Enabling Science Through European Electron Microscopy - ESTEEM: *A European Success Story*

FINAL EVENT

May 31st 2023

ESTEEM3 has received funding from the European Union's **Horizon 2020** research and innovation programme under grant agreement No 823717.

Agenda

09:00 – 09:25	**Welcome introduction on ESTEEM3 main achievements**, *Prof. Peter A. van Aken (MPG), coordinator of ESTEEM3*
09:25 – 10:55	**ESTEEM3 - Joint Research Activities achievements,** *Research Work packages leaders and participants*
10:55 – 11:10	Coffee break
11:10 – 11:30	**ESTEEM3- Networking Activities achievements**, *Prof. Miran Ceh (JSI)*
11:30 – 12:00	**ESTEEM3 -Transnational Access achievements**, *Prof. Peter A. van Aken* **TNA discussion round table:** TNA for industry in the context of EU grants chaired by Dr. John Walmsley (UCA) John Walmsley: He has a vast experience in the field of characterisation of (nano-) materials, particularly using electron microscopy. John is available for discussing external (both academic and non-academic) work and is actively engaged in collaborations with industrial partners. Dogan Ozkaya: External Advisory Board member of ESTEEM3, he has experience in electron microscopy, working in a Johnson Matthey ·company that makes commercial use of advanced TEM equipment and has insights through his interaction with ePSIC/Diamond in the UK.

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Agenda

12:00 – 13:30	Lunch break
13:30 – 15:30	**TA users feedbacks and discussions – 4 TA user members.** 13:30-13:50: PhD. Piotr Woźniak- HSMCO project from Institute of Low Temperature and Structure Research (Collaboration with UCA) *Nano-gold on hierarchically structured gadolinium-doped ceria as an active oxidation catalyst: synergistic effect of chemical composition and structural hierarchy* 13:50-14:10: PhD. Elisa Zanchi- CeramECS project from Politecnico di Torino (Collaboration with AGH) *Advanced microscopy characterization of innovative ceramic coatings for hydrogen technologies* 14:10-14:30: Dr. S.M. Collins -Lumi-Nano-LBP project from the University of Leeds (Collaboration with CNRS) *Hybrid composites for bright and stable light emission* 14:30-14:50: Dr. Sophia HOUARI - FLUOTOOTH and MIHTOOTH project from Paris Cité University and INSERM (collaboration with MPG) *Tooth enamel pathologies of environmental origin: from macro- to nano-scale* 14:50-15:10: Coffee break 15:10-15:30: PhD., Leander Michels - ISGI project from Elkem Silicon Products (industrial partner-collaboration with UCAM) *Electron microscopy characterization of graphite-microparticle interface.*
15:30 – 15:50	Routes of sustainability: eDREAM and ARIE, *Prof. Rafal Dunin-Borkowski (Forschungszentrum Jülich)*
15:50 – 16:00	Conclusion – *Prof. Peter A. van Aken*

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WELCOME INTRODUCTION
ON ESTEEM3
MAIN ACHIEVEMENTS

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

What is ESTEEM3 ?

ESTEEM3 is an integrated infrastructure network of **European Electron Microscopy Facilities** providing **Transnational Access (TA)** for the academic and industrial research community in materials, physical, chemical and life sciences to the most powerful electron microscopy instrumentation and techniques available at the nanoscale.

ESTEEM3

- Follow-on-project of **ESTEEM** and **ESTEEM2**
- Duration: *January **2019*** to *December **2022***
- Cost-neutral extension until *June **2023 (54 months)***

What are the aims of ESTEEM3 ?

The general aims of the consortium are to:

- **Establish a strategic leadership in electron microscopy** to guide future developments and promote advanced electron microscopy to the wider research community

- **Provide transnational access** for the academic and industrial research community in physical sciences to some of the most powerful characterisation techniques available at the nanoscale

- **Provide training in innovative methods in electron microscopy** and a forum for discussing emerging cutting-edge electron microscopy techniques

Who manages ESTEEM3 ?

- **The Coordination Team:**

Peter van Aken	Miran Čeh	Angus Kirkland	Aude Garsès
Coordinator	Deputy coordinator	Deputy coordinator	Project manager

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Jožef Stefan Institute

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euronovia

- **Tasks:**
  (i) Support the day-to-day project management
  (ii) Monitor all NA, JRA, TA activities
  (iii) Serve as a helpdesk for the project partners

Who are ESTEEM3 partners ?

The **consortium** includes a total of
20 project beneficiaries:

Beneficiary	City and country
1. Max Planck Institute MPG (Coordinator)	Stuttgart, Germany
2. Research Institute Jülich JUELICH	Jülich, Germany
3. French National Centre for Scientific Research CNRS	Paris, France
4. University of Antwerp UANTWERP	Antwerp, Belgium
5. University of Oxford UOXF	Oxford, United Kingdom
6. University of Cambridge UCAM	Cambridge, United Kingdom
7. Jozef Stefan Institute JSI	Ljubljana, Slovenia
8. Graz University of Technology TUGraz	Graz, Austria
9. University of Zaragoza UNIZAR	Zaragoza, Spain
10. University of Cadiz UCA	Cadiz, Spain

Who are ESTEEM3 partners ?

The **consortium** includes a total of
20 project beneficiaries:

Beneficiary	City and country
11. University of Krakow AGH-UST	Krakow, Poland
12. Chalmers University of Technology CHALMERS	Gothenburg, Sweden
13. Norwegian University of Science and Technology NTNU	Trondheim, Norway
14. National Research Centre CAT	Rome, Italy
15. Attolight SA ATTO	Lausanne, Switzerland
16. Corrected Electron Optical Systems CEOS	Heidelberg, Germany
17. DENSsolutions BV DENS	Delft, Netherlands
18. NanoMEGAS SPRL NM	Brussels, Belgium
19. Quantium Detectors Limited QD	Harwell, United Kingdom
20. Euronovia	Paris, France

ESTEEM3 – JRA

ESTEEM3 member laboratories and SMEs develop
Joint Research Activities (JRA):

- Development of advanced TEM techniques for the solution of key problems:
  - Imaging, Diffraction and Metrology
  - Spectroscopy
  - In-situ TEM
- Electron microscopy for specific applications
  - Materials for ICT, energy, health and transport
- Automation and big data

Targets of ESTEEM3: JRA

Category	Target
Open software	15
Technical reports (JRA deliverables)	33

The links to the open software and technical reports can be downloaded from the **ESTEEM3 website:**

https://www.esteem3.eu/

ESTEEM3 – NA

Networking Activities (NA): ESTEEM3 implements a wide variety of activities aiming at improving the dissemination of outputs:

- **Open-Access publications** in high-ranked journals
- Lectures at **selected conferences**
- Setting up of an **educational hub**
- Organisation of a **series of schools, workshops and webinars** offering training by specialists in advanced techniques
- Collaboration with other **European projects** and **infrastructures**
- Promotion on **social media** and **ESTEEM3 website**

https://www.esteem3.eu/

Targets of ESTEEM3: NA

Category	Target
ESTEEM staff trained through staff exchange	50
Schools and workshops	11
Participants at schools/workshops	600
Other educational and training events including presentations to industrial users	25

Outreach – Main Achievements

- Production of **bi-annual newsletters**
  - **7** editions so far: Sept 2019, April 2020, Oct 2020, June 2021, Nov 2021, June 2022, Dec 2022
  - Last newsletter in June 2023
  - **561 contacts**

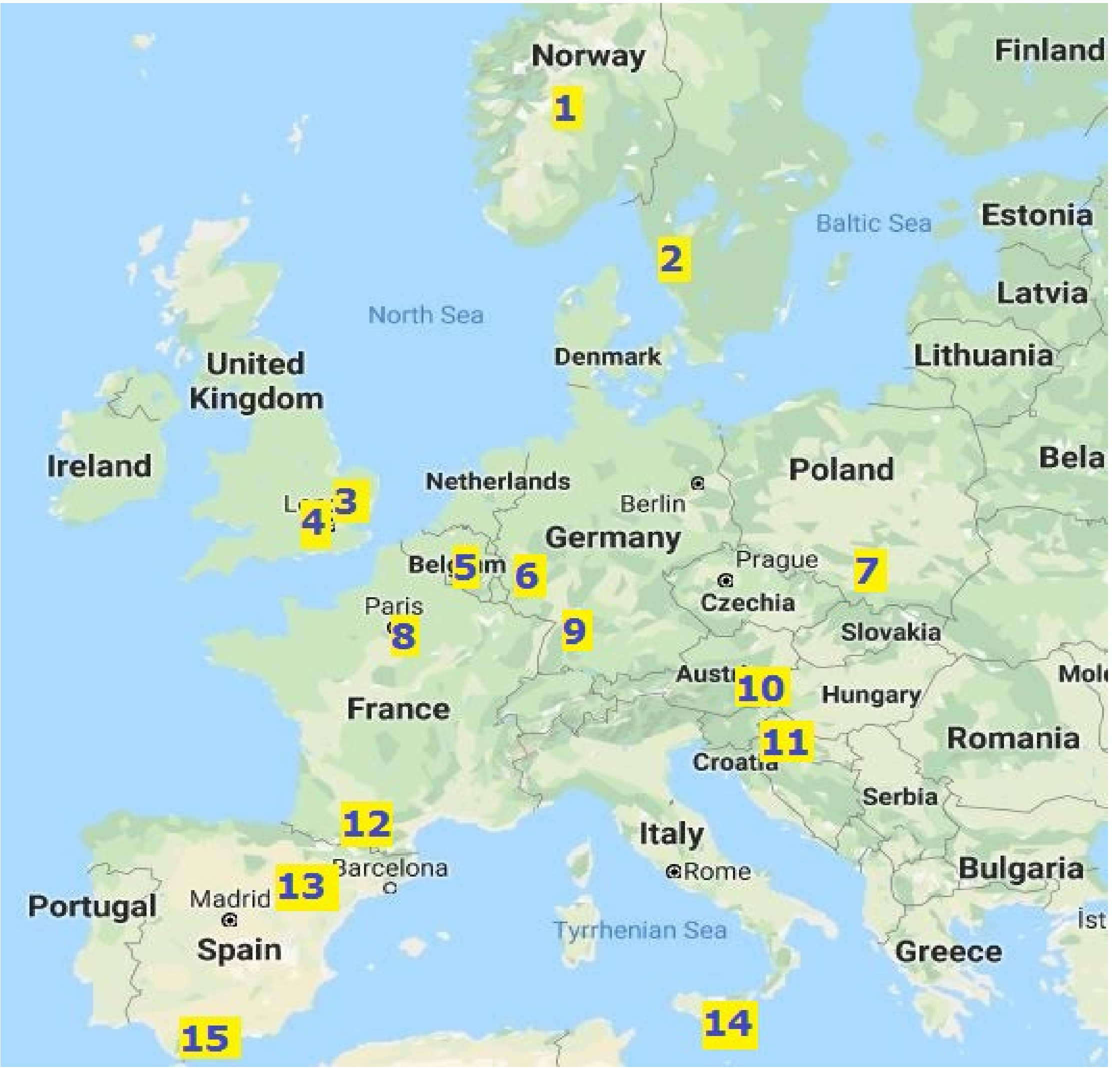
- Promotion on **social media**
  - **494 members on LinkedIn**
  - **524 followers on Twitter**

- **Collaboration** with other EU projects
  - **EUSMI** – European Soft Matter Infrastructure
  - **Q-SORT** – The Quantum Sorter
  - **TEESMAT** – The Open Innovation Test bed for Electrochemical Energy Storage MATerials

ESTEEM3 – TA

- **Provide Transnational Access (TA)** for the academic and industrial research communities in material, physical, chemical and life sciences to the most powerful electron microscopy instrumentation and characterisation techniques available at the nanoscale and beyond

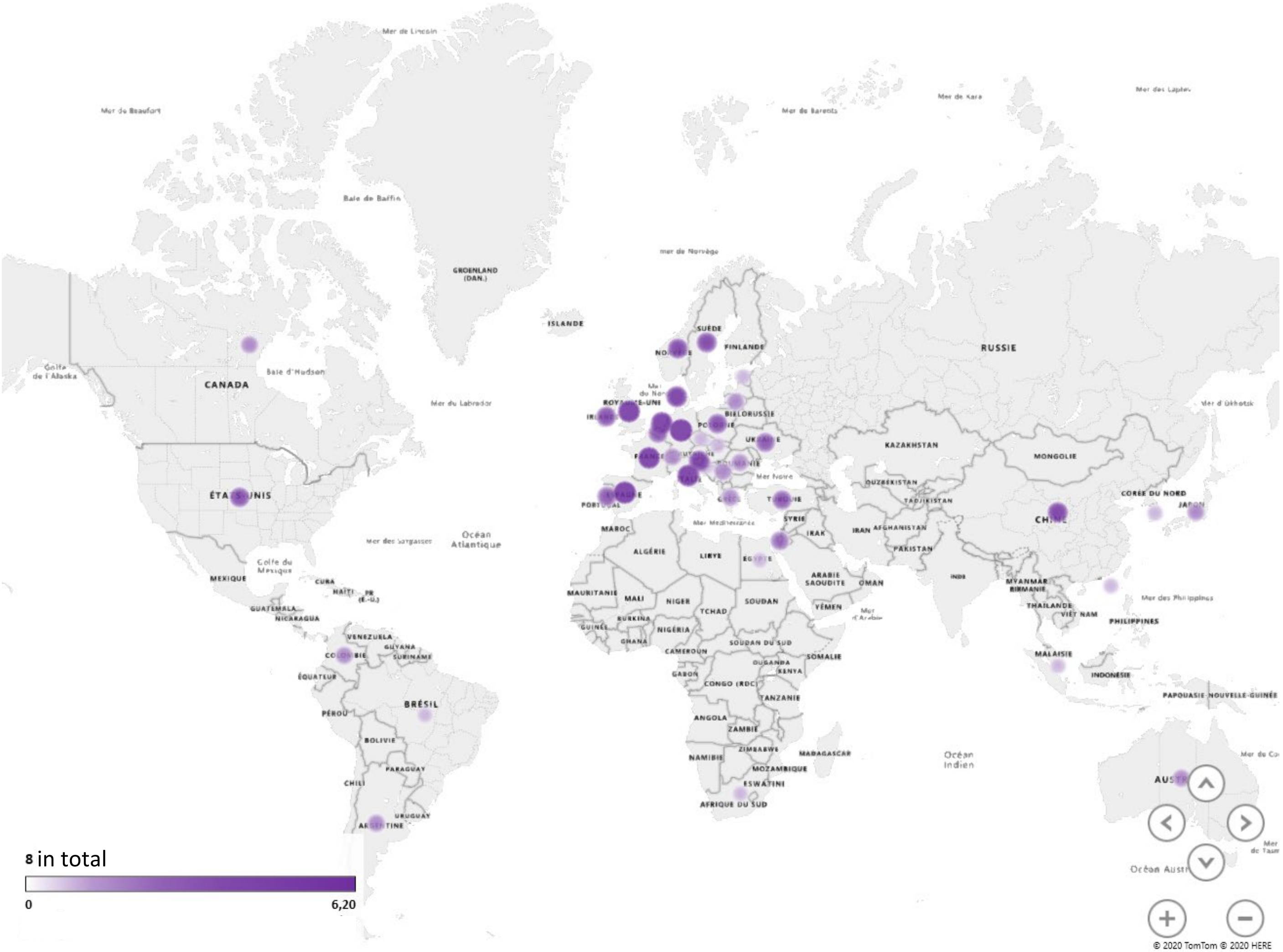
Who provides TA in Europe ?



ESTEEM3–TA laboratories

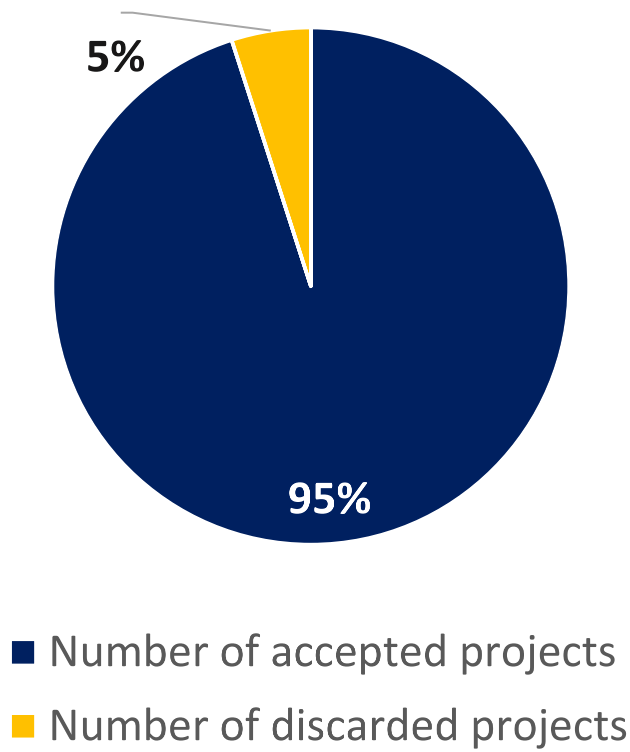
1. Gemini Trondheim
2. CMAL Gothenburg
3. WEMS Cambridge
4. OXTEM Oxford
5. EMAT Antwerp
6. ER-C Juelich
7. IC-EM Krakow
8. LPS Orsay
9. StEM Stuttgart
10. FELMI-ZFE Graz
11. JSI Ljubljana
12. CEMES Toulouse
13. LMA Zaragoza
14. Beyondnano Catania
15. A-DME Cadiz

A wide range of TA users' countries

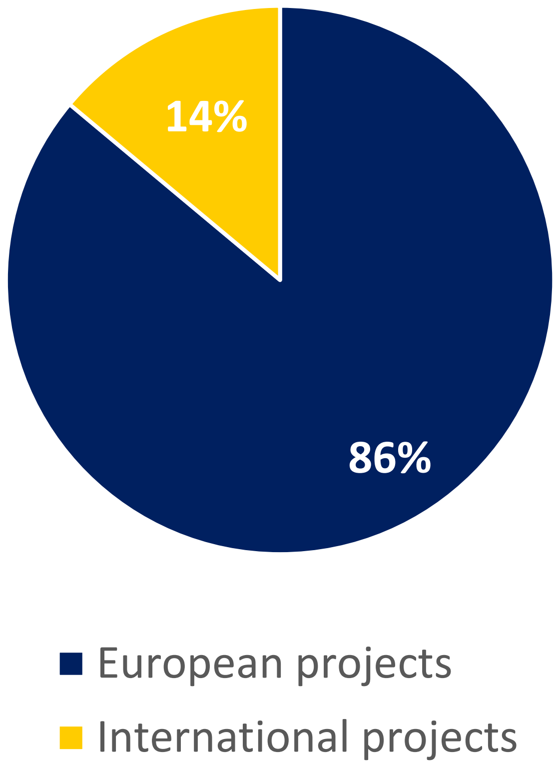


Results in M52 – April 2023

Success Rate



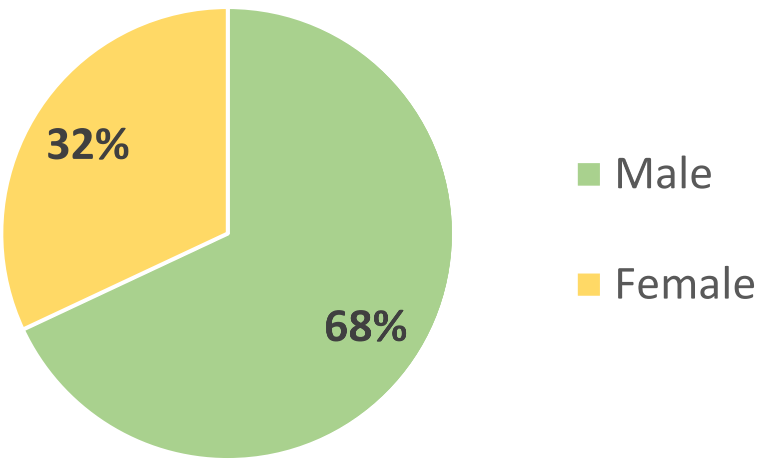
Country of Origin



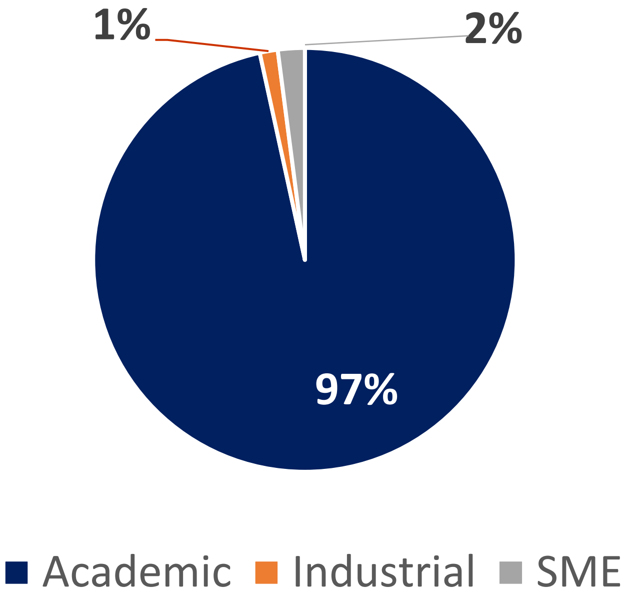
Approx. **500** accepted TA projects

Results in M52 – April 2023

Gender Balance



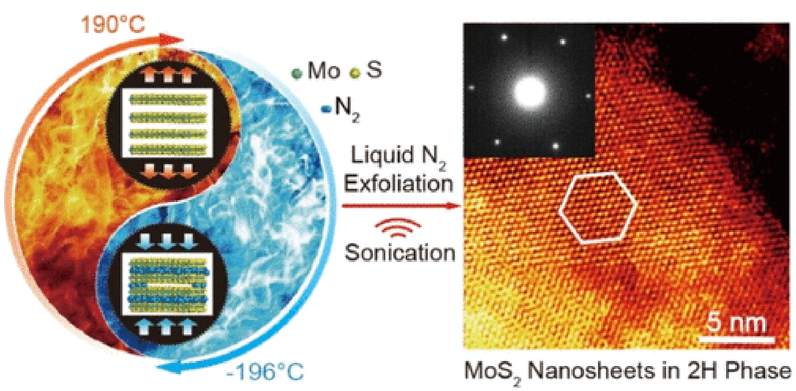
Sector



Targets of ESTEEM3: TA

Category	Target
Access days to TEM installations	2200
Prepared samples for TEM	780
Access days for data processing	2950
Number of European TA projects	450
Number of international TA projects	50

TA success stories: *2D Nanosheets*



2D Nanosheets project of Prof. Runfeng Chen from the **Nanjing University of Posts and Telecommunications, China**.

TA to **Stuttgart Center for Electron Microscopy** (StEM) in Germany

Liquid-phase exfoliation of 2H-MoS_2 ultra-thin nanosheets using liquid nitrogen, published in journal **ACS Sustainable Chemistry & Engineering**.

TA success stories : *NanoSensor*

The ***NanoSensor*** project by Dr. David Maria Tobaldi from the **University of Aveiro**

TA to Advanced Division of Electron Microscopy (DME-UCA) facility of the **University of Cadiz**

Project on understanding the performance of electro-chemical non-enzymatic glucose sensors by nanoscale analyses

TA success stories : *ECO2D*

Industry TA project: ***ECO2D*** by Patrice Le Cornec from **Solvay**, France

TA to **Beyondnano** in Catania, Italy

Project on systematic structural and chemical characterization at atomic resolution of high-quality exfoliated 2D materials without any solvent residual using eco-friendly solvent PolarClean of Solvay

TA success stories: *Superconducting Pb on semiconducting InAs nanowires*

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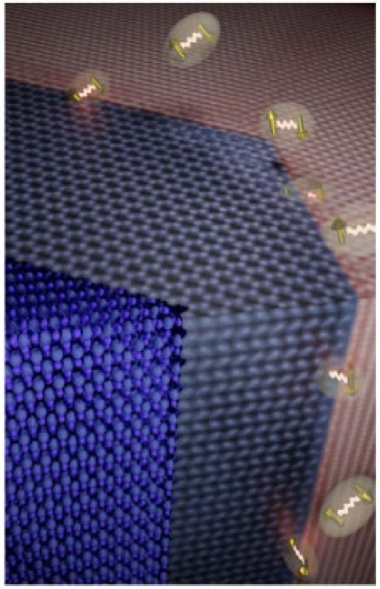

The ***Superconducting Pb on semiconducting InAs nanowires*** project by Dr. Thomas Kanne from the **University of Copenhagen**, Denmark

TA to **Chalmers University of Technology** in Gothenburg, Sweden

Project on epitaxial semiconductor/super-conductor heterostructures with novel quantum states. Depending on interface integrity, these materials act as a hybrid exhibiting engineered physical properties.

