultramic.2019.112912





## JRA 2 - Eligible areas

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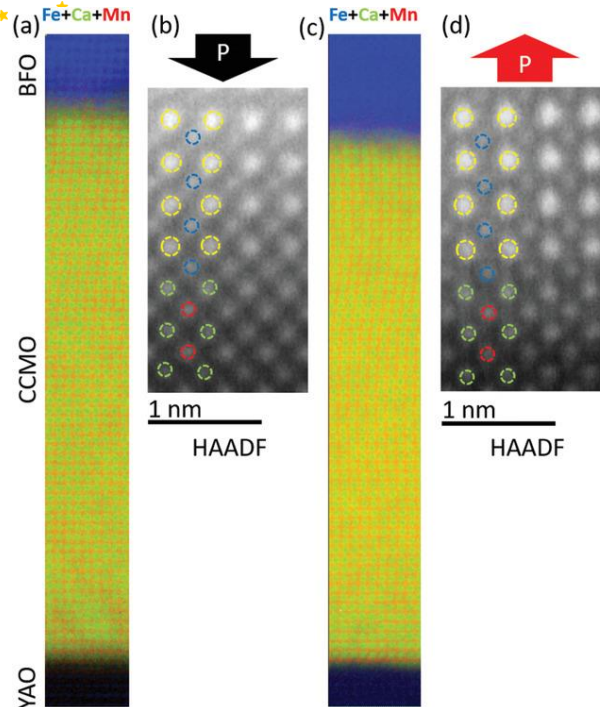


- Proposals should describe relation to the main sectors addressed by JRA 2:
  - Materials for ICT.
  - Materials for Energy.
  - Materials for Health.
  - Materials for Transport.



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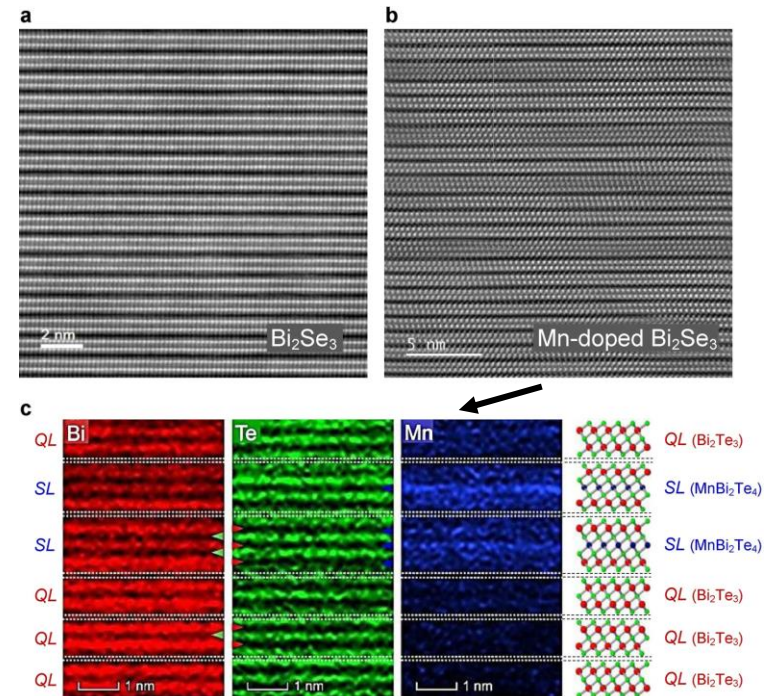
ICT



## In-Depth Atomic Mapping of Polarization Switching in a Ferroelectric Field-Effect Transistor

- Nanoscale changes at interfaces observed directly.
- HAADF and EELS chemical maps.

Li et al, Adv. Mater. Interfaces 2020, 7, 2000601



Large magnetic gap at the Dirac point in  $\text{Bi}_2\text{Te}_3/\text{MnBi}_2\text{Te}_4$  heterostructures

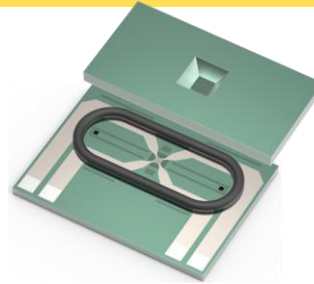
- HR-STEM cross sections and EDS maps show atomic structures.