ESTEEM3
JOINT RESEARCH ACTIVITIES
ACHIEVEMENTS

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Workpackage 4
Imaging, Diffraction and Metrology

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Overall Summary WP4

Imaging Diffraction and Metrology

- Improving reproducibility of TEM experiments by novel calibration procedures
- Exploring electron beam shaping for improved measurement precision
- Quantifying measurement precision from counting statistics and the optical state of the EM to guide experiments
- Controlling the electron dose

Deliverables

D4.1: Report on comparison of approaches to electron ptychography (M16 – OXF)
D4.2: Report on diffraction methods (M34 – CAM)
D4.3: Report on comparison of precision versus dose for imaging and diffraction methods (M46 – OXF)
D4.4: Report on improved calibration routines (M24 – ANT)
D4.5: Report on evaluation of beam shaping for improved depth of field (M34 – ANT)
D4.6: Report on evaluation of precision estimates for TEM experiments (M46 – TOU)

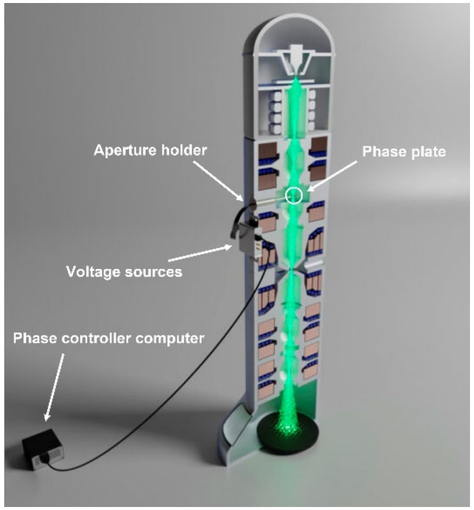
Highlights WP4

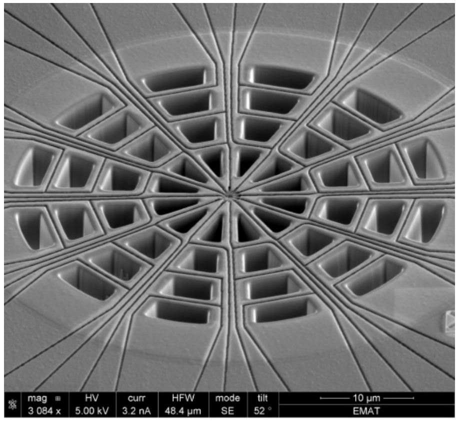
- Unlimited speed 4D STEM with event based recording: get ALL scattering data at normal imaging scan speeds -> needs to move to commercial phase
- Adaptive optics successfully demonstrated -> spinoff AdaptEM realized
- Real time ptychography and ICOM imaging demonstrated: high contrast at ultra low dose imaging. Transparent for user. -> open source, commercial implementation possible.
- Neural networks for denoising and preprocessing of Ptychographic data
- Ptychographic reconstructions of biological Cryo EM samples

Highlights WP4

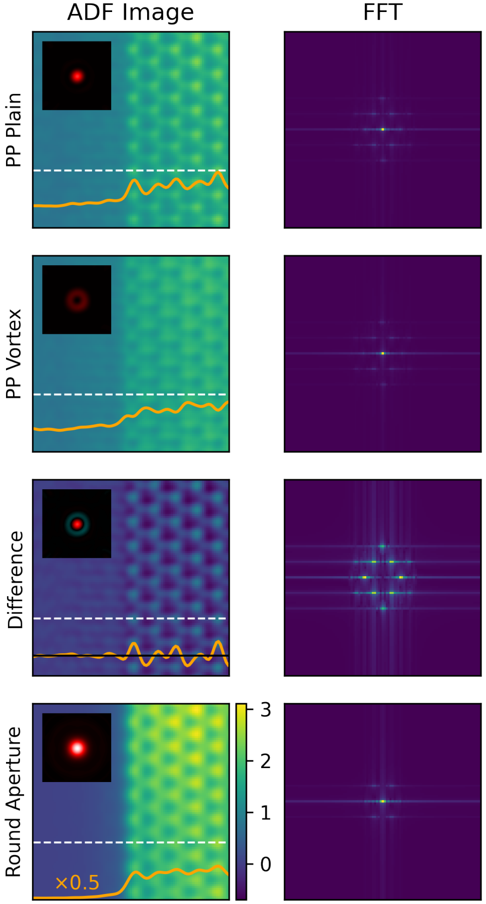
- Automated orientation mapping (Pyxem)
- Automated STEM diffraction recording (SED)
- Applications of SED
  - Applications to soft materials, strain mapping, diffraction tomography.
  - Sensitive materials, e.g. Nanoscale phase mapping in Halide Perovskites
- Theoretical Framework for comparing 4D STEM transfer functions
- Alternative scan strategies lower beam damage at same dose
- Combining reconstructed phase with ADF atom counting for local chemical variations in oxide nanoparticles

Adaptive optics with electrons

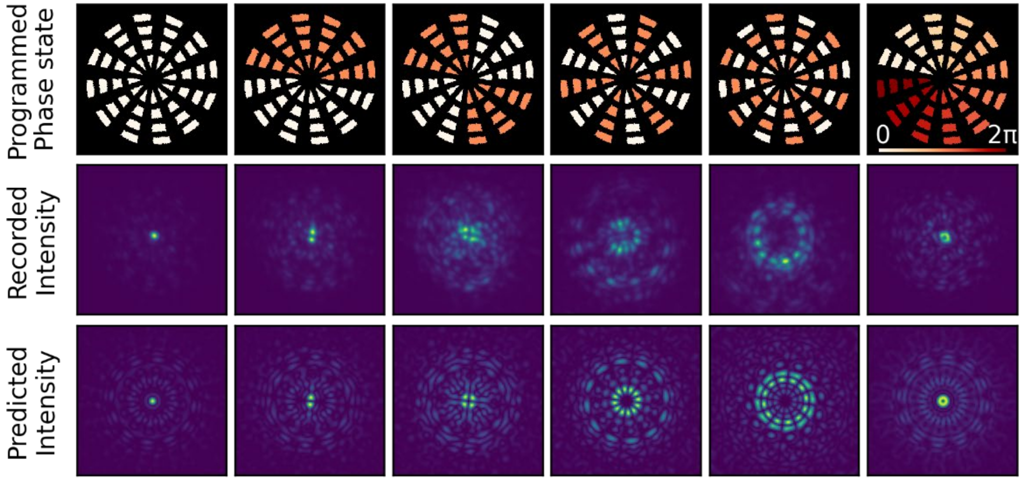




differential imaging for fast and compact aberration correction



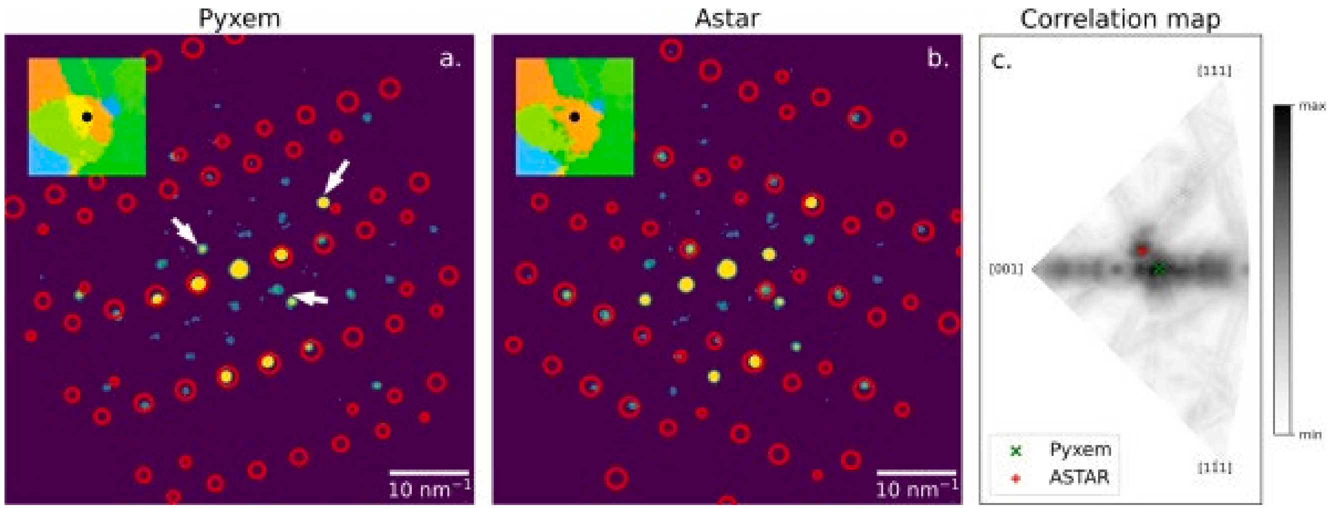
phase plate brings zoo of exotic electron wave functions

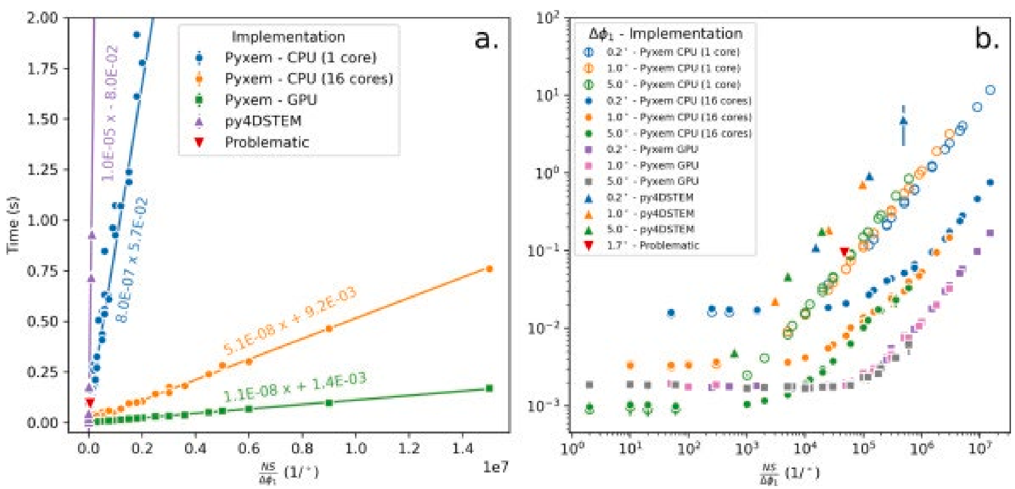


Quantum Wavefront Shaping with a 48-element Programmable Phase Plate for Electrons
Chu-Ping Yu, Francisco Vega Ib́ ã nez, Armand B́ ech́ e, Jo Verbeeck PRX 2023

AdaptEM

Fast indexing diffraction pattern





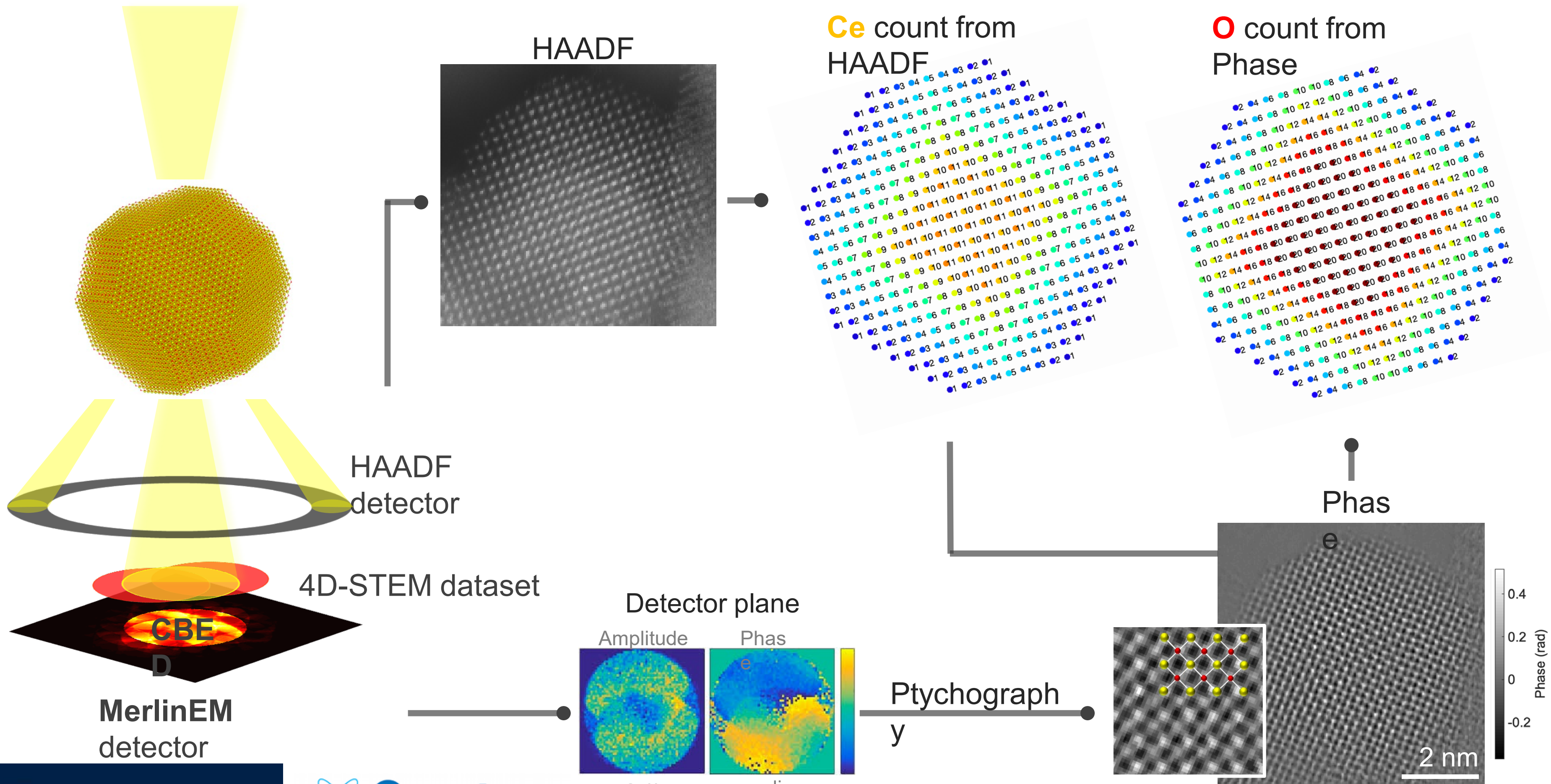
fast data-aquisition also calls for fast and robust data interpretation noone can filter through TBs of data by hand anymore

NanoMEGAS
Advanced Tools for electron diffraction

UNIVERSITY OF CAMBRIDGE

Niels Cautaerts, Phillip Crout, Håkon W. Ånes, Eric Prestat, Jiwon Jeong, Gerhard Dehm, Christian H. Liebscher, Free, flexible and fast: Orientation mapping using the multi-core and GPU-accelerated template matching capabilities in the Python-based open source 4D-STEM analysis toolbox Pyxem, Ultramicroscopy, Volume 237, 2022

Heavy + light elements in CeO2



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Quantum DETECTORS

WP4 Conclusion

- made more of **every** electron
- opens areas that were previously difficult:
  - beam sensitive samples
  - heterogeneous samples
  - faster results
  - reproducibility revolution through automation

- method development with real potential for mature product to serve ‘customers’ in Europe and beyond

Workpackage 5

New spectroscopies

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Key information

Work package number	**5**			**Lead beneficiary**					**ORS, ANT**			
Work package title	Spectroscopy											
Participants number	N°	1	2	3a	3b	4	6	8	9	11	12	14	15
Short name of participants		STU	JUL	TOU	ORS	ANT	CAM	GRA	ZAR	UCA	CHA	CAT	ATTO
Person/months per participant:	PM	6	*	*	36	9	*	12	2	12	*	*	2
Start month	1			**End month**					48			

Tasks & deliverables

- **Task 5.1**: *3D Dynamical and Phase sensitive Nanometer Measurement of Optical Properties* (ORS, ATTO, STU, GRA, ANT, TOU, CHA)

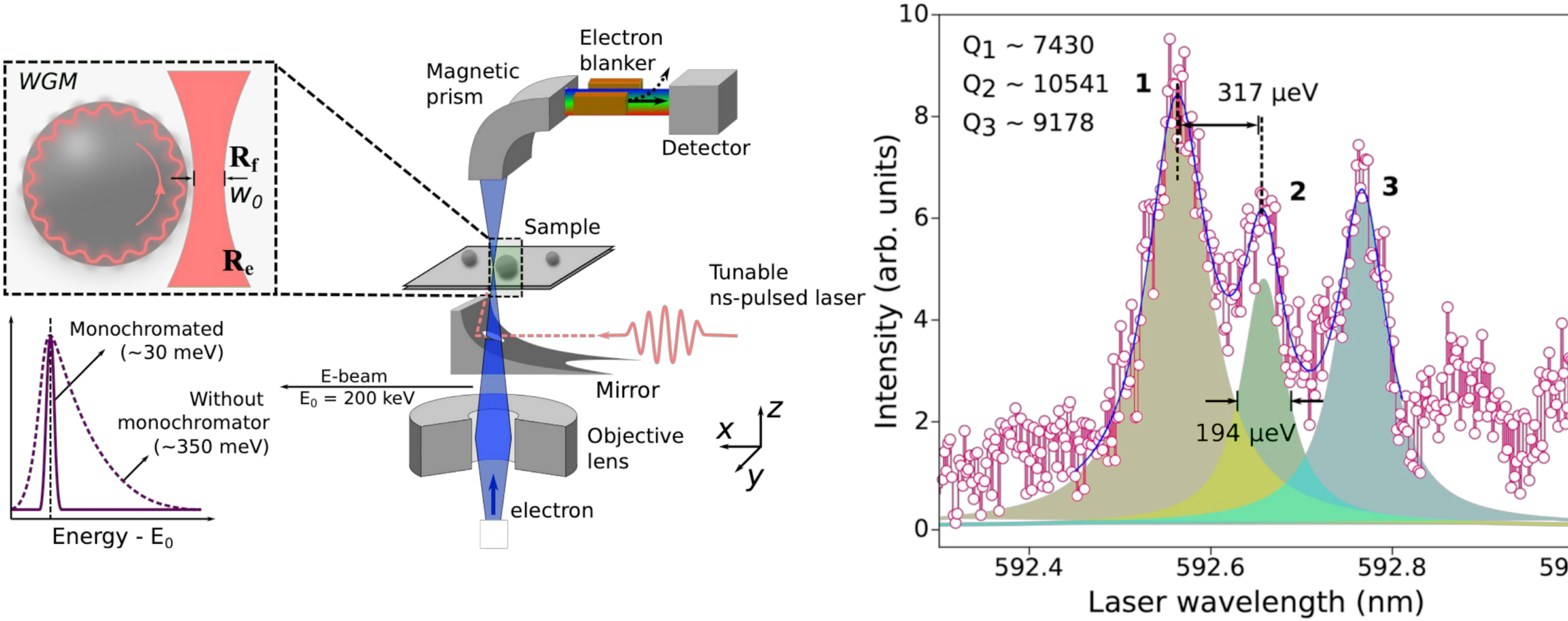
- **Task 5.2**: *Phonon and Vibrational Spectroscopies* (ORS, CHA, GRA, CAT, CAM)

- **Task 5.3**: *Increasing Accuracy and Reproducibility Through Correlation and Coincidence Experiments* (GRA, CAD, ORS, ATTO, ANT, STU, ZAR, JUL)

Deliverables

D5.1: Report on ps-resolved STEM-CL experiments (M30 – ORS)
D5.2: Report on plasmon and phonon tomography (M40 – ORS)
D5.3: Report on quantitative chemical atomic resolution mapping (M52 – GRA) *writing*
D5.4: Report on correlation spectral imaging (M52 – ORS)

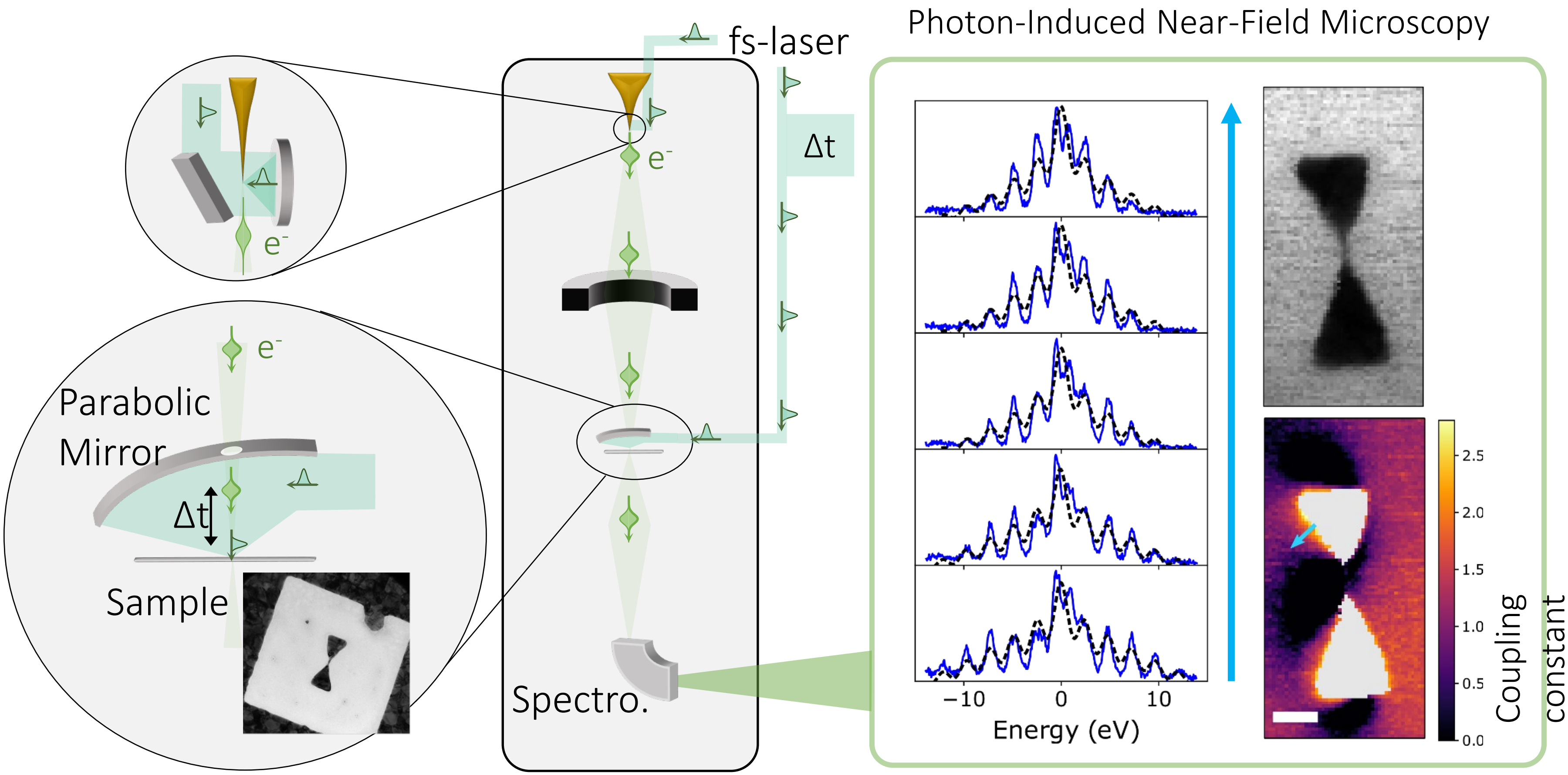
5.1 EEGS



Y. Auad et al., *μeV electron spectromicroscopy using free-space light*
Nature Communications *in press* (2023)

Extreme spectral resolution

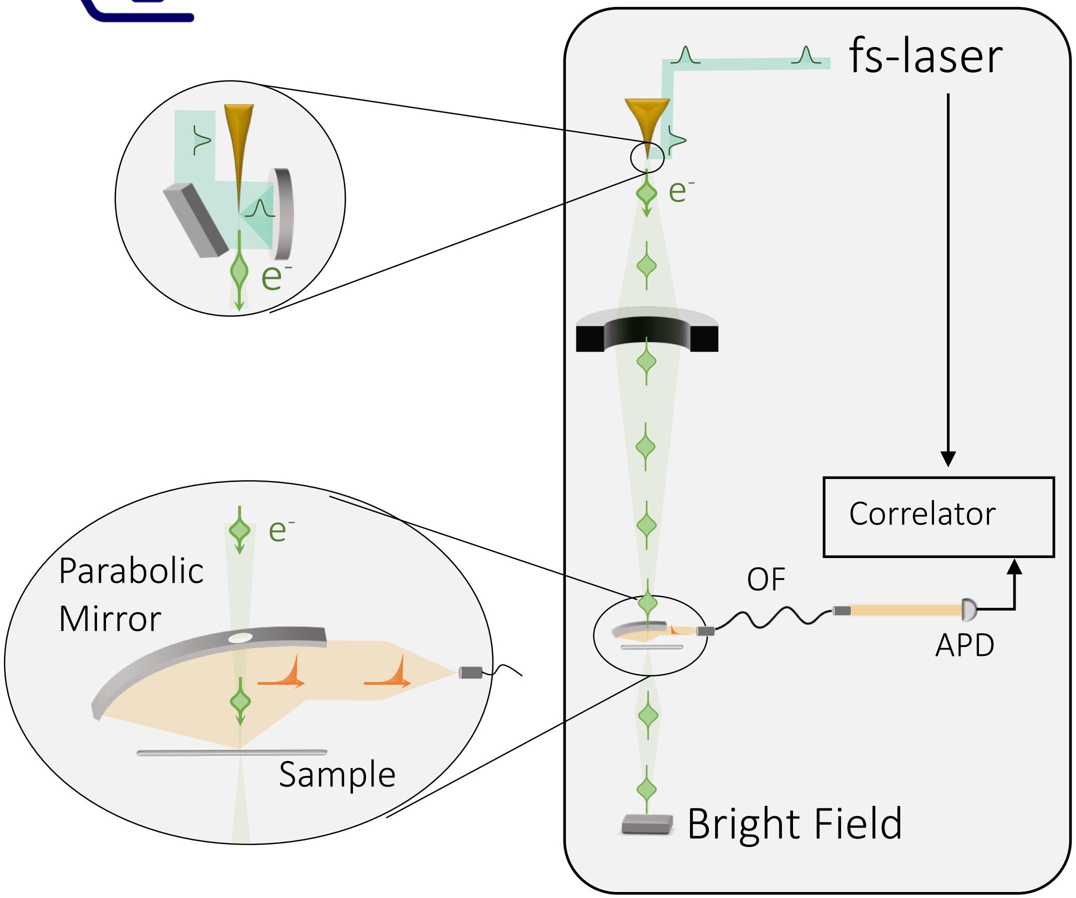
5.1 PINEM

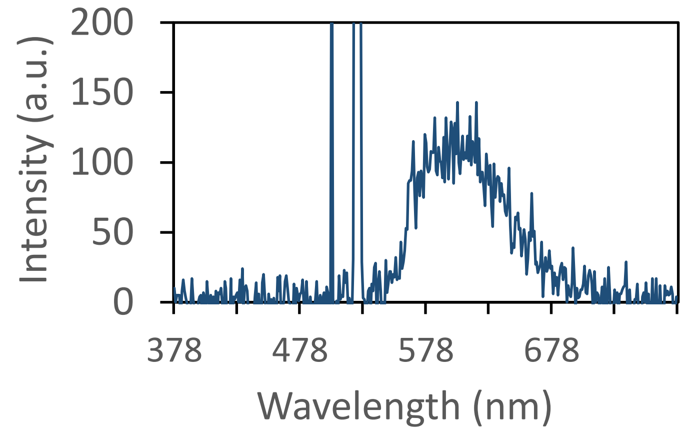


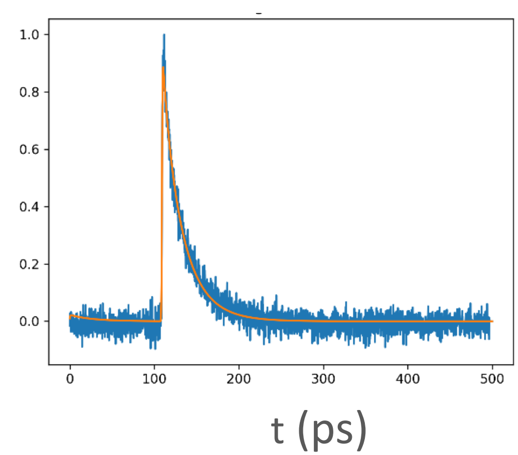
S. Meuret et al., *Photon induced near-field electron microscopy from nanostructured metallic films and membranes*
arXiv:2303.11195 (2023)

Interferences effects

5.1 TR-CL





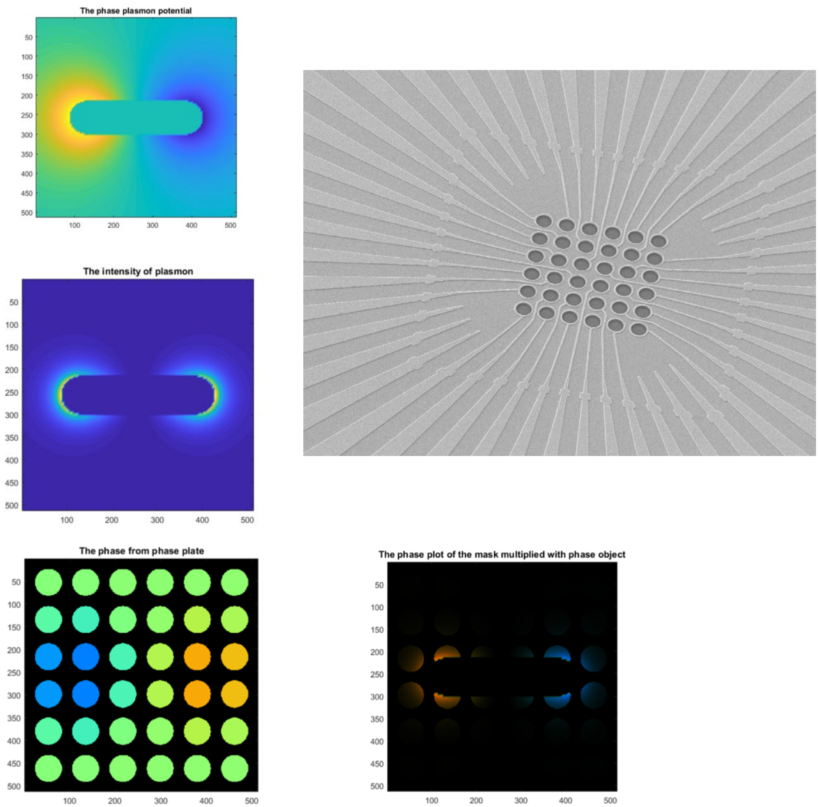


First TR-STEM-CL

Meuret, S. et al. *Time-resolved cathodoluminescence in an ultrafast transmission electron microscope.* **Appl. Phys. Lett**. 119, (2021).

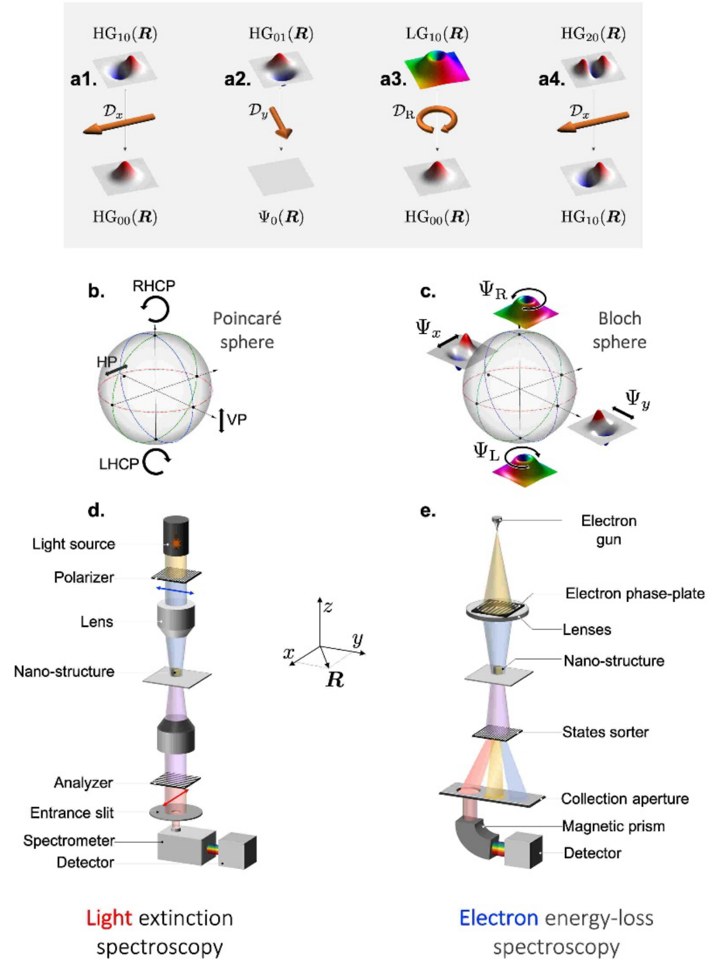
5.1 Phase shaping

Using a 6x6 programmable phase plate reveals 'phase' of plasmons (ANT)



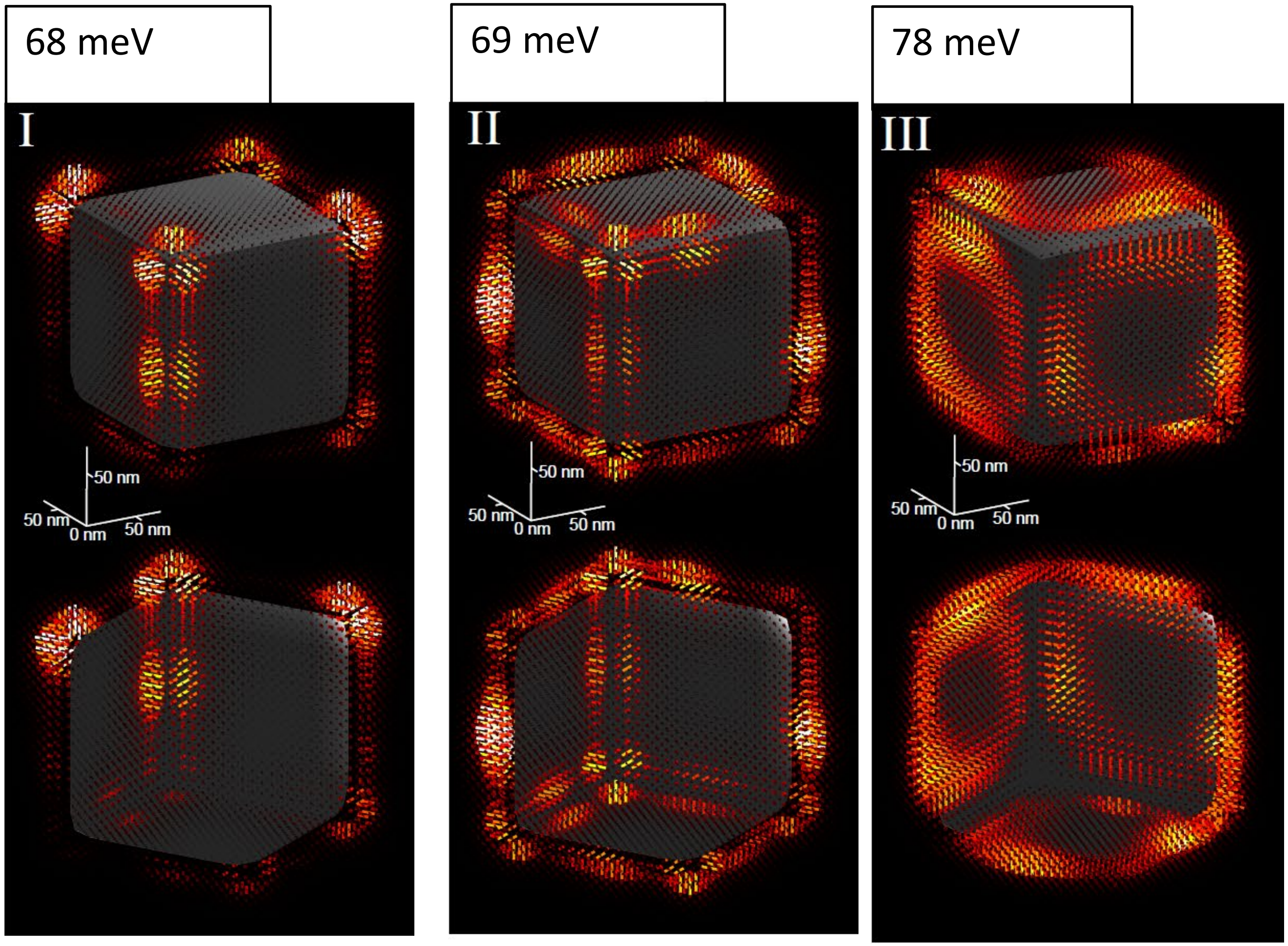
https://adaptem.eu

Control and understanding of phase shaping



Lourenço-Martins, M. et al. *Optical polarization analogue in free electron beams*. **Nat. Phys.** 17, 598–603 (2021).

5.2 Phonons

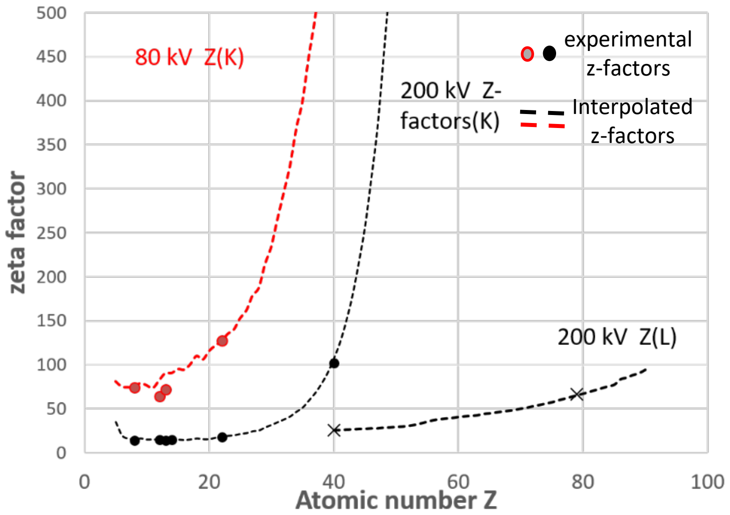


Li, X. et al. *Three-dimensional vectorial imaging of surface phonon polaritons*.
Science (80), 371, 1364–1367 (2021).

3D & vectorial mapping of SPP

5.3 Quantitative EDX

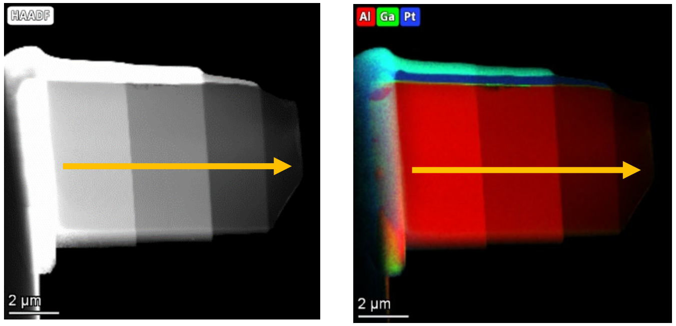
Z-factors for K and L lines at 200 and 80 kV for four-quadrant detector

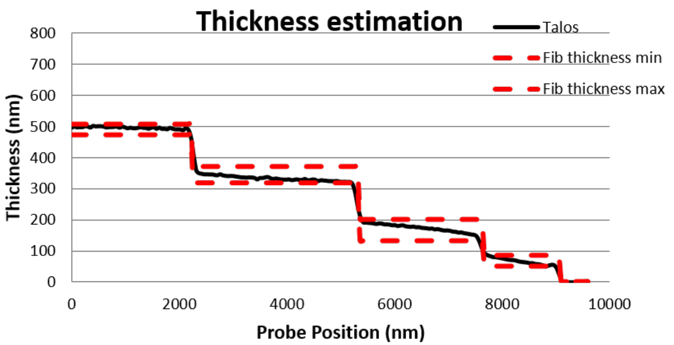


- Commercial oxides have been used as standards (MgO, TiO_2, SiO_2, Al_2O_3 and ZrO_2)
- Solid angle and efficency of the EDX detector have been considered.
- Z-factors for each segment have been also estimated.

Precise and accurate elemental quantification using 4 quadrant X-EDS detector

Thickness measurement of a pure Al lamella by using z-factors

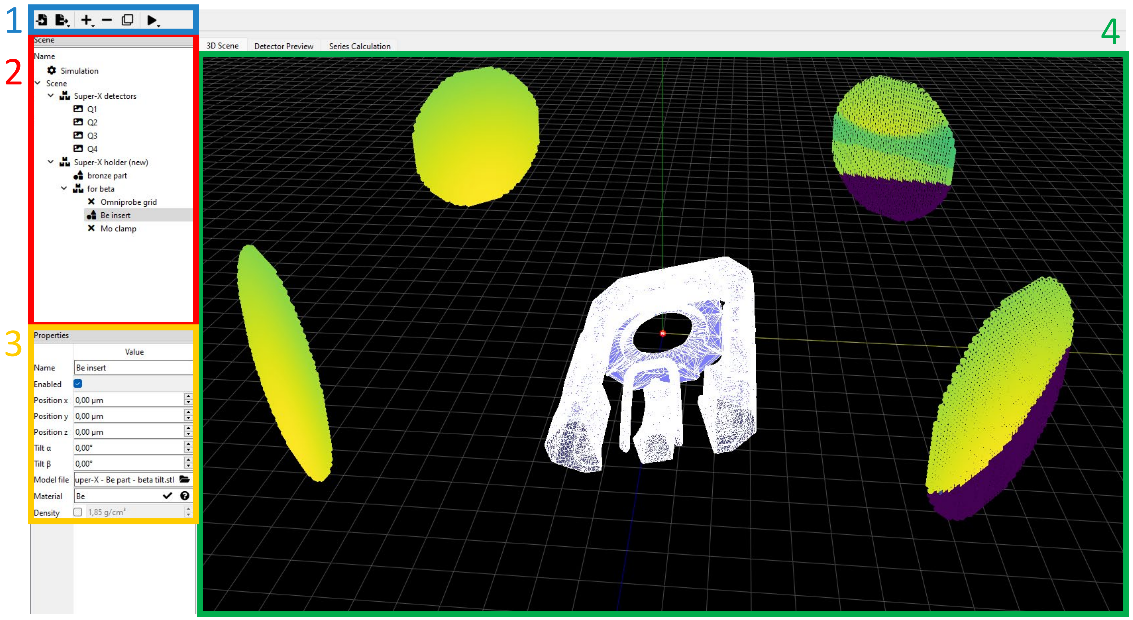




The thickness estimated (solid black line) is in a good agreement with that estimated by analysing the imaged acquiered on the FIB (dash red lines) profile view.

Miguel López-Haro et al., *Quantitative, Spectro-kinetic Analysis of Oxygen in Electron-Beam Sensitive, Multimetallic Oxide Nanostructures,* **Microscopy and Microanalysis**, ozad037 (2023)

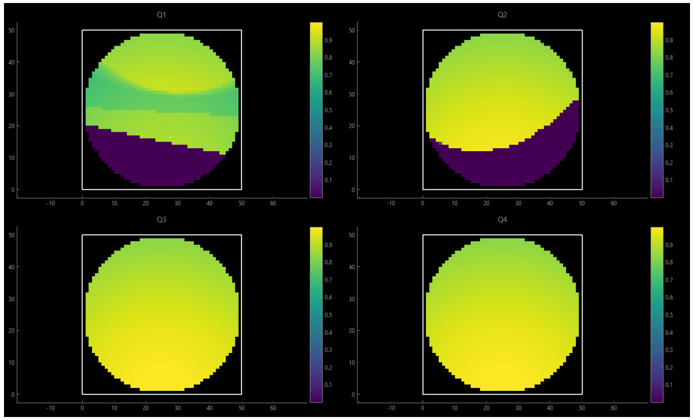
5.3 Quantitative EDX



Super-X 4 detector configuration with dedicated holder (tilt: α = 15°, β = 10°)

Basic GUI functions:

1. buttons to save/load configuration, add/remove items to configuration and start ray tracer
2. list of all items (parts of holder) and detectors
3. properties, selection of materials and geometry of selected item in (2)
4. preview of entire configuration in 3D

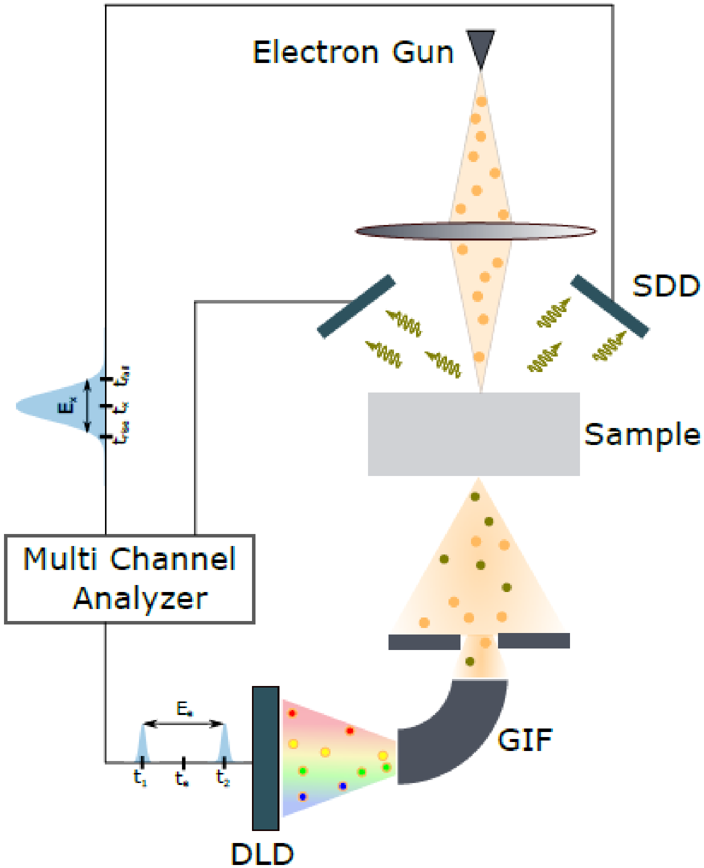


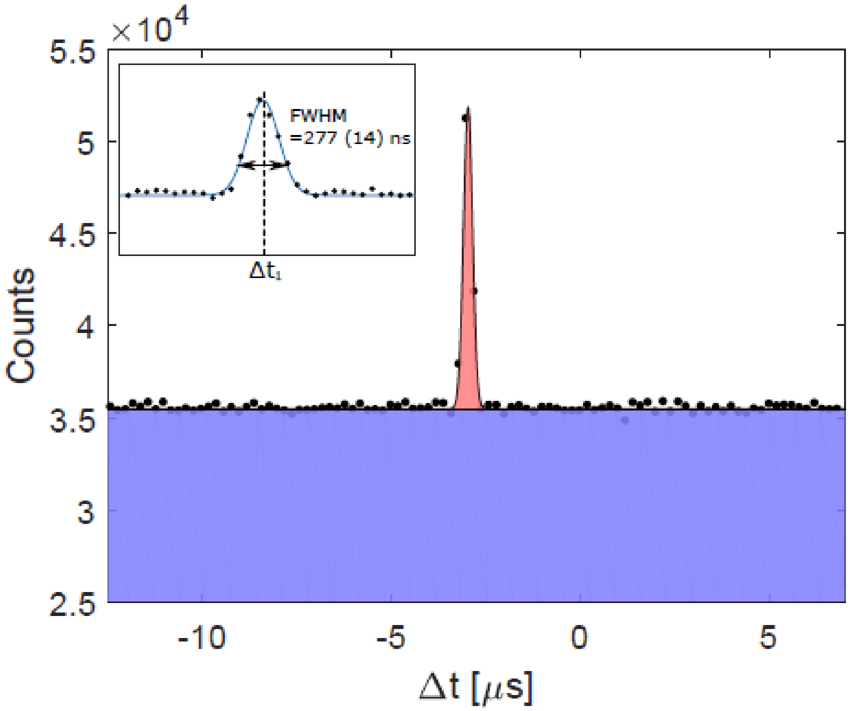
calculated EDX shadowing for each detector

Development of a raytracing software tool written in Python for quantitative EDX analysis. Using a sample holder with parts consisting of different materials in a certain configuration correction files can be generated, which adjust the X-ray intensities in order to yield correct quantitative results.

N. Grogger, J. Lammer, D. Knez, G. Haberfehlner, W. Grogger; paper in preparation

5.3 Coincident experiments (EELS/EDX)

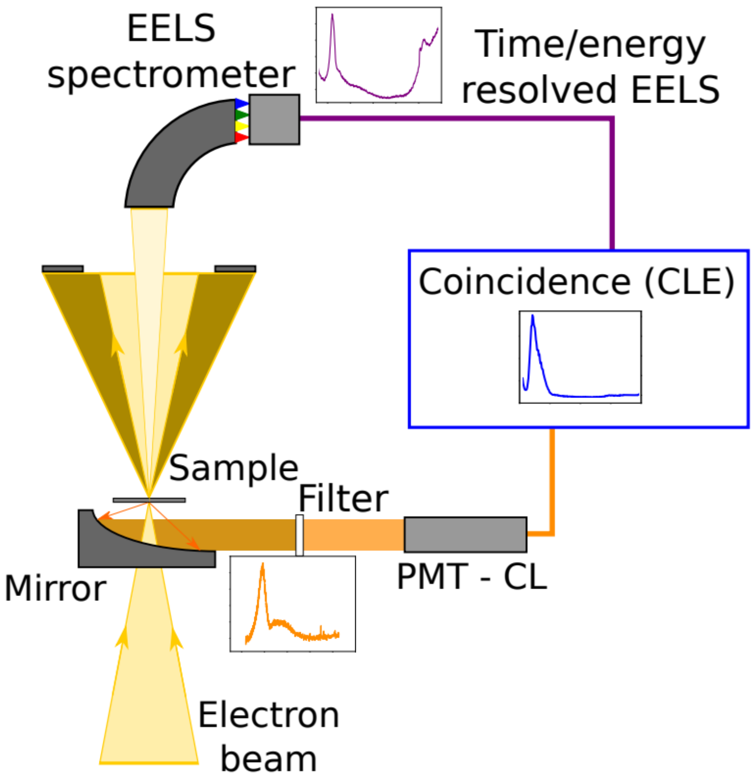


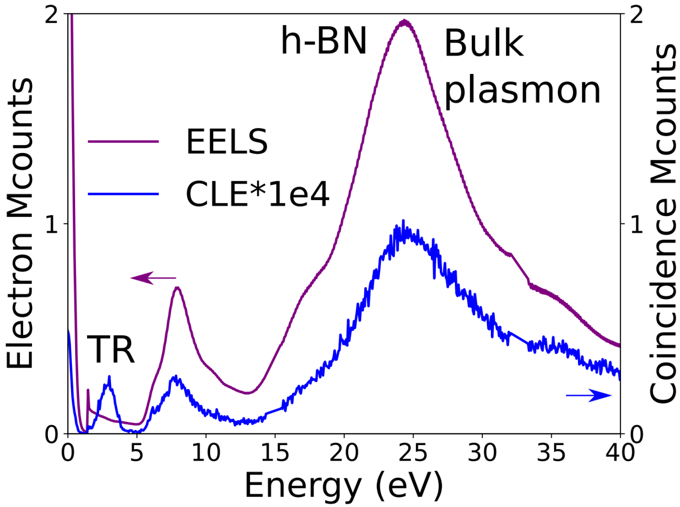


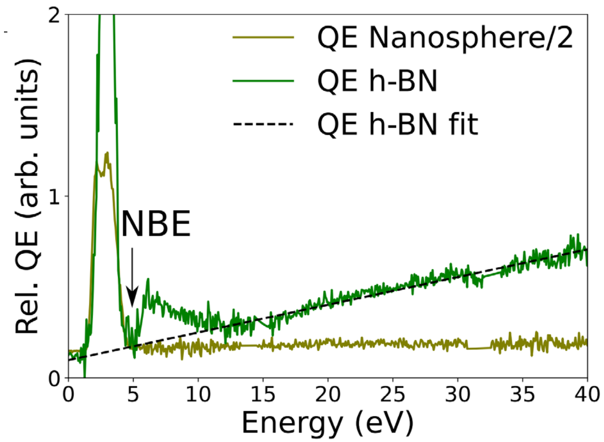
Jannis et al., *Coincidence Detection of EELS and EDX Spectral Events in the Electron Microscope* **Appl. Sci.** 11(19), 9058 2021

Removal of background and better access trace elements

5.3 Coincident experiments (EELS/CL)







Access to the excitation fate from absorption to emission

Varkentina, N., Auad, Y. et al. *Cathodoluminescence excitation spectroscopy: Nanoscale imaging of excitation pathways.* **Sci. Adv.** 8, (2022).