Rienks, et al, Nature 2019, 576, 423



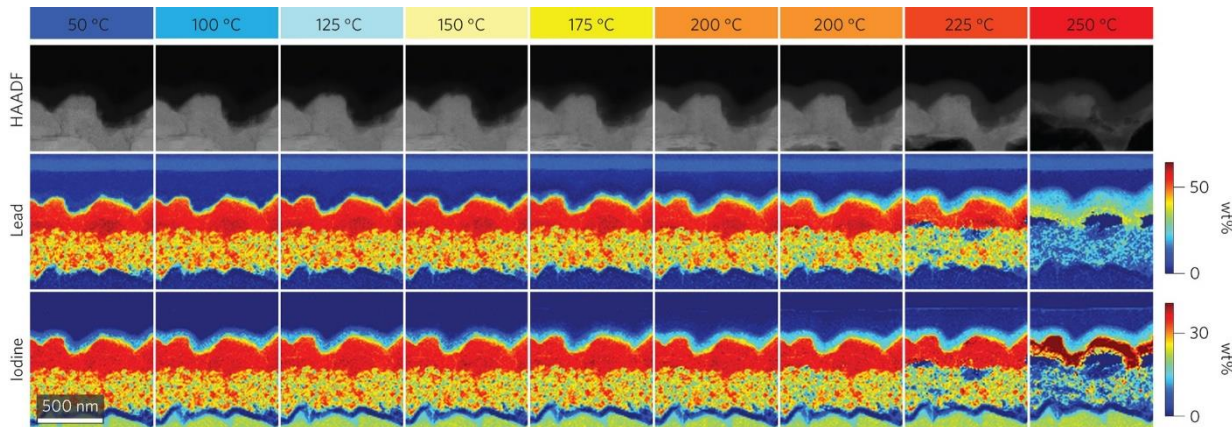


# Energy - in-situ TEM studies



Courtesy DENS solutions

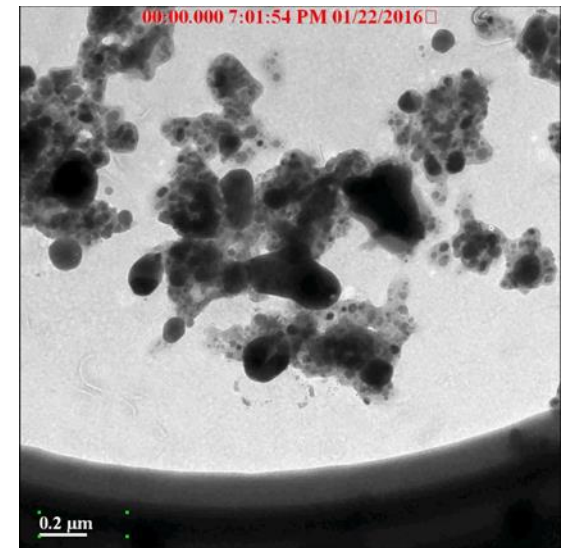
Temperature: 730°C  
Pressure: 1 Bar  
Gas: He+H<sub>2</sub>+O<sub>2</sub> (flow)



*In situ* observation of heat-induced degradation of perovskite solar cells

- Diffusion of I and Pb after increasing temperature steps.
- Diffusion of I into the HTM from the MAPbI<sub>3</sub> is clearly visible at low temperature.
- Pb migration is triggered at higher temperature (~175 C).

Divitini et al, Nature Energy, 2016, 1, 15012

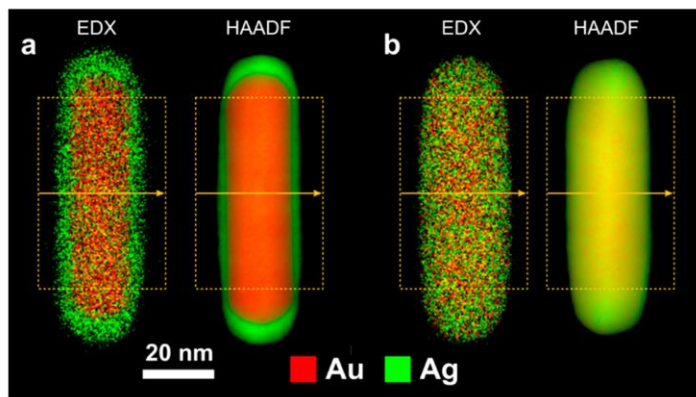


In-situ reduction of NiO catalyst

Researchers:

Dr. R. Fara, Dr. M. Willinger. Fritz Haber Institute Berlin

Dr. Qiang Xu. DENSsolutions



## 3D investigation of AgAu nanoparticles using heating tomography

- Structure reconstructed from STEM images and EDX analysis.
- Metallic NPs have wide range of biomedical applications
- Alloying in AgAu nanoparticles
- Importance for sensing, (optical) hyperthermic cancer treatment

Skoriov et al, ACS Nano 2019, 13, 13421–13429

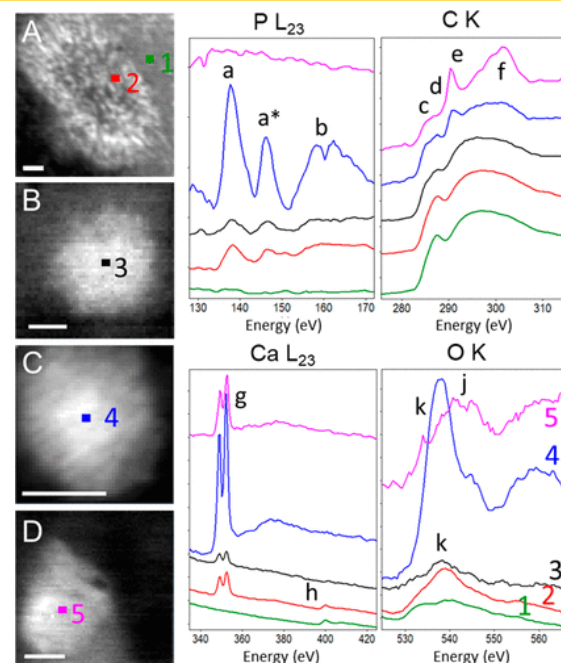
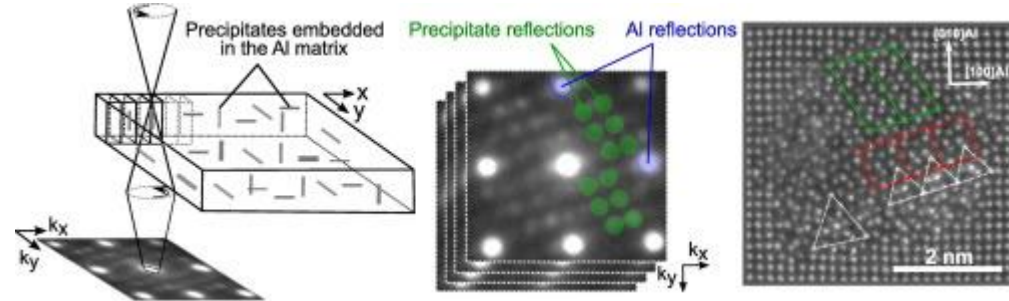
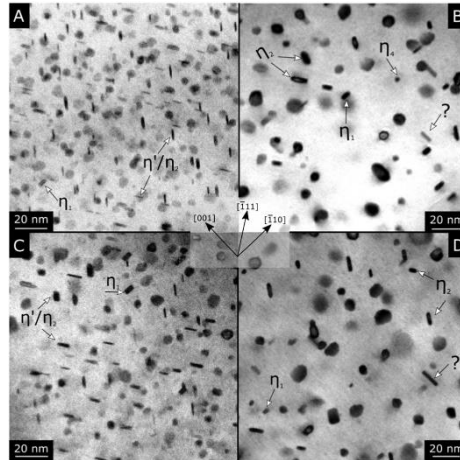
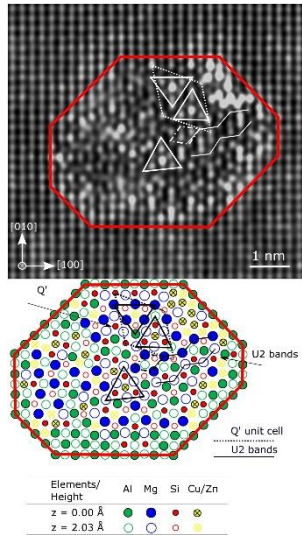


Figure 7. Edge features for different calcifications: red curves are associated with small MPs enclosed in a vesicle for the position indicated by the red square in the HAADF image A; black and blue curves correspond to large MPs made of calcium phosphate (respectively black and blue squares in B and C); purple curves correspond to a large MPs made of pure calcium carbonate (purple square in D). For comparison, the green curves correspond to organic material and resin (green square in A). Scale bar = 50 nm.