Workpackage 6
in situ TEM

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

Objectives

➔ Developing consistent **advanced *in-situ* experiments** to obtain **precise** and **quantitative information** on the modification of (nano)materials under **various types of external stimuli** (strain, electrical, magnetic, optical) and/or in **different environments**.

Tasks and deliverables

- **Task 6.1**: Sample preparation and in-situ TEM methodologies - DENS, ZAR, LJU, CAT, TOU, ORS, UA
  - *6.1.1:* Robust protocol to extract the TEM lamellae from a chip, welded to the stimuli platform, thinned down to electron transparency
  - *6.1.2:* Explore the development of MEMS technologies to perform *in-situ* biasing, straining or/cooling, heating TEM experiments
  - *6.1.3:* Procedures for preparing and transferring TEM samples in neutral atmosphere to perform in-situ experiment under gas environment

  ✓ **D6.1 – Report on protocol for advanced sample preparation of devices for in-situ experiment**

- **Task 6.2**: In-situ experiments in spectroscopy modes - ZAR, ORS, TOU, GRA, UA
  - Coupling simultaneously *in-situ* experiments and spectroscopic measurements, or independently

  ✓ **D6.2 – Report of protocol on *in-situ* EDS and EELS techniques**

Tasks and deliverables

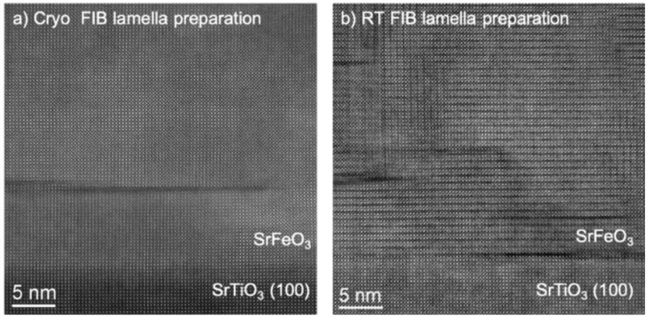
- **Task 6.3**: TEM in liquid and gas environment - LJU, DENS
  - Developing operando experiments in the TEM with dedicated TEM holders that can maintain the liquid or gases in a closed environment allowing imaging, diffraction and spectroscopy of samples in given media without altering the vacuum of the microscope
  - ✓ **D6.3 – Report on the effect of radiolysis in liquid cell**

- **Task 6.4**: In-situ biasing – TOU, ZAR, DENS, CAT, UA
  - Developing *in situ* TEM experiments applying AC or DC current or voltage to analyze the failure mechanisms in various materials and nanodevices
  - ✓ **D6.4 – Report on protocol for achieving low resistive FE(I)BID Ohmic contacts**

WP6 highlights ZAR

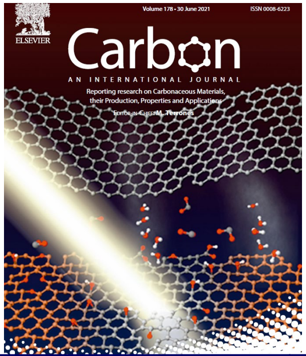
Task 6.1 - Sample preparation

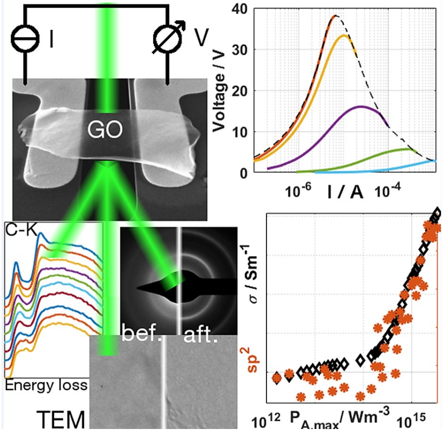
- Advanced sample preparation in cryogenic conditions for in-situ experiments.
  ➔ *Cryo-FIB preparation of metastable pervoskite oxides ($SrFeO_3$) for resistive switching experiments.*



Task 6.2 - In-situ experiments in spectroscopy modes

- EEL spectroscopic analysis of in situ thermal reduction of Graphene Oxide.



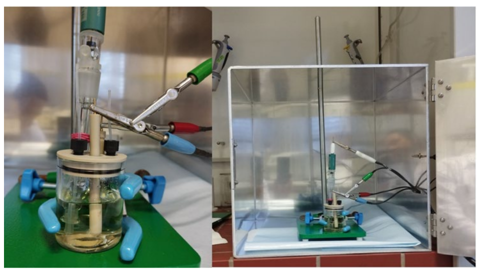


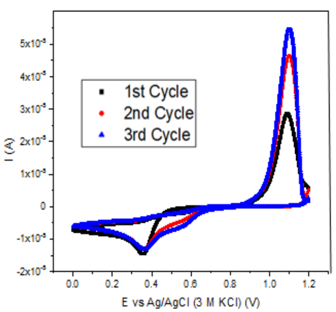
- *M. Pelaez-Fernandez et al. Carbon 178, 477 (2021)*
- *S. Hettler et al. 2D Materials 8, 031001 (2021)*

WP6 highlights LJU

Task 6.3 - TEM in liquid environment

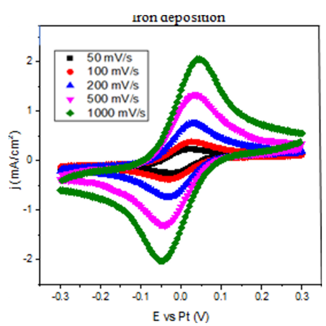
Step 1: *Ex-situ* electrochemical characterization using standard electrochemical cell





- Voltammogram for [HAuCl4]= 1 mM
- Supporting Electrolyte [KCl] = 0.1M;
- Working Electrode : Glassy Carbon
- Reference Electrode : Ag/AgCl 3M KCl
- Counter Electrode : Pt

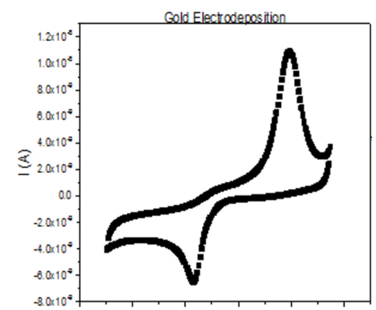
Step 2: *Ex-situ* electrochemical characterization using MEMS chips



- Voltammogram for $[K_4Fe(CN)_6]$ = 2 mM
- Supporting Electrolyte [KCl] = 1M
- Working Electrode : Glassy Carbon
Reference & Counter : Platinum
Flow Rate = 300 µL/h

Step 3: *Ex-situ* electrochemical characterization in in-situ holder

Step 4: *In-situ* electrochemical characterization in TEM



- Voltammogram for [HAuCl4]= 1 mM
[NaCl] = 0.1M,
- Scan Rate = 50 mV/s
Flow Rate: 150 µL/h
- Working Electrode : Glassy Carbon
- Reference Electrode : Platinum

WP6 highlights DENS

Task 6.3 - TEM in liquid environment

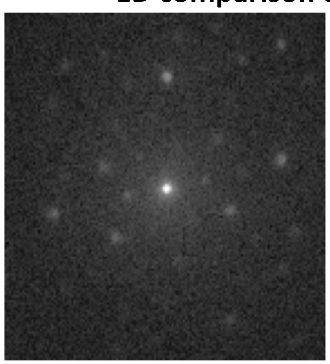
Precession electron diffraction in a gas Nano-Reactor

(collaboration between DENSsolutions & NanoMegas)

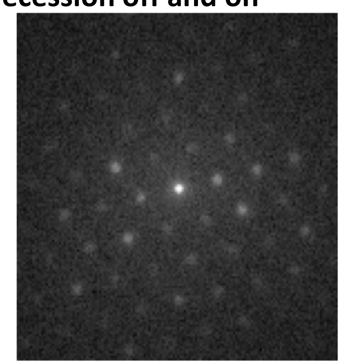
Material questions to answer: How the phase and strain of nuclear fuel material Zr evolve under pure oxygen and pure water vapor environment ? What's the mechanism driving faster cracking under water vapor?

Closed cell environmental holders only have one tilt axis, with proper FIB sample preparation, PED can give more reliable orientation and strain analysis results.

ED comparison of precession off and on



No precession



1˚ precession

Technical and material science manuscripts under preparation

Iron Oxide Reduction and Oxidation

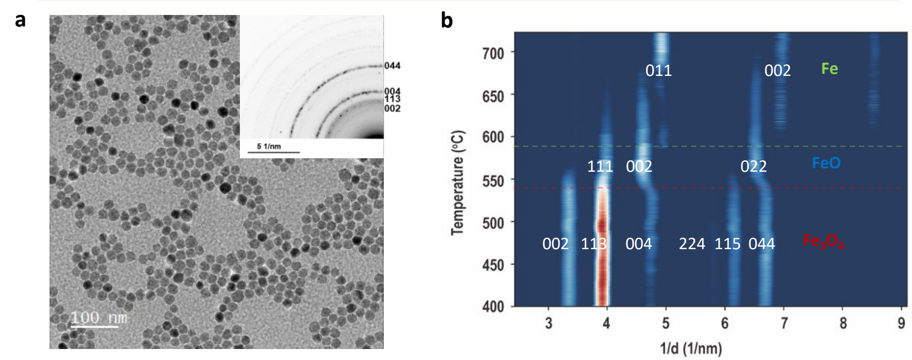
(collaboration between DENSsolutions and IOP, CAS, China)

Reduction is critical preliminary step in catalyst activation. When people started to step from ETEM to closed cell in this direction, discrepancy in temperature values caused a lot questions in instrument leak tightness. IOP's material science motivations and DENS's instrument and workflow configuration / improvement motivation met to this collaboration.

A few technical conclusions:

- 1 bar is different from 1 mbar chemically
- In situ system maintenance and workflow are critical

As-synthesized Fe_3O_4 particles and integrated in-situ SAED of Fe_3O_4 nanoparticles under reducing environment

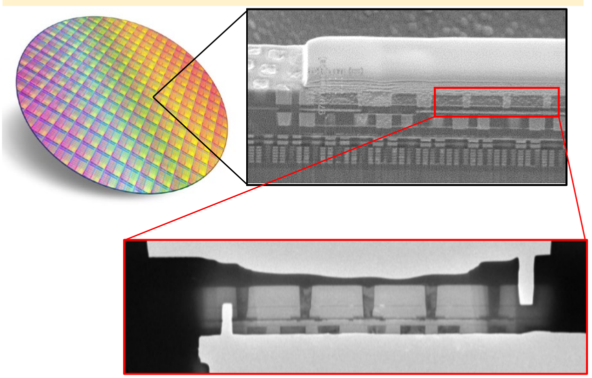


WP6 highlights TOU

Task 6.1 - Sample preparation

- FIB sample preparation protocols allow *in-situ* biasing experiments on devices and thin layers

From wafer to in-situ device by FIB

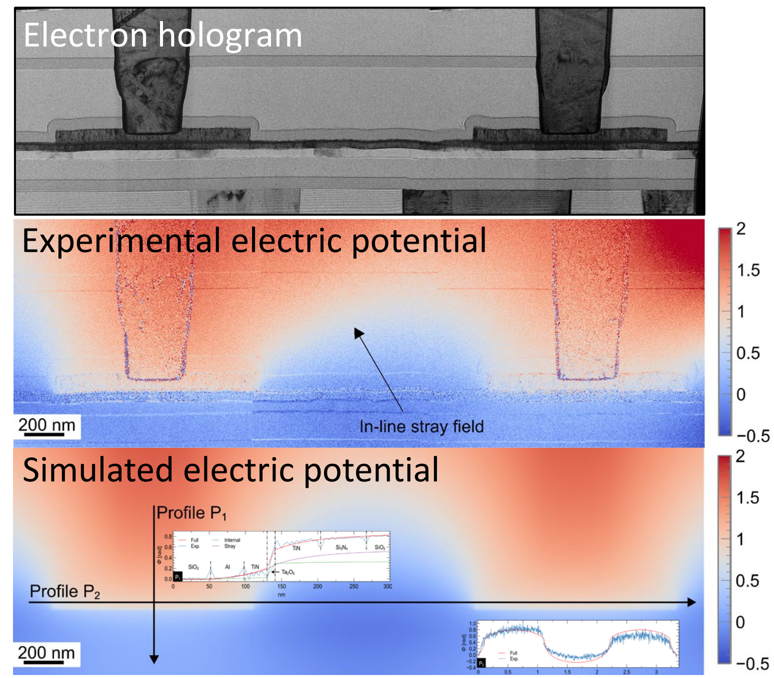


MIM nanocapacitors (STMicroelectronics)

Task 6.4 - In-situ biasing

- Electric fields measured by automated electron holography on working devices from industry

From operando holography to electric fields



Brodovoi et al. APL (2022) 10.1063/5.0092019, hal-03752638
Gatel et al. PRL (2022) 10.1103/PhysRevLett.129.137701, hal-03787333
Technical and material science manuscripts under preparation

WP6 highlights UA

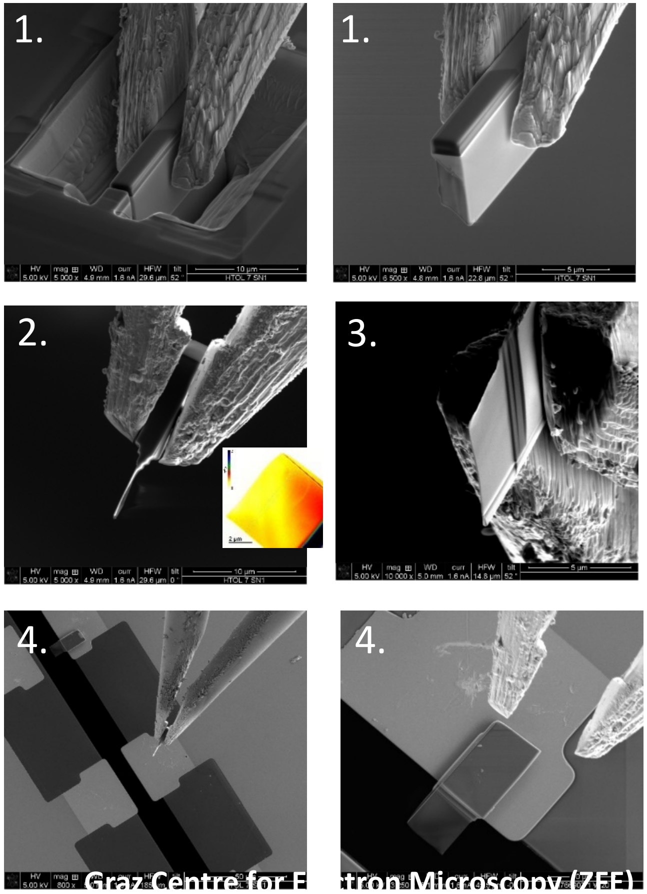
Task 6.4 - In-situ biasing

- Orientation dependent biasing with pure electric field (sample contacted to 1 electrode) [No current flow or Joule Heating]
- Trigger insulator to metal phase transition with electric field, current and voltage control (reproduced bulk behavior on FIB lamella)
- Electrical switching of piezoelectric capacitor and measurement of fatigue behavior (up to 4E9 cycles) [bulk behavior reproduced with in-situ fatigue]…

WP6 highlights GRA

Task 6.1 - Combined Kleindiek devices for in situ preparation

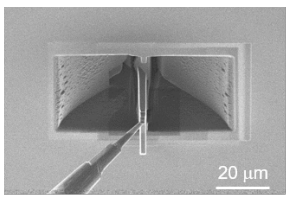
- Combination of plug-in tools in FIB:
  - ROTIP-EM Rotational Tip
  - MGS2-EM Microgripper
- Free rotation of Microgripper for different procedures:
  1. *Lamella liftout*
  2. *Conventional ("frontside") thinning (t/λ map as insert)*
  3. *Backside thinning*
  4. *Sample deposition on chip*
- Multiple benefits vs. conventional lamella preparation & transfer:
  - ➢ No metal deposits necessary for transfer and deposition
  - ➢ Free rotation of lamella for thinning options
  - ➢ Only possibility for cryo conditions

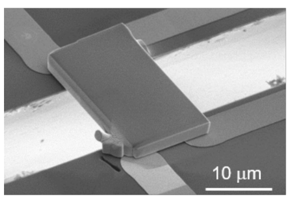


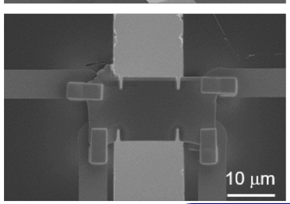
WP6 highlights STU

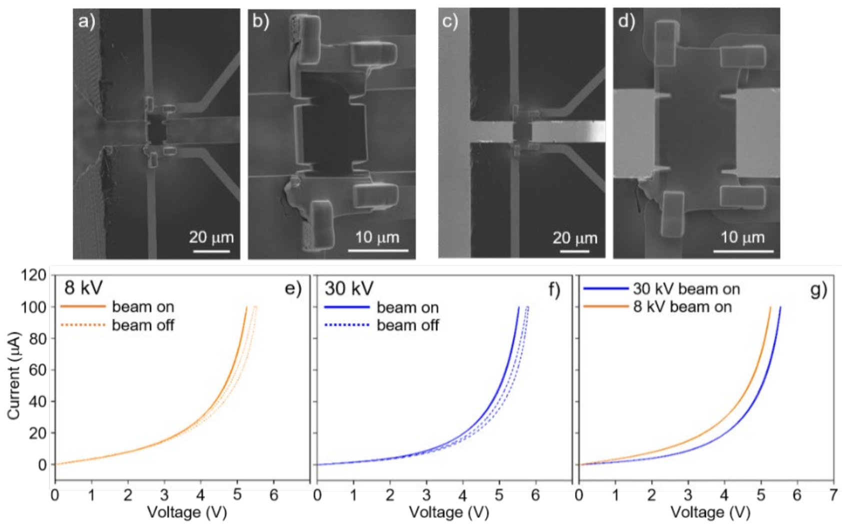
Task 6.1 - Preparation of High-Quality Samples

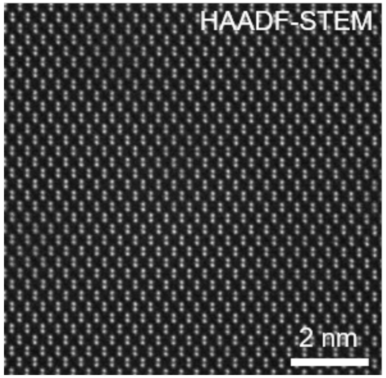
- *Novel FIB-based methodology development for the preparation of clean and artifact-free specimens on MEMS-based chips for in-situ electrical and electro-thermal experiments*











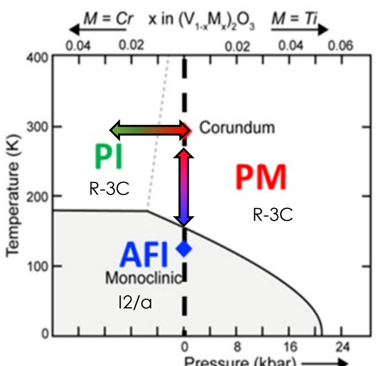
V. Srot et al. Microscopy and Microanalysis ***29*** *(2023) 596-605*

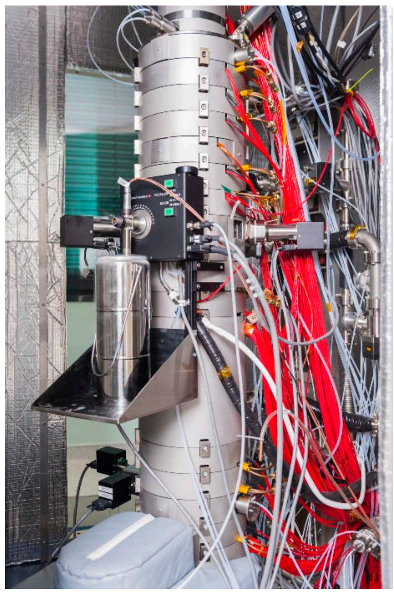
Electric-field-activated metal/insulator transitions in Cr-V_2O_3 investigated by *in situ* electron spectromicroscopy techniques

in the framework of the ANR project IMPULSE

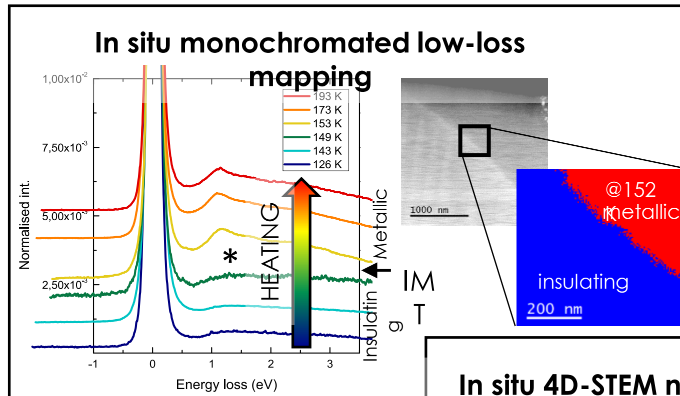
@ STEM group at the Laboratoire de Physique des Solides, Université Paris-Saclay (Orsay, France)

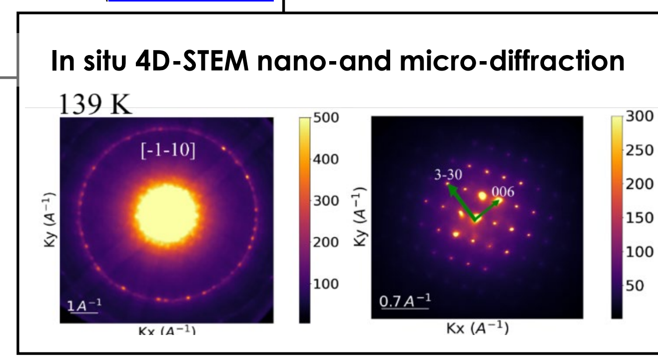
➤ a 2-year post-doctoral position





NIOX CHROMATEM and Henny Z *in situ* options





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https://equipes2.lps.u-psud.fr/stem/category/jobs

Equipement d'excellence

esteem3

Workpackage 7
Materials for ICT

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

WP-7 Objectives / Tasks

To **implement and develop different TEM characterization techniques** (from specimen preparation to diffraction and imaging, electron tomography, spectroscopy, holography and in-situ investigations, with adequate energy and spatial resolution) for the study of ICT materials.

To **explore the properties and performance** characteristics of novel materials and material combinations for ICT, to **provide the understanding necessary to improve and develop materials** to engineering readiness.

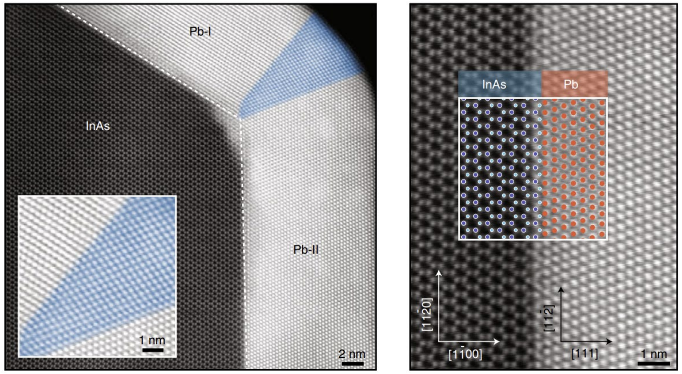
Task 7.1: *Semiconducting and magnetic materials* (GRA, ZAR, CHA, TOU, CAT)

Task 7.2: *Functional complex oxides, carbon and related nanostructures* (ZAR, STU, ORS, TOU)

Task 7.3: *Photonic materials* (GRA, ZAR, STU, ORS, CHA)

Task 7.4: *Sample preparation* (GRA, ZAR, STU, CHA, TOU, CAT)

7.1 Semiconducting and Magnetic Materials

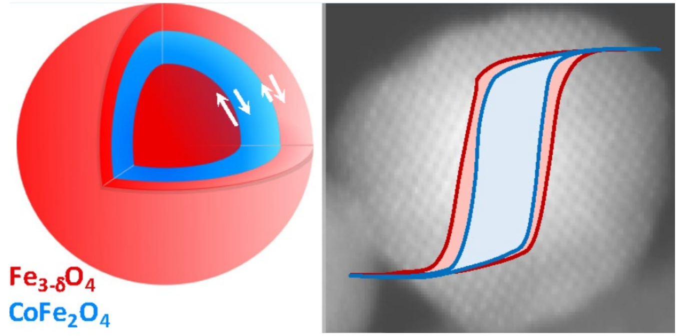


Quantum devices

Semiconductor–superconductor hybrids

Nature Nanotechnology 2021 16, 776

CHA

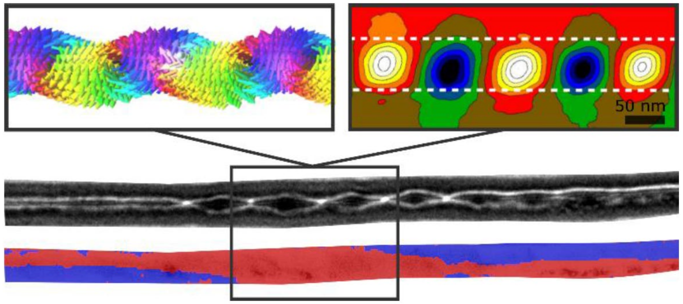


Interfacial magnetic properties

Nanoparticles

ACS Applied Materials & Interfaces 2021 13 (14), 16784

ZAR

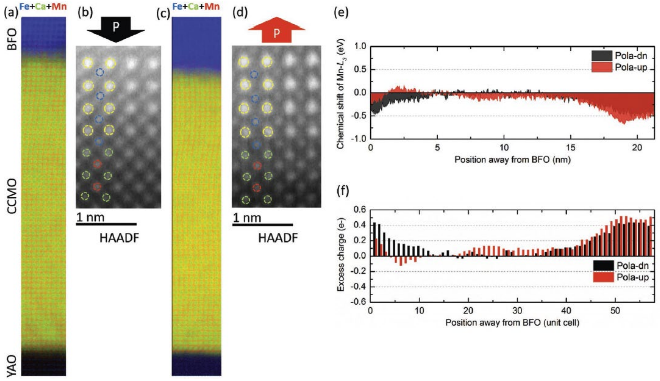


Exotic magnetic configurations

Nanowires

ACS Nano 2020, 14, 2, 1399–1405

TOU

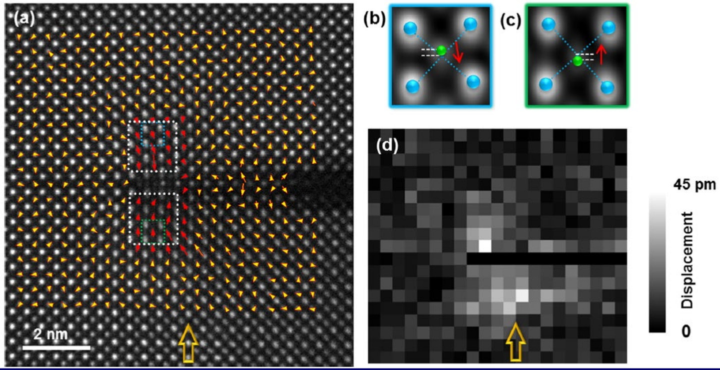


Ferroelectrics

Polarization switching in MOTT transistor

Adv. Mater. Inter. 2020 7(14), 2000601

ORS

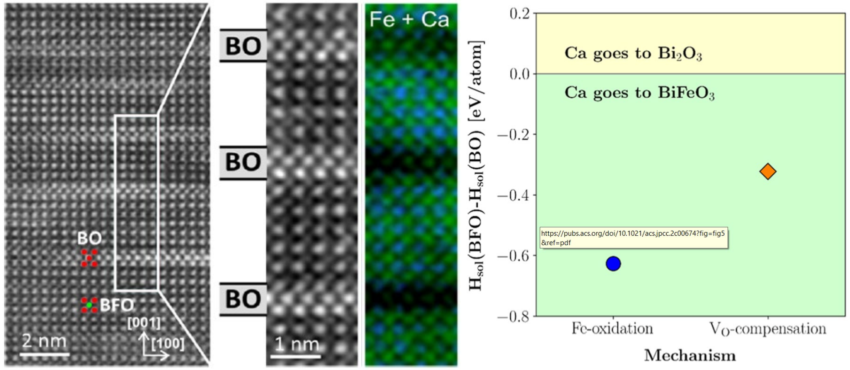


Flexoelectrics

Strain polarization coupling in STO

Nano Lett. 2020 20, 88

STU



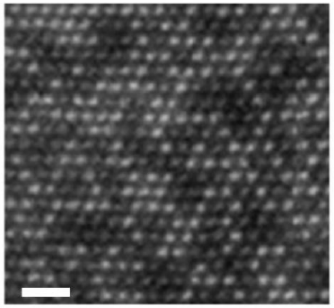
Multiferroics

Tuning BFO functionality by doping

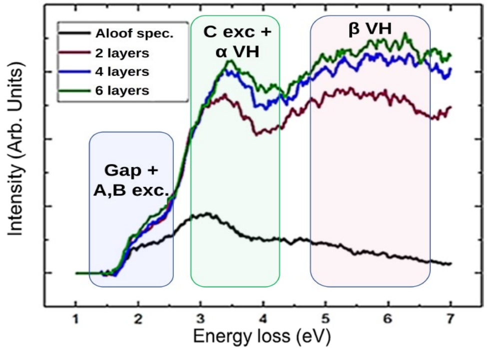
Nano Lett. 2020 20, 88

GRA

7.3 Photonic Materials



$Mo_{0.5}W_{0.5}S_2$

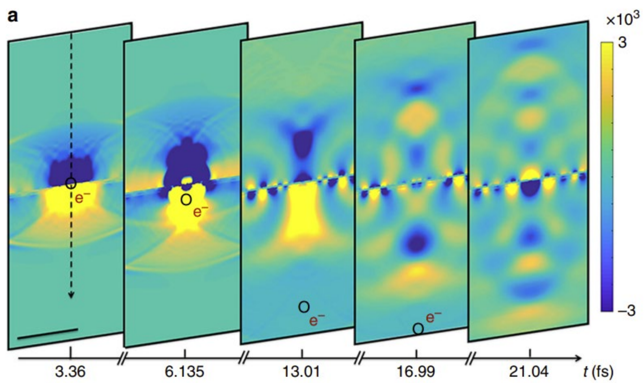


Band gap engineering

Transition metal dichalcogenide semiconductors for optoelectronics

Nanomaterials 2021 11, 3218

ZAR

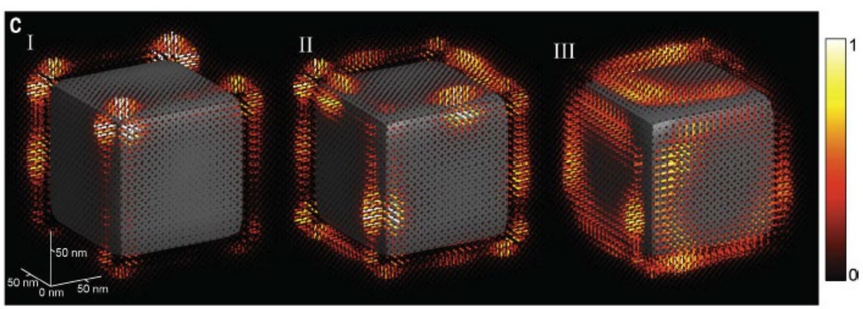


Coherent electron driven photon sources

Ultrafast spectral interferometry

Nature Comm. 2019 10, 599

STU



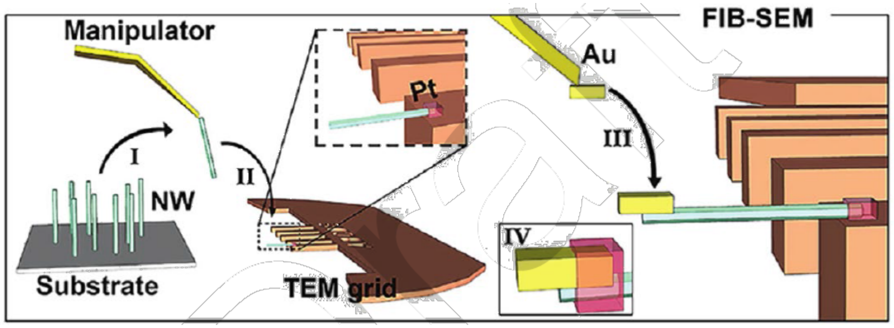
3D Phonon visualization

Mapping surface phonon polariton optical responses

Science 2021 371, 1364

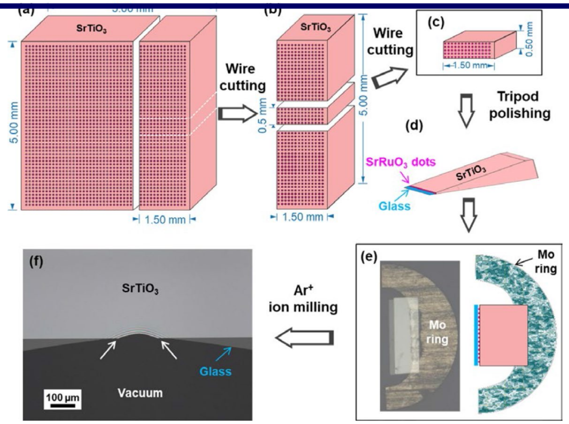
ORS/GRA

7.4 Sample Preparation



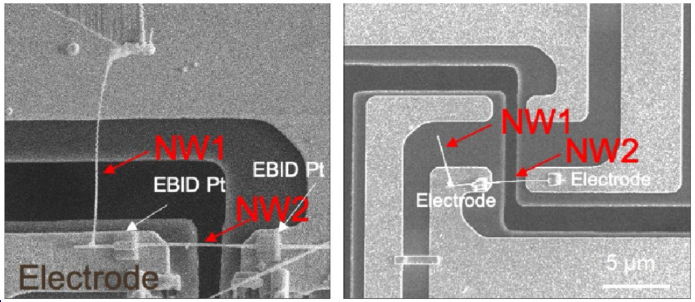
Preparation for *in-situ* electrical measurements on nanowires

Protocol on ESTEEM Website



Preparation of quantum dots

Protocol on ESTEEM Website



Specimen transfer to MEMS devices for *in-situ*

Protocol on ESTEEM Website

Workpackage 8
Materials for Energy

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

Materials for Energy

Task 8.1 Nanomaterials for Sustainable Development

Task 8.2: Dynamic Characterization of Energy Materials

Task 8.3: Characterization of Devices for Energy Applications

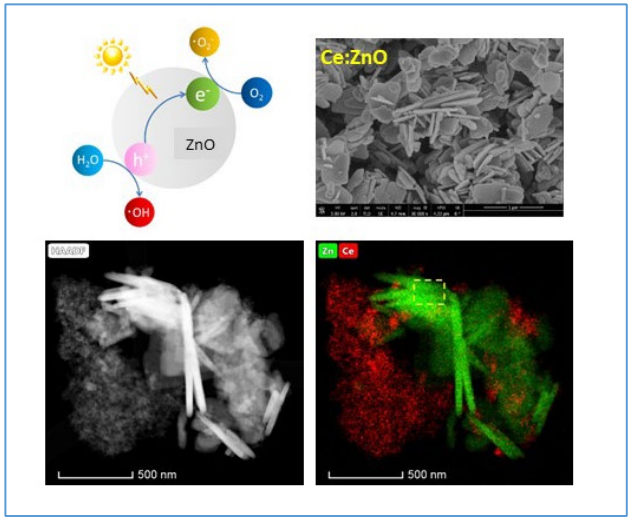
Task 8.4: Sample Preparation of Materials for Energy

Short name of participants		CNRS-TOU	CNRS-LPS	UCAM	JSI	UCA	AGH-UST	CAT
Person/months per participant:	PM	*	*	*	9	16	12	9

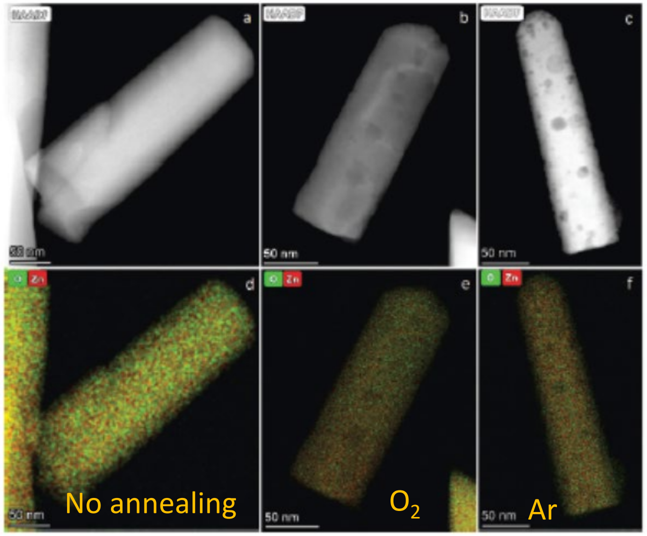
Task 8.1 Nanomaterials for Sustainable Development

Advanced electron microscopy techniques have been applied to characterised several nanostructure

Photocatalytic behavior of ZnO:Ce nanostructures

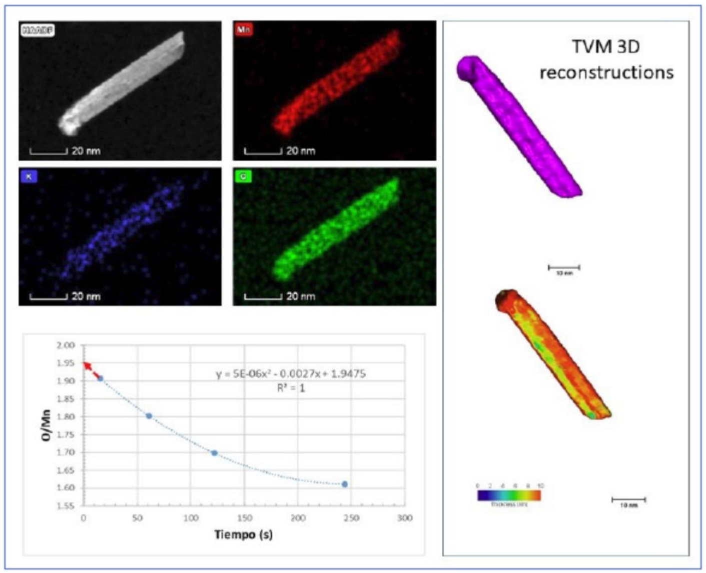


Optically active defects in ZnO nanorods subjected to thermal annealing in different environments (O_2 and Ar)



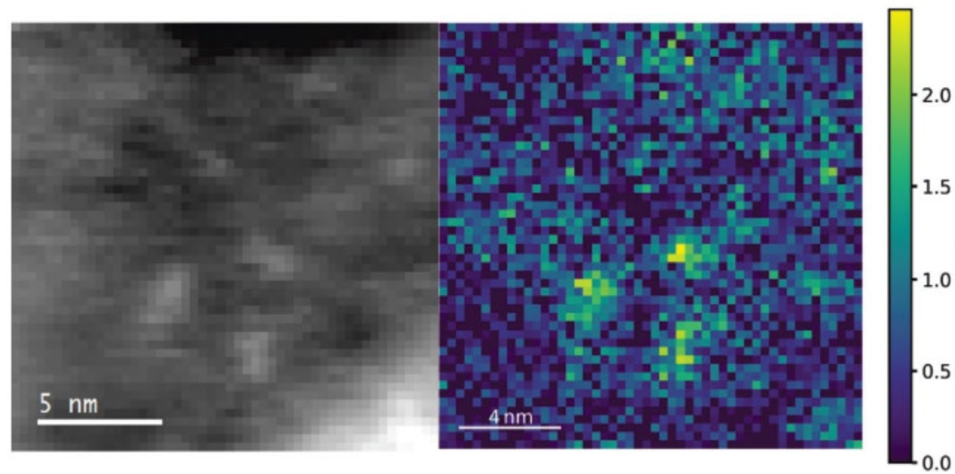
Advanced electron microscopy techniques have been applied to characterised several nanostructure

Oxygen evolution on beam sensitive energy nanomaterials based on hollandite and ceria



Using an alternative approach to determine the z-factors (developed within WP5), the phase transformation from fluorite type CeO_2 to the cubic sesquioxide, Ce_2O_3, phase has been tracked by exposing the sample to the electron beam.

Development of 2D materials-based heterostructures for electrocatalysis



Graphitization of Ni-based metal organic

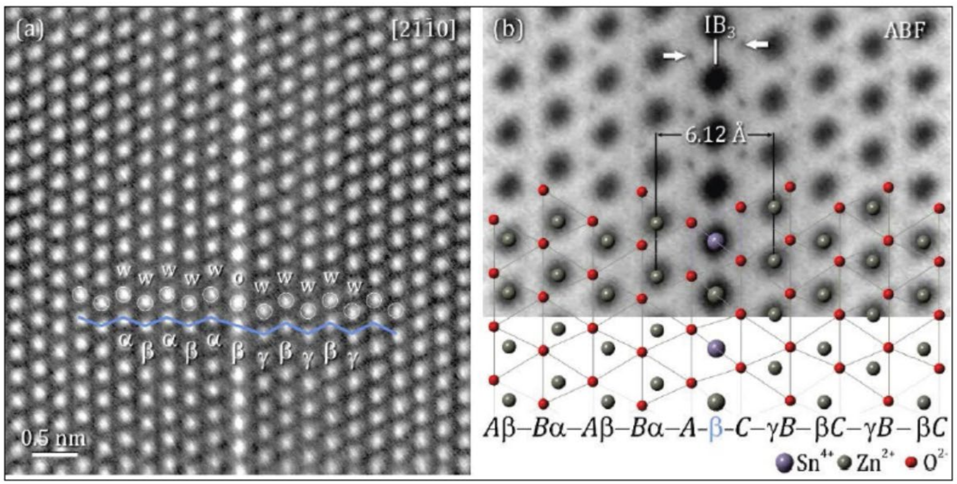
↓

Synthesis of single-atom nickel catalysts (SAC) on two-dimensional nitrogen-doped carbon nanosheets

Task 8.1 Nanomaterials for Sustainable Development

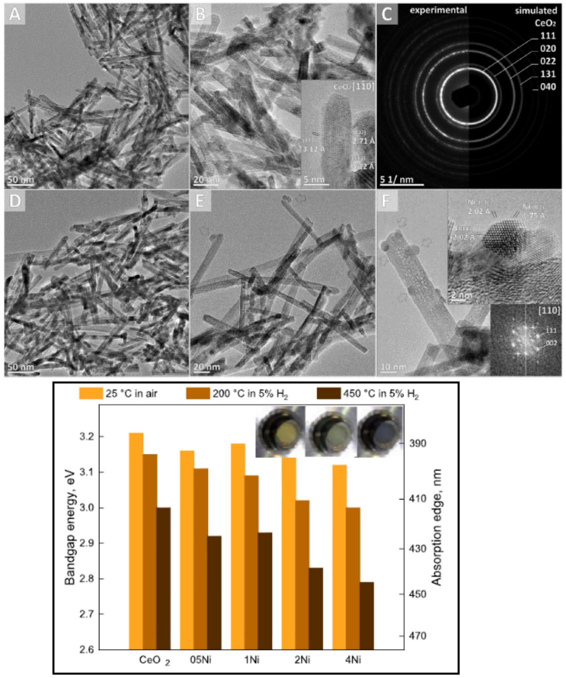
Advanced electron microscopy techniques have been applied to characterised several nanostructure

TEM and DFT Study of Basal-plane Inversion Boundaries in SnO2-doped ZnO



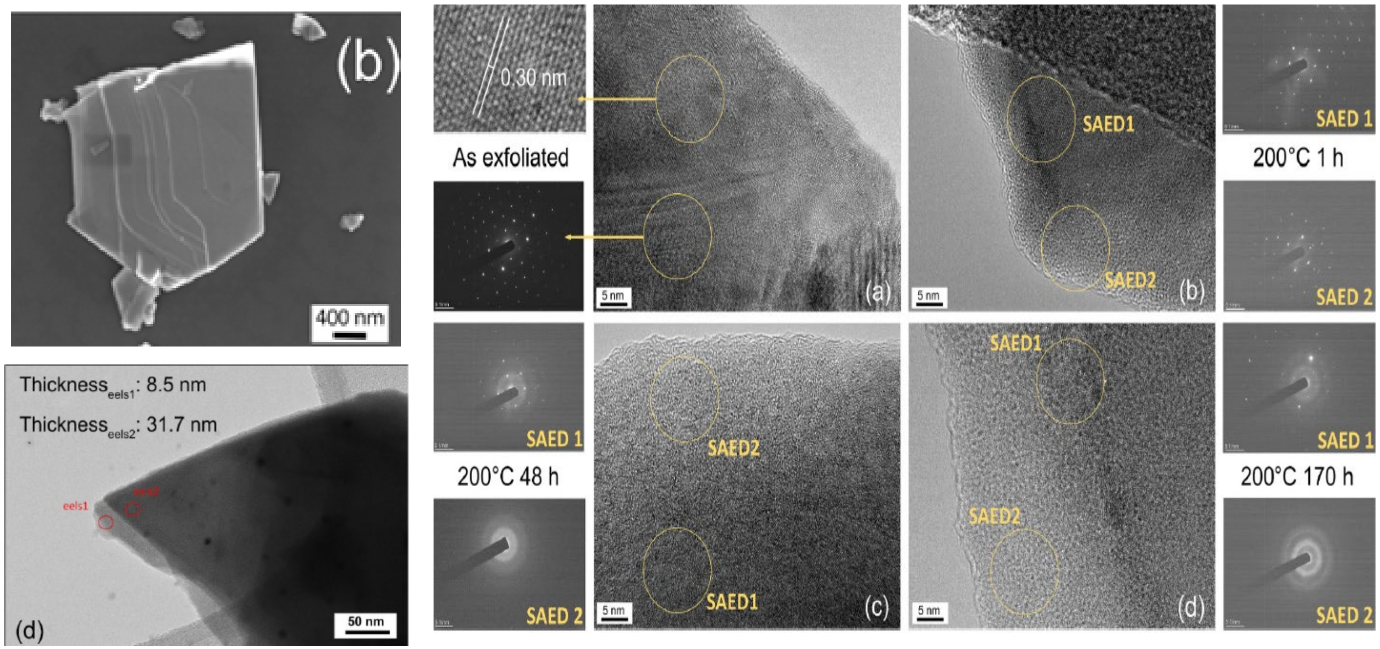
High-resolution STEM images of the Sn-rich IB in [21-1-0] projection. (a) HAADF-STEM image displaying a higher contrast for atomic columns along the IB-plane. Cation positions facilitate a direct identification of IB translation. (b) ABF-STEM image showing the positions of O-columns (weak dark dots).

Catalysts: Ni/CeO2-x catalysts for methane dry reforming reaction



Bandgap energies for bare CeO[2]-R and Ni/CeO_2 catalysts containing 0.5–4 wt% Ni in air at 25 ◦C and 200 ◦C and 450 ◦C in 5% H_2/N_2 atmosphere. Inset shows the 2Ni catalyst at mentioned temperatures and atmospheres.

Sensors: Layered amorphous *a*-SnO_2 gas sensors obtained by controlled oxidation of 2D-$SnSe_2$



HRTEM images and related SAED patterns of the: (a) as exfoliated $SnSe_2$ flake; (b) 1 h; (c) 48 h; (d) 170 h annealed $SnSe_2$ flakes in air at 200 ◦C.

2D-layered amorphous *a*-SnO2 is utilized for the first time to detect NO_2, H_2 and NH_3 gases at 100 ◦C

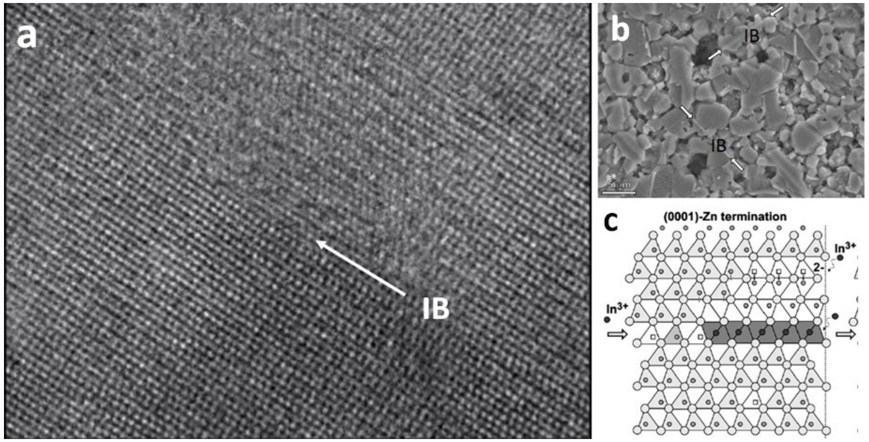
Amorphous *a*-SnO_2 thin-layers (10–40 nm thick) grown over 2D-SnSe

Sensors & Actuators: B. Chemical 350 (2022) 130890,

TA access_LAMOS

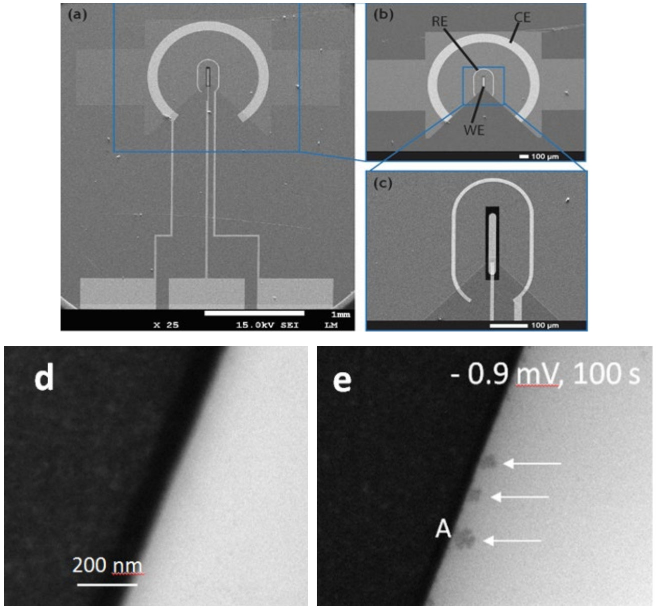
Task 8.2: Dynamic Characterization of Energy Materials

In-situ heating experiment of In^{+3}-doped ZnO



a) In-situ excerpt from movie showing a sequence of formation of the IB in In+3 doped ZnO. (b) IB's in In+3 doped polycrystalline ZnO. (c) The structure of the IB.