## Nanoscale study of kidney calcifications formation

- Core loss EELS analysis of mineral particles.

Gay et al, ACS Nano 2020, 14, 1823–1836



Precipitation in an extruded AA7003 aluminium alloy:

- Observations of 6xxx-type(Mg-Si) hardening phases in 7xxx (Mg-Zn) alloy.

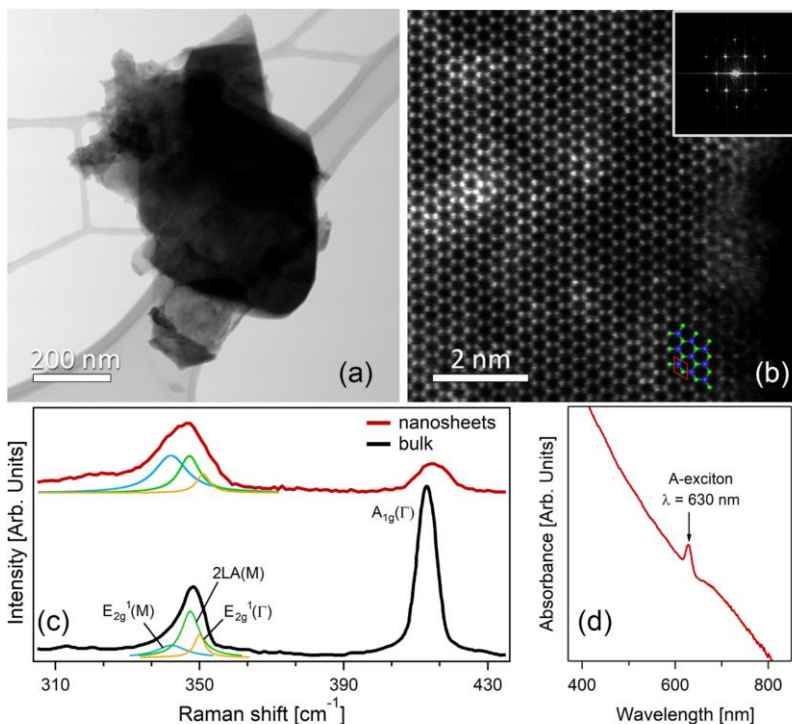
The evolution of precipitate crystal structures in an Al-Mg-Si(-Cu) alloy studied by a combined HAADF-STEM and SPED approach

- Mapping crystallography.





# Example of completed industry TA



- Liquid phase defoliation using a non-toxic and biodegradable solvent.
- TEM study complements other techniques.
- WS<sub>2</sub>, MoS<sub>2</sub> and graphene studied.
- Figure shows;
  - TEM image of overlapping WS<sub>2</sub> flakes
  - Atomic-scale aberration corrected STEM imaging (60 kV) of single layer flake (inset shows W and S positions, blue and green respectively).

Sustainable liquid-phase exfoliation of layered materials with non-toxic Polarclean solvent

Paolucci et. Al. Accepted for publication in ACS Sustainable Chemistry & Engineering  
<http://dx.doi.org/10.1021/acssuschemeng.0c04191>



# Other ESTEEM 3 opportunities



- ESTEEM3 also arranges training courses and workshops:
- TEM-UCA workshop on transmission electron microscopy of nanomaterials (Cadiz), 2021
- 6th Stanisław Gorczyca European School on electron microscopy and tomography (Krakow), 2021
- QEM: Review and New Advanced TEM techniques (Toulouse), 2021
- European workshop on quantitative STEM imaging (Ljubljana), 2022
- Sample preparation:
- Workshop on advanced TEM specimen preparation (Stuttgart), 2021



# Summary



- ESTEEM3 can provide Industrial TA access to leading European state-of-the-art electron microscopy research infrastructures.
- Free under the conditions defined in the application procedure.
- Potential industry applicants for TA are encouraged to make contact for further discussion.
  - Please find -  
[https://www.eesteem3.eu/lw\\_resource/datapool/systemfiles/cbox/551/live/lw\\_datei/comic-esteem3-en.pdf](https://www.eesteem3.eu/lw_resource/datapool/systemfiles/cbox/551/live/lw_datei/comic-esteem3-en.pdf)



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Thank You For  
Your Attention