In-situ LC TEM experiment

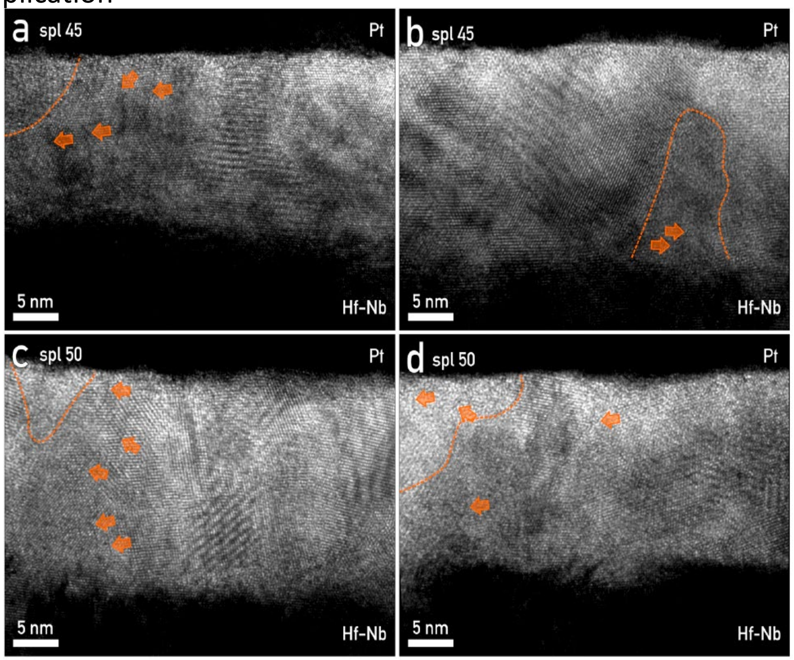


SEM micrograph of three electrode chip system for LC TEM (d) Work electrode before, and (e) after applying potential of -0.9 mV for 100 s. The nucleation of NI nanoparticles can be observed.

Task 8.3: Characterization of Devices for Energy Applications

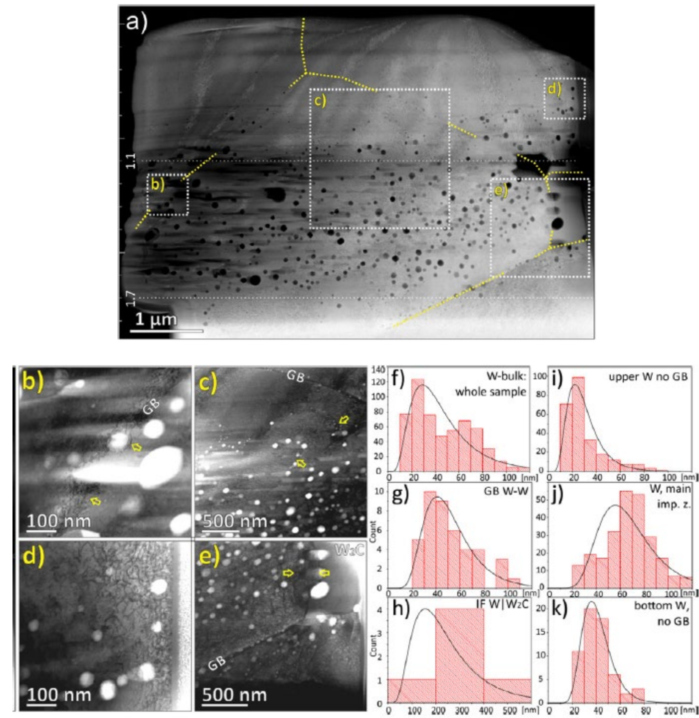
Anodic Memristors Grown on Hf–Nb Combinatorial Thin-Film Alloys

Development of novel materials with coexisting volatile threshold and non-volatile memristive switching is crucial for neuromorphic application



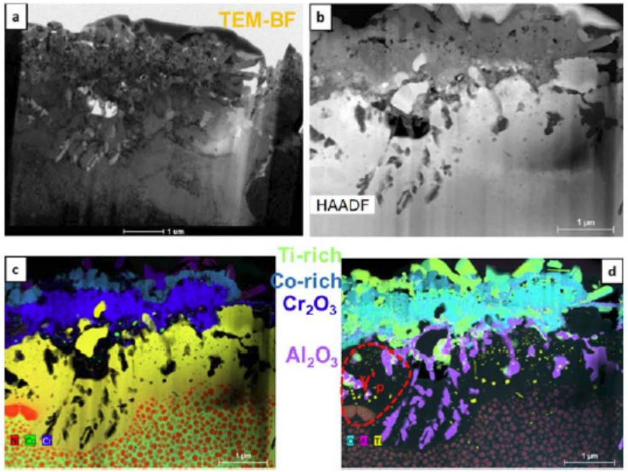
*HR-TEM micrographs of Hf–Nb oxide layer. (**a**,**b**) Sample Hf-45 at.% Nb, (**c**,**d**) sample Hf- 50 at.% Nb. Crystalline HfO_2 was sporadically interrupted by amorphous Nb_2O_5 regions (outlined with a dashed orange line). The conductive filaments emerging from metal layers are marked by orange arrows*

W/W2C composite for nuclear fusion reactor: The effect of helium (He)

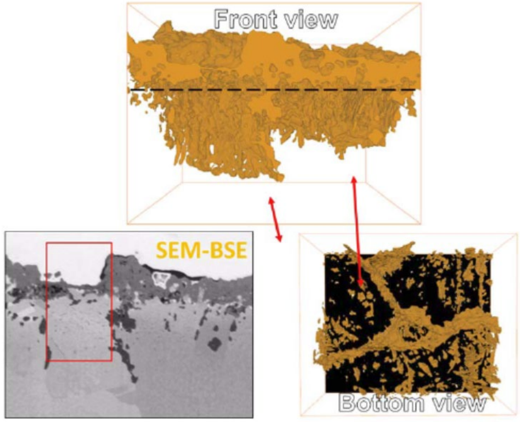


a) HAADF-STEM overview micrograph, with marked GBs *(yellow lines) and regions of BF-STEM insets (white squares): b) bubbles at the W-W GB; c) bubbles outside the main implantation zone are limited to GBs (yellow arrows); d) He bubbles in the W grain outside main implantation zone, near sample surface; e) large elongated bubbles on inclined W|W 2 C interface (arrows mark upper and bottom contact*).

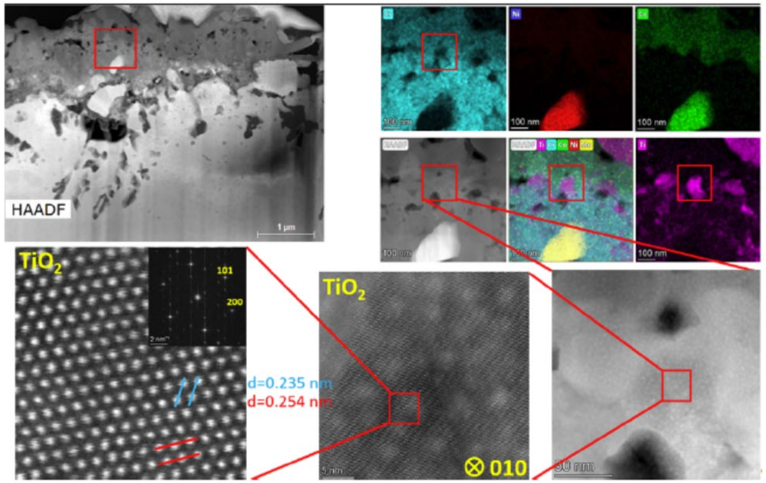
Oxidation behaviour and scale formation in modern nickel-based superalloy produced via powder metallurgy route



Lamellae of the near-surface area of ME16 superalloy oxidized for 1000 hours at 730 °C. a) TEM-BF image of the lamellae; b) STEM-HAADF image of the lamellae; c) Compositional STEM-EDXS elemental distribution map of Ni, Co, and Cr in the lamellae; d) Compositional STEM-EDXS elemental distribution map of O, Al, and Ti in the lamellae.



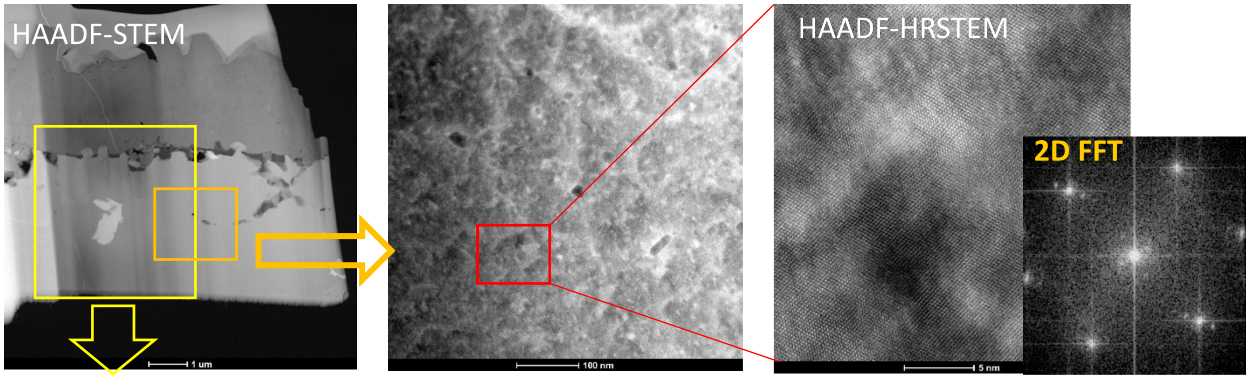
FIB-SEM tomographic reconstruction of oxides in the near-surface area of ME16 superalloy oxidized for 1000 hours at 730 °C.



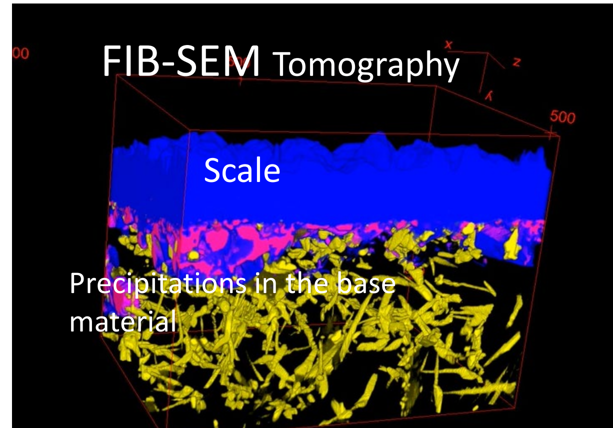
HRSTEM-HAADF/EDXS analysis of rutile at the interface of Co- and Cr-rich parts of the oxide scale.

Task 8.3: Characterization of Devices for Energy Applications

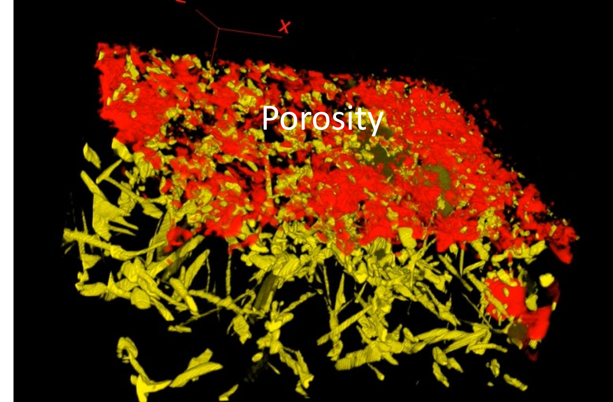
- **Influence of the high-temperature oxidation on the microstructure of Sanicro 25 steel used in energy systems**

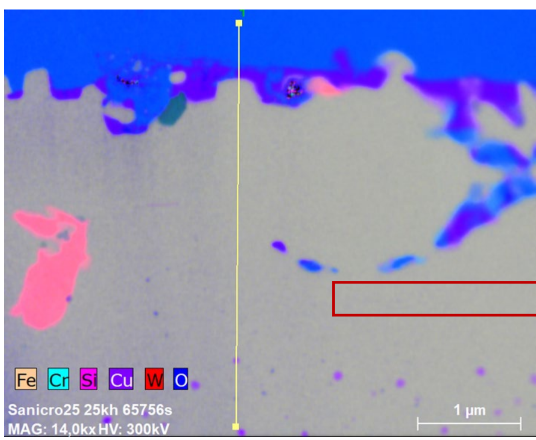


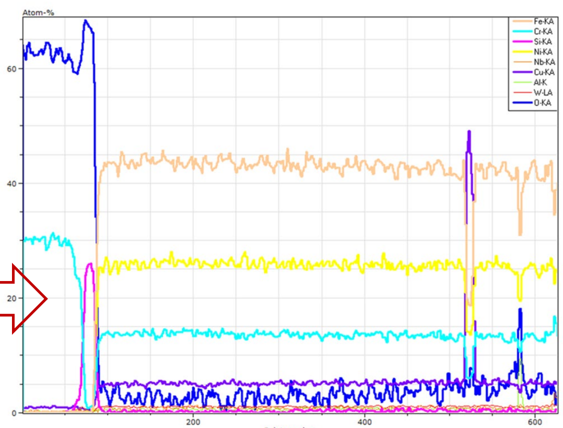
High-resolution TEM analysis of strengthening precipitates and a layer of scale after oxidation for 25 kh in steam

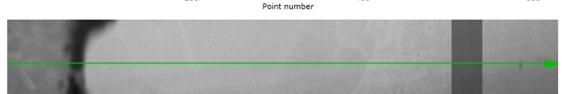


3D visualization of selected elements of the microstructure of the scale layer and the base material









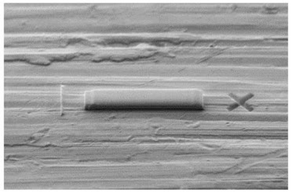
STEM-EDX analysis of the surface and linear distribution of selected chemical elements

Task 8.4: Sample Preparation of Materials for Energy

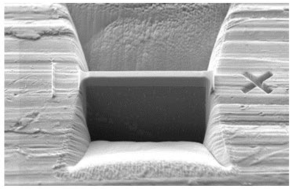
- ✓ TEM Sample Preparation using FIB, the FIB lift-out technique
- ✓ Correlative multiscale electron microscopy, 3D FIB-SEM tomography with detailed TEM investigation - Targeted Sample Preparation Stage
- ✓ Aerosol jet printing of battery electrodes for in situ electrochemical TEM

Protocols for sample preparation techniques of materials for energy

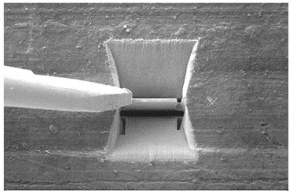
TEM Sample Preparation using FIB, the FIB lift-out technique



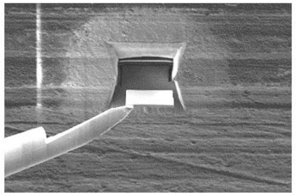
Stage 1: Selection of the area to cut out the lamellae (ROI) and place for deposition of the Pt layer.



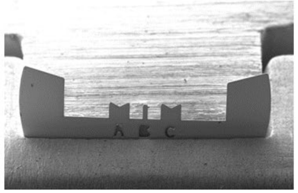
Stage 2: Pre-cutting of the lamellae



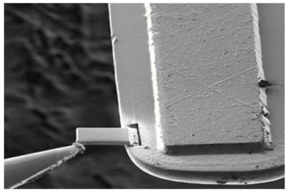
Stage 3: Welding the needle to the pre-cut lamella for pulling it out.



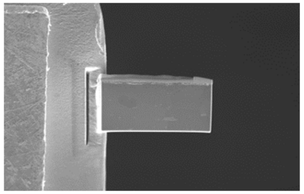
Stage 4: Pull out the pre-prepared lamella.



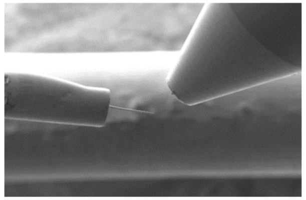
Stage 5: Scheme of the front side of a standard TEM grid.



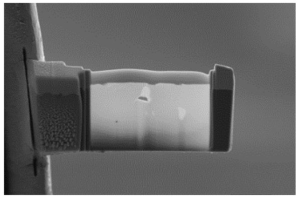
Stage 6: The stage of fixing the lamellae on the grid.



Stage 7: View of the mesh with the lamella attached.



Stage 8: The stage of final thinning of the lamellae.

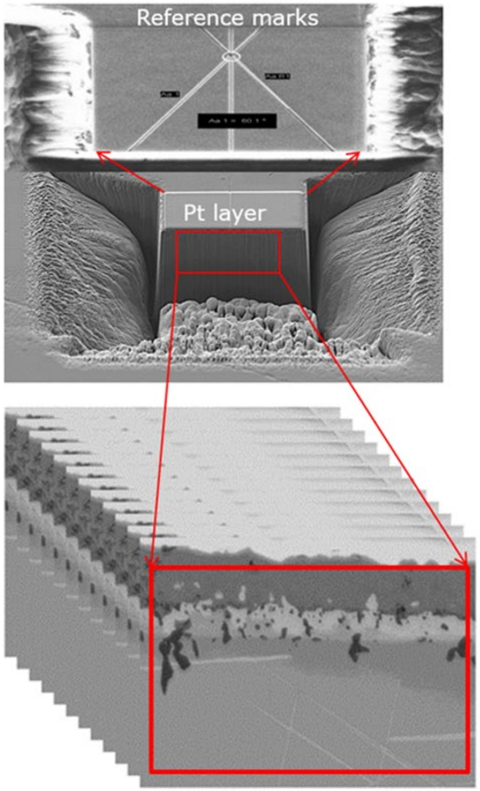


Stage 9: Finished thin lamella.

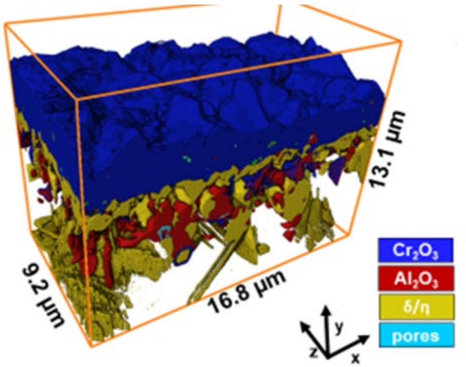
Protocols for sample preparation techniques of materials for energy

Task 8.4: Sample Preparation of Materials for Energy

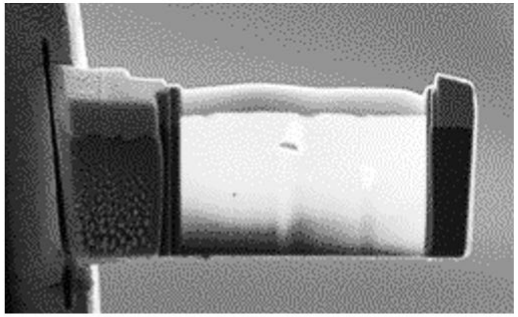
Correlative multiscale electron microscopy, 3D FIB-SEM tomography with detailed TEM investigation - Targeted Sample Preparation Stage



Tomographic series of SEM-BSE images from the area of the substrate material and the scale layer of the oxidized nickel superalloy sample.



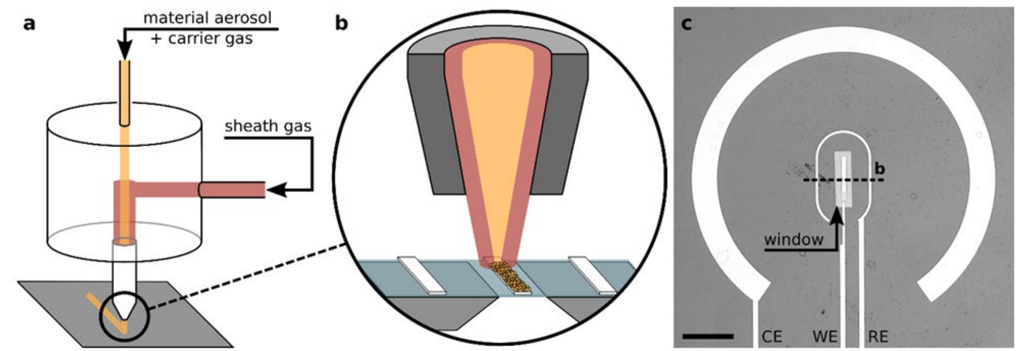
3D imaging of the microstructure elements of the base material and the scale on the surface of the 718Plus nickel superalloy sample.



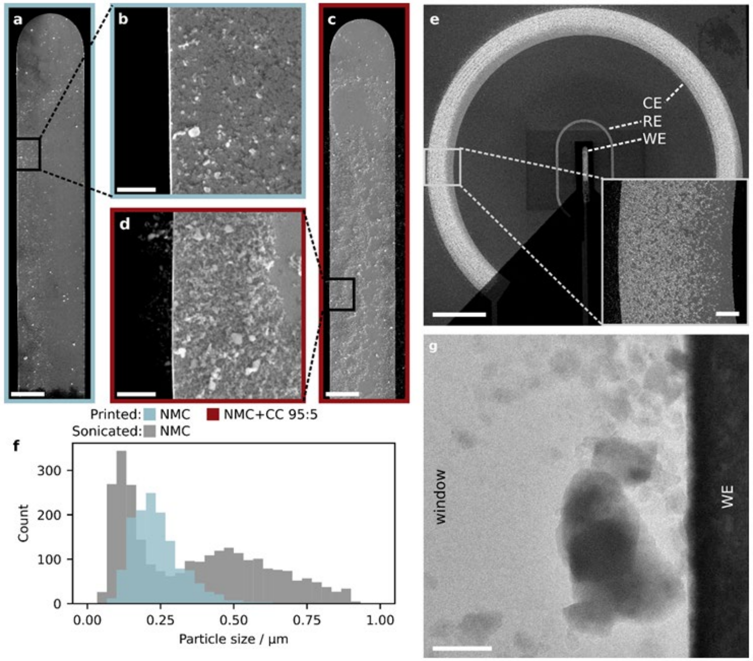
Lamella prepared following the steps of the FIB lift-out technique as the next stage after the tomographic acquisition of a series of images.

Task 8.4: Sample Preparation of Materials for Energy

Aerosol jet printing of battery electrodes for in situ electrochemical TEM



Schematic of the aerosol jet printing approach for EC-TEM sample preparation. (a) Schematic of the print head, where material aerosol (yellow) and sheath gas (red) are fed into the nozzle



SEM micrographs of aerosol jet printed lines on the working electrode of a Protochips EC-TEM chip.

The combination of the versatility, flexibility, size selectivity, positioning accuracy and spatial resolution make aerosol jet printing a very promising candidate for in situ or *operando* TEM sample preparation.

Materials for Energy

Related Published papers;

1.-Gutiérrez-Tarriño S., Portorreal-Bottier A., Trasobares S., Calvente J.J., Calvino J.J., Olloqui-Sariego J.L., Oña-Burgos P. Applied Surface Science, 623, 157001, 2023.
2.-Mandeep S., Aviraj I., González Garnica A.I., Mariathomas P., Ramanathan R., Patrick T., Christofferson A., Spencer M., Low M., Ahmed T., Walia S., Trasobares S., Manzorro R., Calvino J.J., García-Fernández E., Orte A., Domínguez-Vera J.M., Bansal V. ACS Nano 17, 9, 8083–8097, 2023.
3.-Psaradaki I, Costantini E, Rogantini D, Mehdipour M, Corrales L., Zeegers S. T, de Groot F., den Herder J.W.A., Mutschke M., Trasobares S., de Vries C.P., Waters L.B.F.M. Astronomy & Astrophysics 670, A30, 2023.
4.-J. K. Morzy, W. M. Dose, P. E. Vullum, M. C. Lai, A. Mahadevegowda, M. F. L. De Volder, C. Ducati. Origins and importance of intragranular cracking in layered lithium transition metal oxide cathodes, submitted December (2022)
5.-J.K. Morzy, PhD thesis, University of Cambridge (2022)
6.- A. Šestan, L. Sreekala, S. Markelj, M. Kelemen, J. Zavašnik, C. H. Liebscher, G. Dehm, T. Hickel, M. Čeh, S. Novak, P.Jenuš, *Acta Materialia*, 226 (2022) 117608, https://doi.org/10.1016/j.actamat.2021.117608
7.-I. Zrinski, J. Zavašnik, J. Duchoslav, A.W. Hassel, A. I. Mardare, Nanomaterials 2022, 12, 3944, https://doi.org/10.3390/nano12223944
8.- *J. Morzy,* A. Sartor, W. M. Dose, C. Ou, S. Kar-Narayan, M. F. L. De Volder, and C. Ducati,. Microdevices *Adv. Mater. Interfaces* (2022), 2200530 https://doi.org /10.1002/admi.202200530.
9.-P. Wozniak, M. A. Małecka, L. Chinchilla, S. Trasobares. Materials Research Bulletin 151 (2022) 111816. https://doi.org/10.1016/j.materresbull.2022.111816
10.-P. Wozniak, M. A. Małecka, P Kraszkiewicz, W. Miśta, O. Bezkrovnyi, L. Chinchilla, S. Trasobares. Catal. Sci. Technol 12, (2022) 7082 https://doi.org//10.1039/d2cy01214f

Related Published papers;

11.- G. žerjav, M. Roškarič, J. Zavašnik, J. Kovač, A. Applied Surface Science, 2022, vol. 579, p. 152196-1-152196-13.
12.- V. Paolucci, J. De Santis, L. Lozzi, G. Giorgi, C. Cantalini, Sensors & Actuators: B. Chemical 350 (2022) 130890, https://doi.org/10.1016/j.snb.2021.130890
13.-G. Žerjav, M. Roškarič, J. Zavašnik, J. Kovač, A. Pintar. Applied Surface Science, 579 (2022) 152196, https://doi.org/10.1016/j.apsusc.2021.152196
14.- L. Cabrera-Correa, L. González-Rovira, J. López-Castro, M. Castillo-Rodríguez, F. J. Botana.Materials Characterization 196, (2023), 112549. https://doi.org/10.1016/j.matchar.2022.112549
15.- I. Zrinski, J. Zavašnik, J. Duchoslav, A.W. Hassel, A. I. Mardare, Nanomaterials 2022, 12, 3944, https://doi.org/10.3390/nano12223944
16.-K. Lorber, J. Zavašnik, J. Sancho-Parramon, M. Bubaš, M. Mazaj, P. Djinović. Applied catalysis. B, Environmental, 2022, vol. 301, p. 1-11.
17.- V. Paolucci, J. De Santis, L. Lozzi, G. Giorgi, C. Cantalini.Sensors & Actuators: B. Chemical, 2022, 350, 130890.
18.- A. Šestan, L. Sreekala, S. Markelj, M. Kelemen, J. Zavašnik, C. Liebscher, G. Dehm, T. Hickel, M. Čeh, S. Novak, P. Jenuš. Acta materialia, 2022, 15
19.-Zhang Y. et al. ACS Catalysis, 11 (2021) 12701-12711.
20.- Koblar M. Master Thesis, Joyef Stefan International Postgraduate School, July 2021.
21.-Kruk A., Gil A., Lech S., Cempura G., Agüero A., Czyrska-Filemonowicz A., Materials (Basel), 14 (2021) 6327.
22.- Lech S, Kruk A., Gil A., Cempura G., Agüero A., Czyrska-Filemonowicz A., Scr. Mater., 167 (2019) 16–20.
23.- Lech S., Polkowski W., Polkowska A., Cempura G., Kruk A. Scr. Mat., 194 (2021) 113657.

Materials for Energy

Related Published papers;

24.- Damaskinos C.M., Zavašnik J., Djinović P., Efstathiou A.M..Applied Catalysis B: Environmental, 296 (2021) 120321.
25.- Ribić V., Rečnik A., Dražić G., Podlogar M., Branković Z., Branković G.,The periodical of the International Institute for the Science of Sintering, 53 (2021) 237.
26.- Tobaldi D. M. et al., Chemical Engineering Journal, 405 (2021) 126651.
27.-Tobaldi D. M. et al., Materials Today Energy, 19 (2021) 100607.
28.- Kruk A., Lech S., Gil A., Cempura G., Agüero A., Wusatowska-Sarnek A.M., Czyrska-Filemonowicz A. Corros. Sci., 169 (2020) 108634. 177.
29.- Manzorro, R., et al., ACS Catalysis, 2019. 9(6): p. 5157-5170

Materials for Energy

Deliverable

D8.1: First report on TEM methods applied to materials for energy (M16 – CAD)
D8.2: Second report on TEM methods applied to materials for energy (M30 – LJU)
D8.3: Third report on TEM methods applied to materials for energy (M42 – CAD)
D8.4: Report on protocols for sample preparation techniques of materials for energy (M46-CAT)

esteem3

Workpackage 9
Materials for Health

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

Work package number	**9**				**Lead beneficiary**					**STU, OXF**			
Work package title	**Materials for Health**												
Participants number	N°	1	3b	4	5	6	7	10					
Short name of participants		STU	ORS	ANT	OXF	CAM	LJU	CAD					
Person/months per participant:	PM	12	*	9	12	*	*	*					
Start month	1				**End month**					54			

Objectives

- Sample preparation protocols:
  - soft materials
  - organic/inorganic hybrid materials
  - soft-hard matter interfaces
- To implement 2D and 3D electron microscopy techniques for the structural and chemical characterisation of materials relevant to health care including pharmaceuticals, materials for environmental purification and biosensors
- To apply advanced spectroscopic methods to investigate nanoparticles and coatings for phototherapy

Tasks

Task 9.1: Sample preparation of materials for health (STU, ORS, ANT, LJU, CAM)

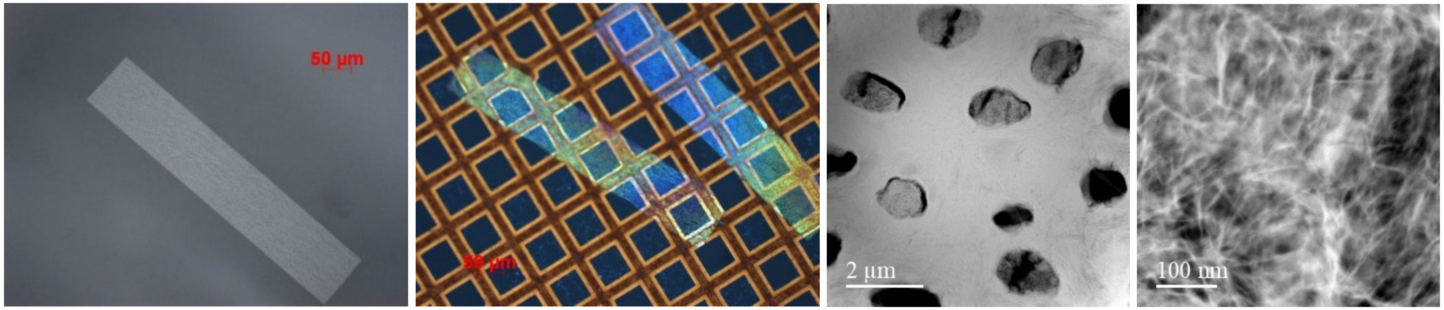
Task 9.2: Structural and chemical characterisation of materials for health (CAM, STU, OXF, ANT, ORS, CAD, LJU)

Task 9.3: Characterisation of nanoparticles for life science applications (OXF, ORS, ANT, LJU)

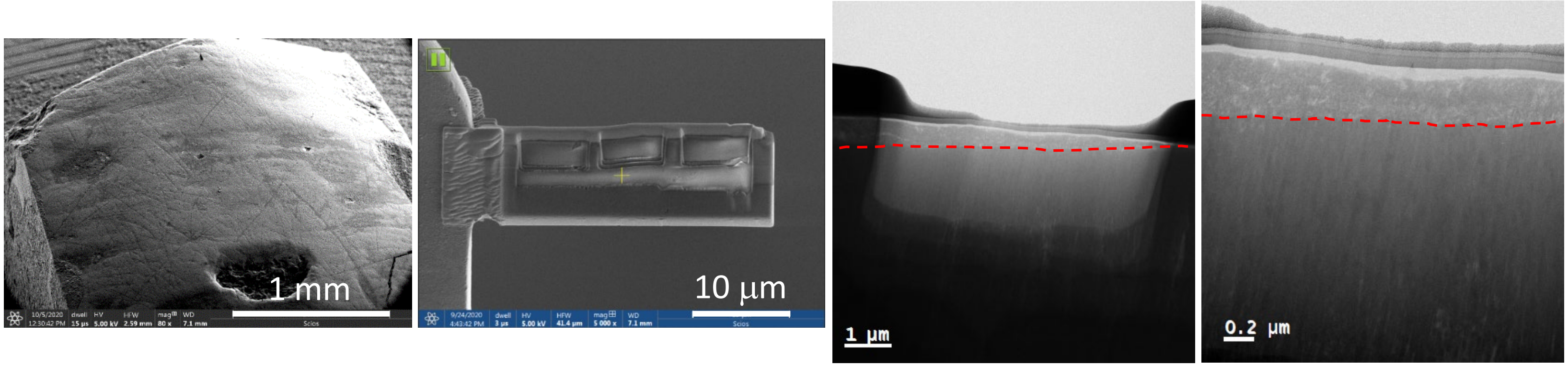
Task 9.1: Sample preparation

- Improving and developing new routines for the sample preparation of hybrid organic/inorganic materials from site specific positions for investigations of dental tissues and dental pathologies

- Preparation of human and rodent dentine by Ultramicrotomy (UM)



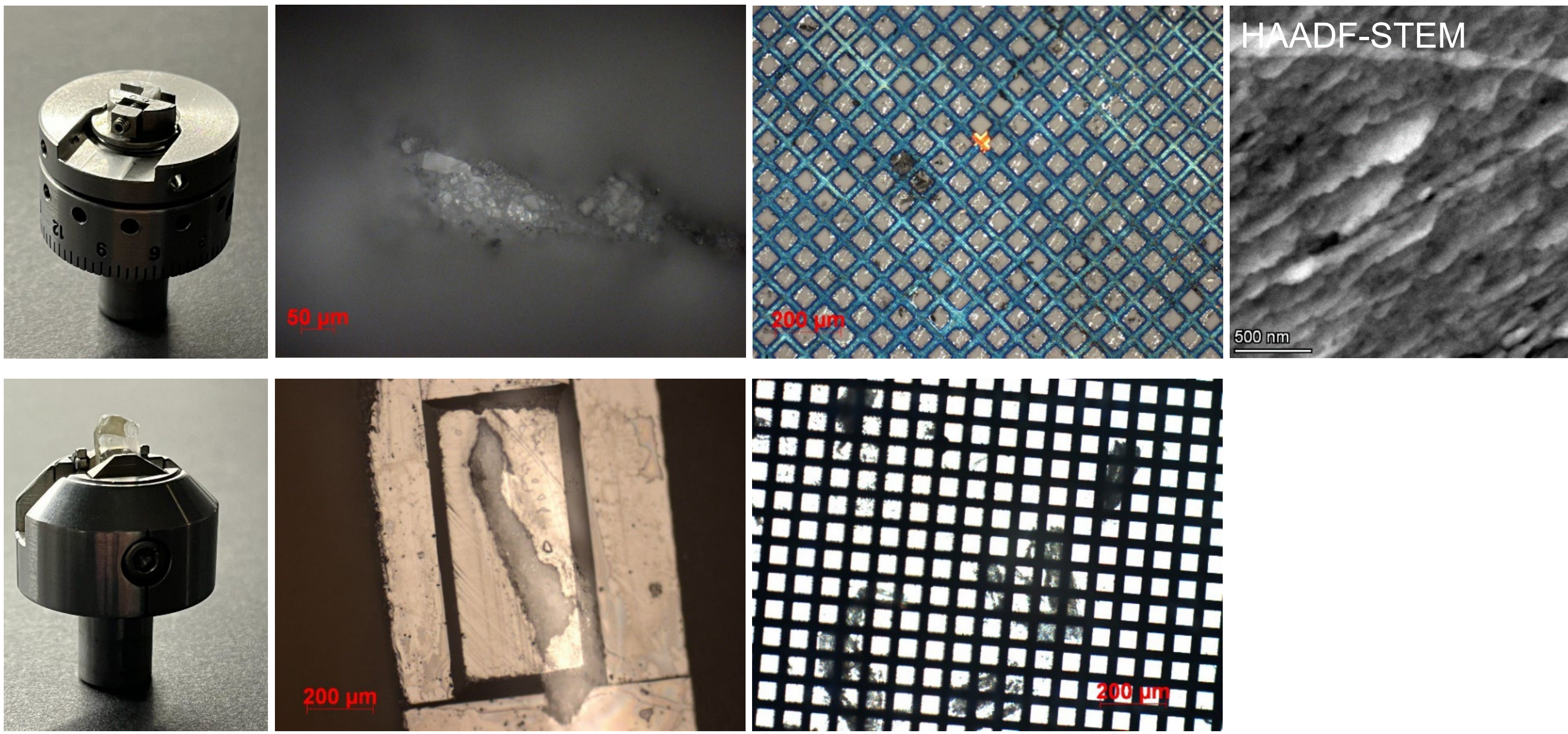
- Preparation of human dental pathologies by FIB (+ Nanomill)



Task 9.1: Sample preparation

STU+CAM

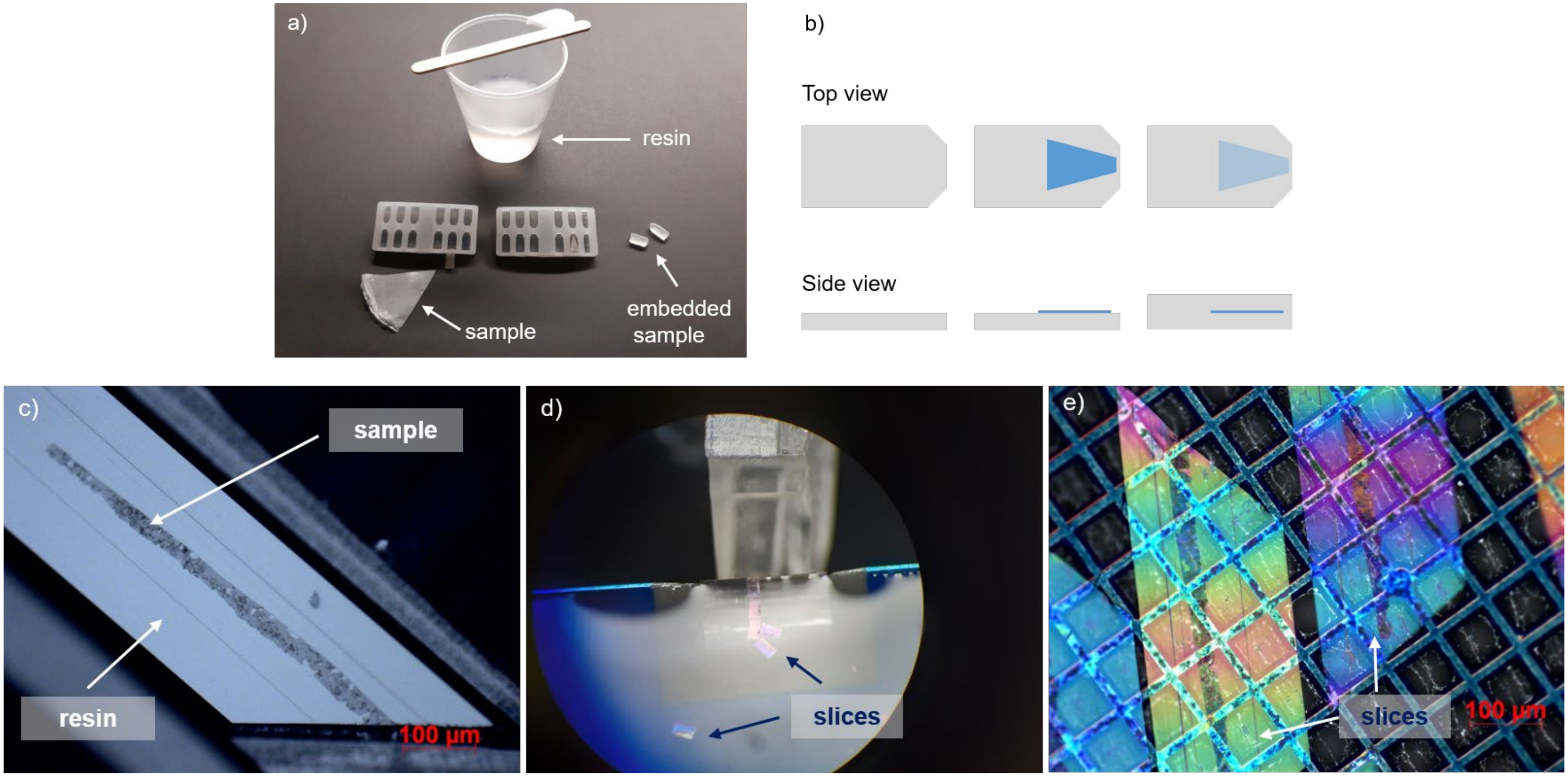
- Developing new routines for the sample preparation of pharmaceuticals (preliminary data)



Task 9.1: Sample preparation

STU

- Developing new routines for the sample preparation of biodegradable biopolyesters

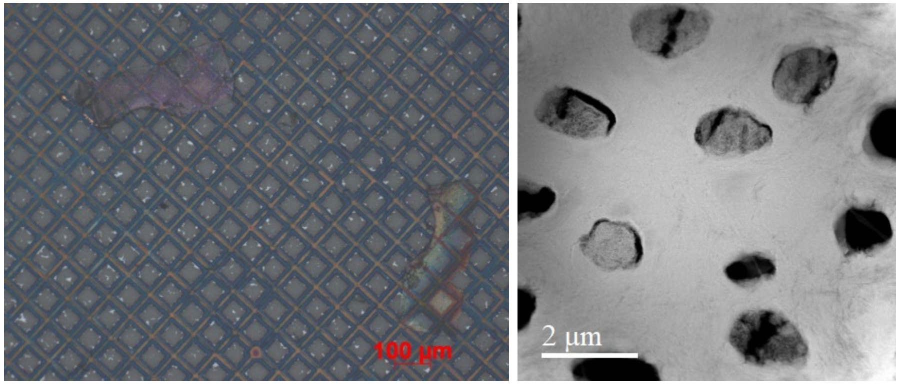


Task 9.2: Structural and chemical characterization

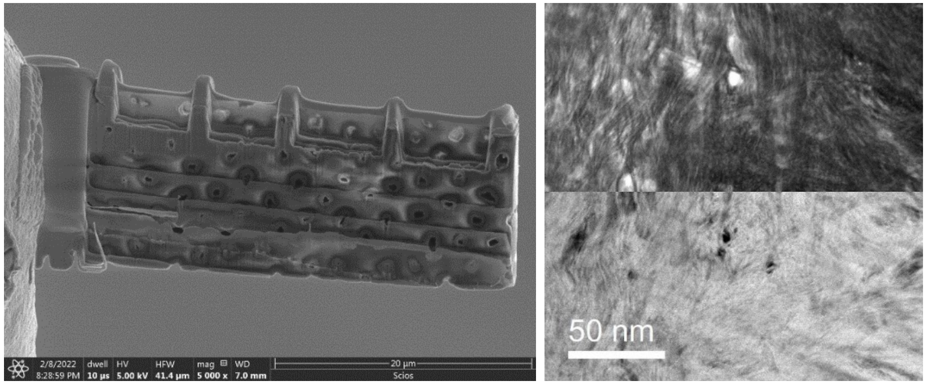
STU

- Structural and chemical and characterization of dental tissues

- UM



- UM + FIB (+NM)

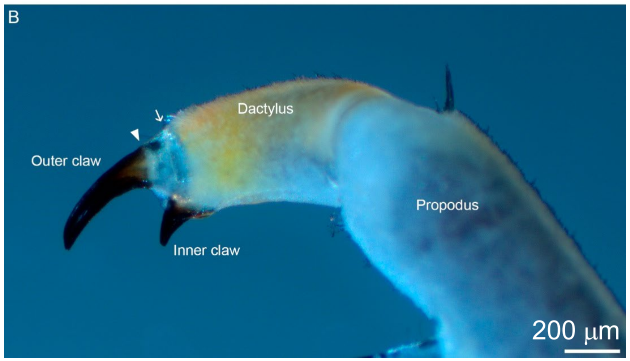


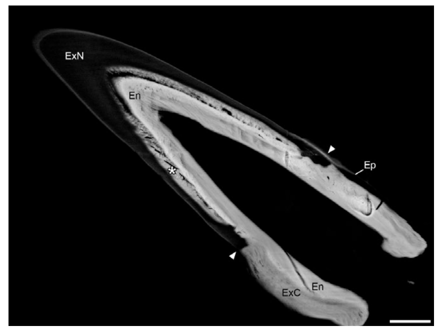
DENTIN	UM	FIB
Mg (at%)	5.4 ± 0.3	4.8 ± 0.2
P (at%)	31.3 ± 1.6	37.1 ± 1.9
Ca (at%)	58.3 ± 2.9	58.1 ± 2.9

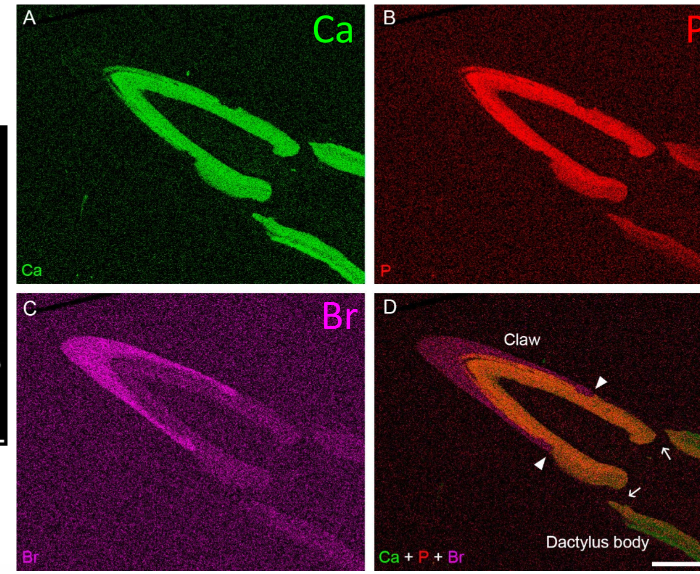
Srot, V. and van Aken, P.A. *et al.*: 2 manuscripts in preparation

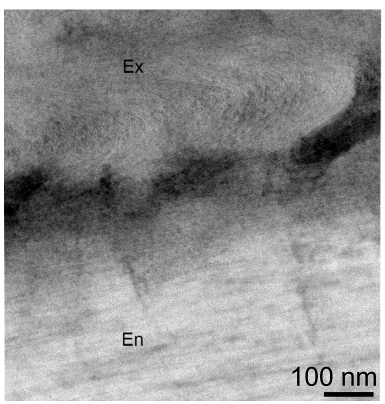
Task 9.2: Structural and chemical characterization

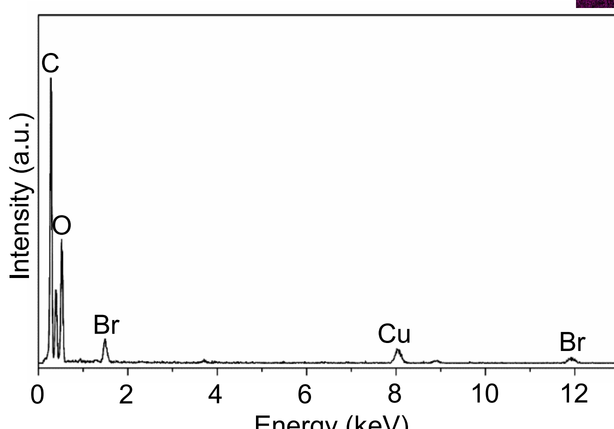
- Mineral distribution and organic matrix orientation in the claws of the sea slater

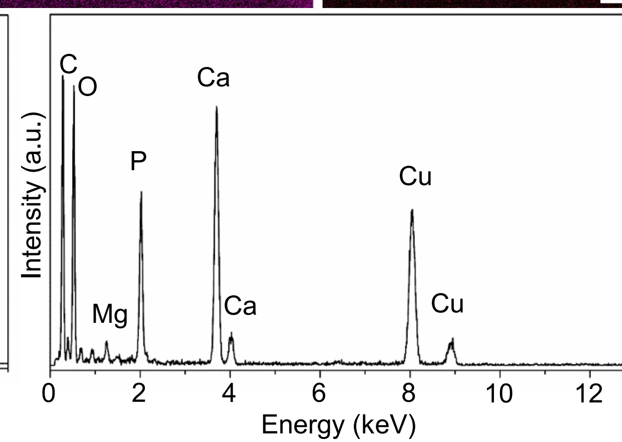










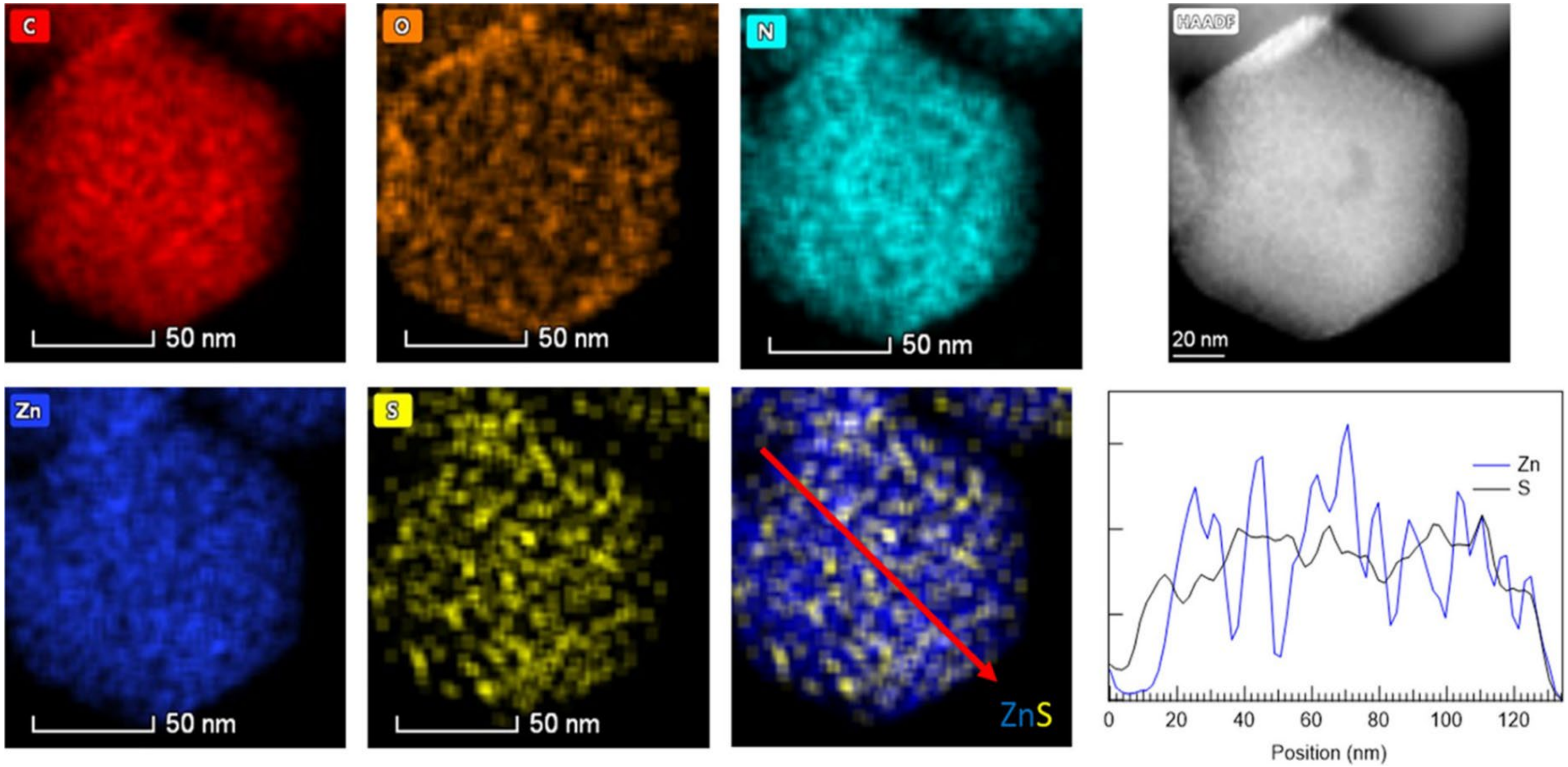


Vittori, M., Srot, V., Korat, L., Rejec, M., Sedmak, P., Bussmann, B., Predel, F., van Aken, P.A. Strus, J., *Minerals* **11** (2021) 1373.

Task 9.2: Structural and chemical characterization

CAD

- One-Step Encapsulation of ortho-Disulfides in Functionalized Zinc MOF. Enabling Metal–Organic Frameworks in Agriculture



Is the molecule encapsulated within the MOF?

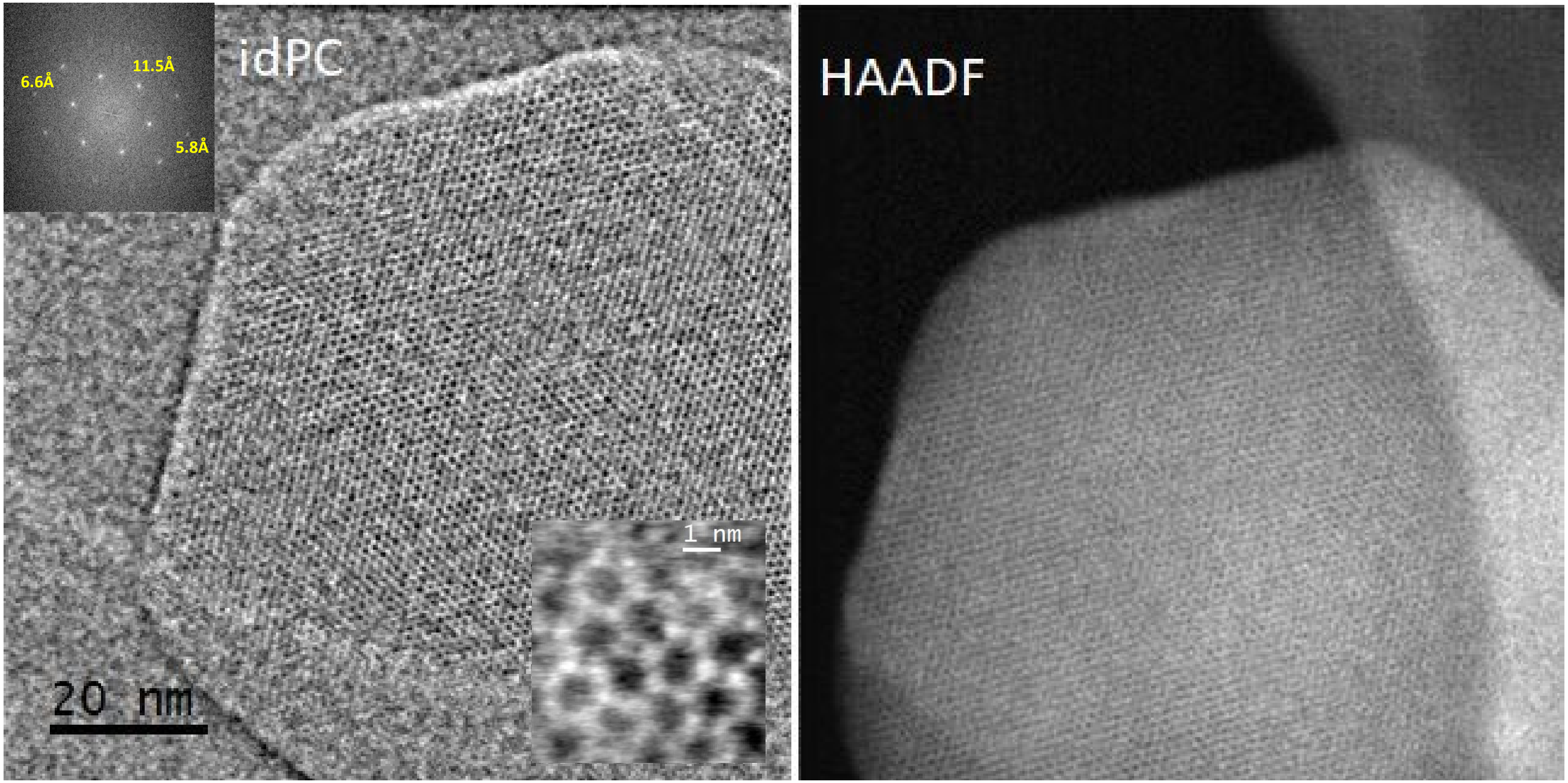
Crystals begin to lose crystallinity when the accumulated dose reaches 25 e- Å-2 and are completely damaged at a dose of 75 e- Å-2.

Beam sensitive materials MOFs

iDPC-STEM using 4Q Detectors
@ FEI TITAN Cubed Themis Microscope

Task 9.2: Structural and chemical characterization

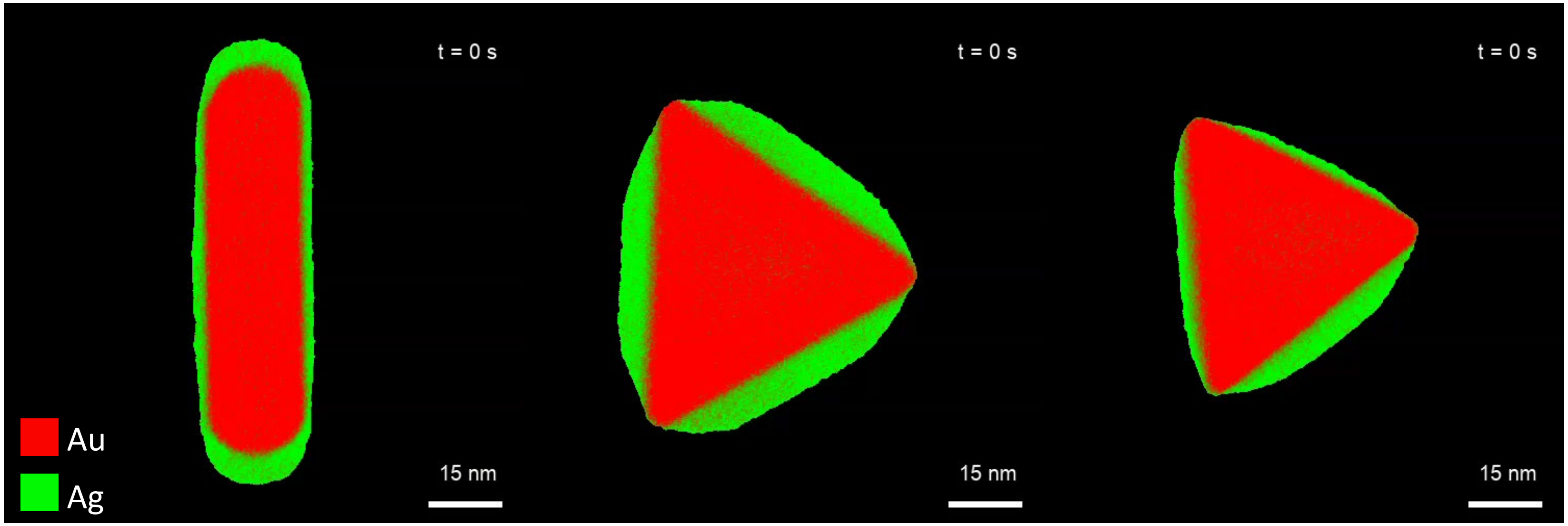
- One-Step Encapsulation of ortho-Disulfides in Functionalized Zinc MOF. Enabling Metal−Organic Frameworks in Agriculture



Mejias, F.J.R, Trasobares, S., Varela, R.M.., Molinillo, J.M.G., Calvino, J.J., Macias, F.A., *ACS Appl Mater Interfaces* **13(7)** (2021) 7997.

Task 9.3: Characterization of nanoparticles

- Alloying in AgAu nanoparticles; importance for sensing, (optical) hyperthermic cancer treatment
- 3D investigation using heating tomography

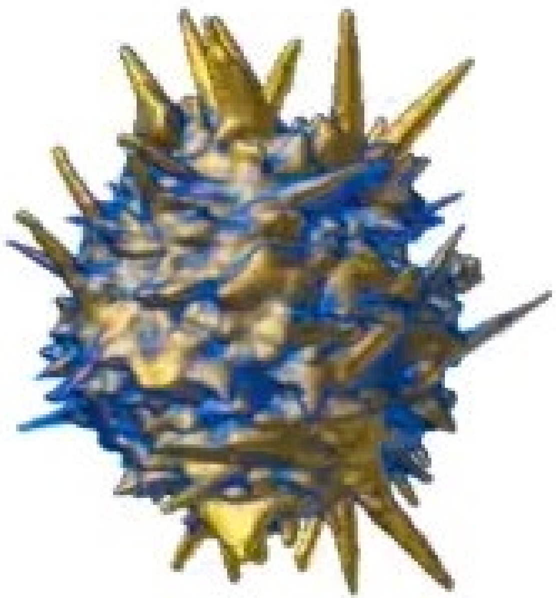


Skorikov A, Albrecht W, Bladt E, Xie X, van der Hoeven JES, van Blaaderen A, Van Aert S, Bals S, *ACS Nano* **13(11)** (2019) 13421.

Task 9.3: Characterization of nanoparticles

- Thermal stability of Fe@Au nanostars; importance for (optical) hyperthermal cancer treatment

- 3D investigation using heating tomography

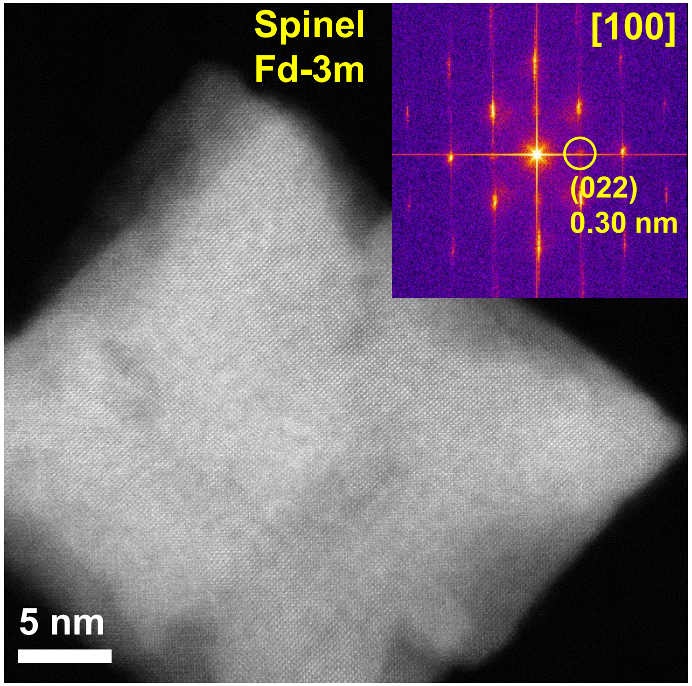


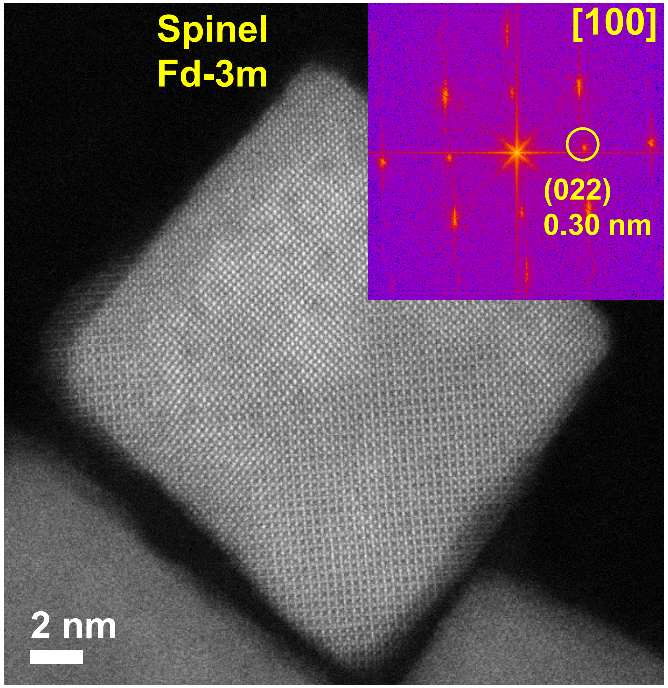
de la Encarnacion C, Lenzi E, Henriksen-Lacey M, Molina B, Jenkinson K, Herrero A, Colas L, Ramos-Cabrer P, Toro-Mendoza J, Orue I, Langer J, Bals S, Jimenez de Aberasturi D, Liz-Marzan LM, *The Journal of Physical Chemistry C* **126** (2022) 19519.

Task 9.3: Characterization of nanoparticles

EMAT

- HRSTEM investigation of monocrystalline Cobalt Ferrite ($CoFe_2O_4$) nanoparticles; importance for magnetic hyperthermia cancer treatment





Chowdhury M, Rösch E, Arenas Esteban D, Janssen K-J, Wolgast F, Ludwig F, Schilling M, Bals S, Viereck T and Lak A, Nano Letters **23** (2023) 58.

Key achievements of the WP9

Achievements	Description
Key findings	• Improved sample preparation of hybrid organic-inorganic materials, biocompatible composites and pharmaceuticals • Development of analytical techniques used for investigations of sensitive biological and bioinspired materials • Advanced use of electron tomography combined with analytical techniques and in-situ heating
Deliverables	✓ D9.1: First report on TEM methods applied to materials for health (M16 – UOXF) ✓ D9.2: Second report on TEM methods applied to materials for health (M36 – STU) ✓ D9.3: Third report on TEM methods applied to materials for health (M48 – UANTWERP) ✓ D9.4: Report on protocols for sample preparation techniques of materials for health (M52 – STU)

Workpackage 10
Materials for Transport

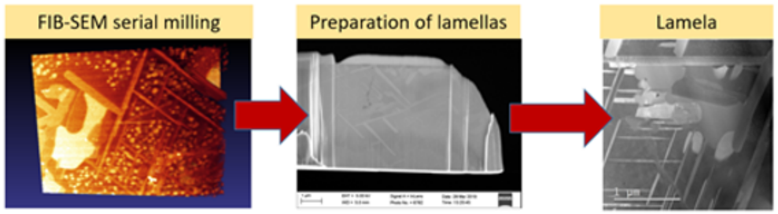
THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

WP10: Materials for Transport

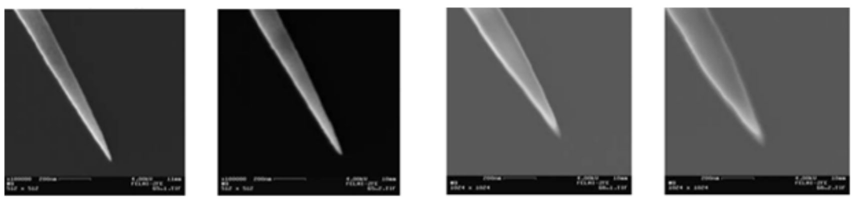
- ***Task 10.1****: Sample preparation for materials for transport (****KRA****,* ***TRO****, TOU, LJU)*
- ***Task 10.2****: Materials for Aeronautics/Aerospace (****KRA****,* ***TRO****, TOU)*
- ***Task 10.3****: Materials for Automotive Body and Chassis Structure (****TRO****,* ***KRA****, TOU)*

Task 10.1: Sample preparation for materials for transport

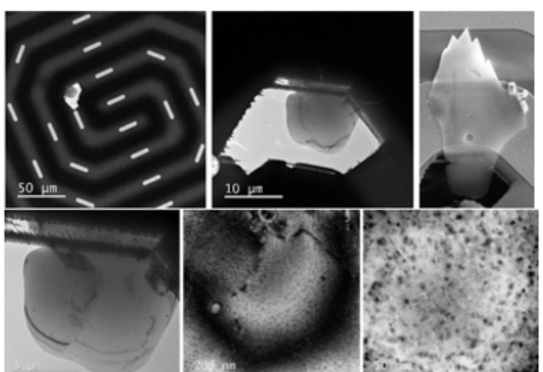
The aim of this task is the implementation of sample preparation methods for various "difficult" materials such as Ni-based superalloys, advanced high-strength steels (AHSS), Ti and Al alloys using different techniques for preparing thin artefact free TEM lamellae.



Correlative multiscale electron microscopy, 3D FIB-SEM tomography with detailed TEM investigation - targeted sample removal from analysed volume.



Sample prepared for TEM tomography from Allvac 718Plus nickel-based superalloy - *Internal cooperation with TU Graz in the frame of ESTEEM3.*

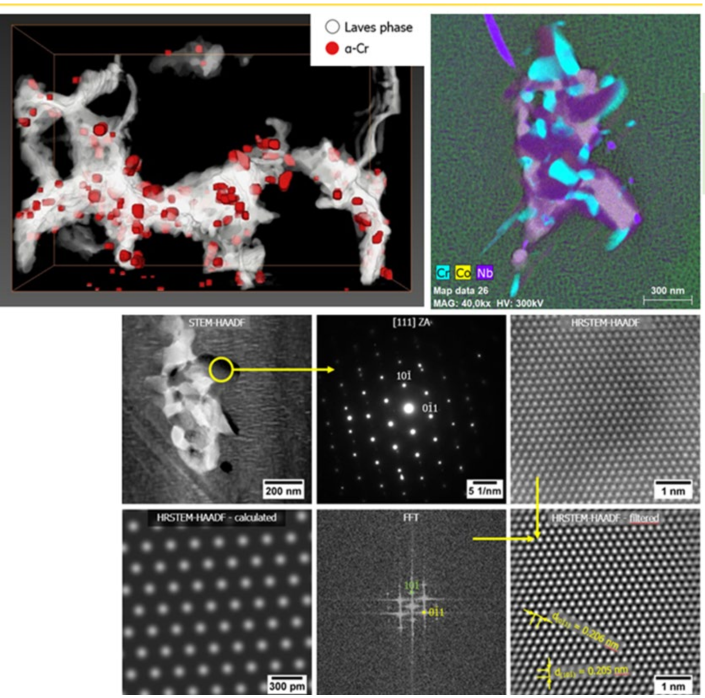


The aluminium lamellae preparation for in-situ heating in TEM. BF-TEM images and a SEM image (top right) of an aluminium lamella attached to a DENS chip for in-situ heating in TEM. Results from NTNU TRO.

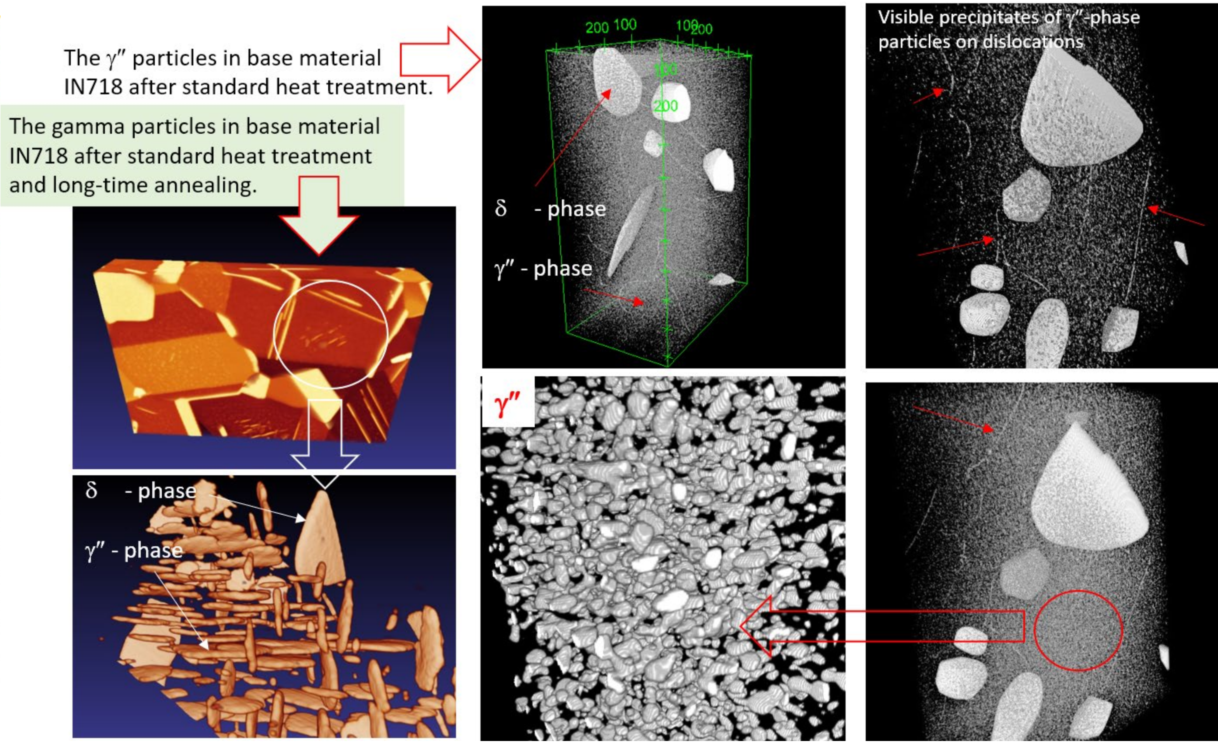
Finally, protocols for sample preparation are under preparation and will be prepared and published on-line on the ESTEEM3 website.

Task 10.2: Materials for Aeronautics/Aerospace

This task presents of the results of the application of analytical TEM, FIB-SEM tomography techniques, STEM-XEDS to 3D visualization and phases identification of structural elements in various engineering materials.



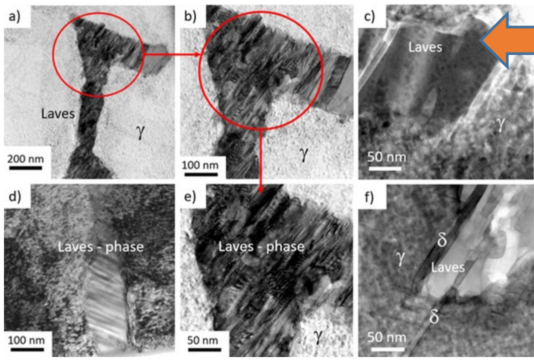
The application of 3D visualization and TEM (STEM-HAADF, SAED, STEM - EDX) techniques to characterize the morphology, spatial distribution and chemical composition of selected structural elements in the fusion zone of the EBW Inconel 718 / ALLVAC® 718PLUS welded joint.



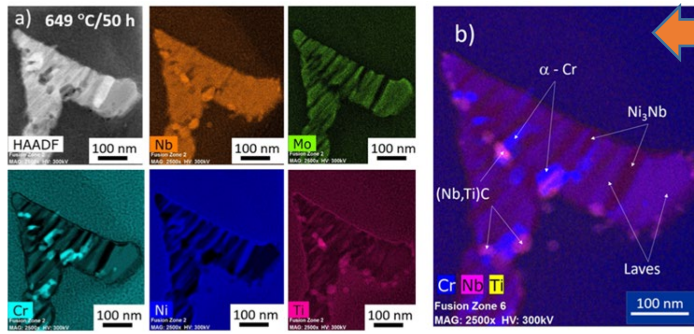
FIB-SEM Tomography from base material IN718

Task 10.2: Materials for Aeronautics/Aerospace

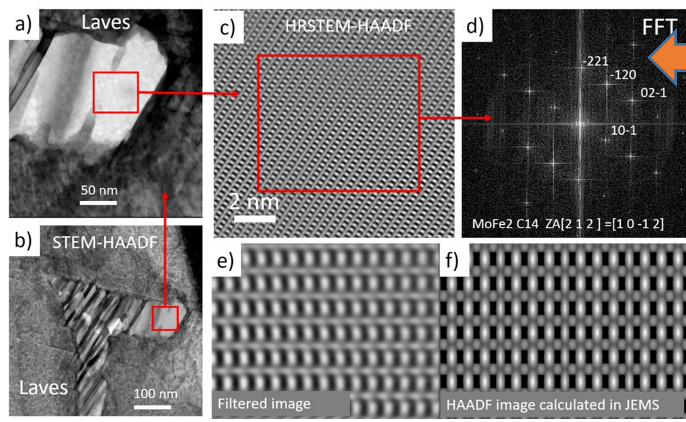
The phases stability presented in the fusion zone of the Inconel 718/ATI 718Plus during isothermal holding at a temperature of 649 °C



Decomposition of the Laves Phase in the Fusion Zone of the Inconel 718/ATI 718Plus® welded joint during isothermal holding at a temperature of 649 °C

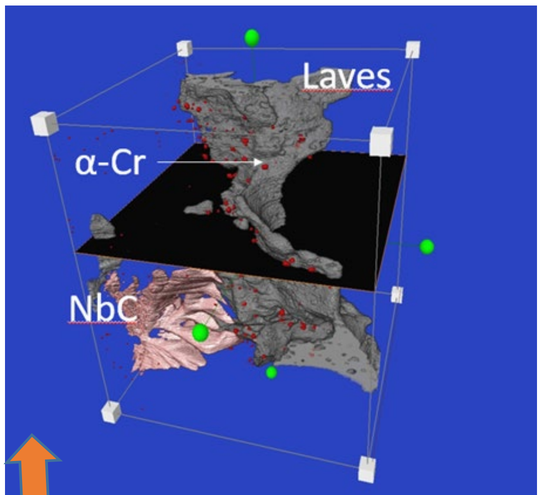


Qualitative STEM-EDX maps of the distribution of selected elements from the Laves phase area in the weld of the IN718/718Plus weld joint subjected to annealing after the welding process for 50 hours at a temperature of 649 °C.



HRSTEM analysis of the Laves phase from the weld area of the IN718/718Plus welded joint after annealing for 50 hours at a temperature of 649 °C.

Decomposition of the Laves Phase in the Fusion Zone can be written as Laves → Laves + δ + α-Cr + MC(II) secondary.

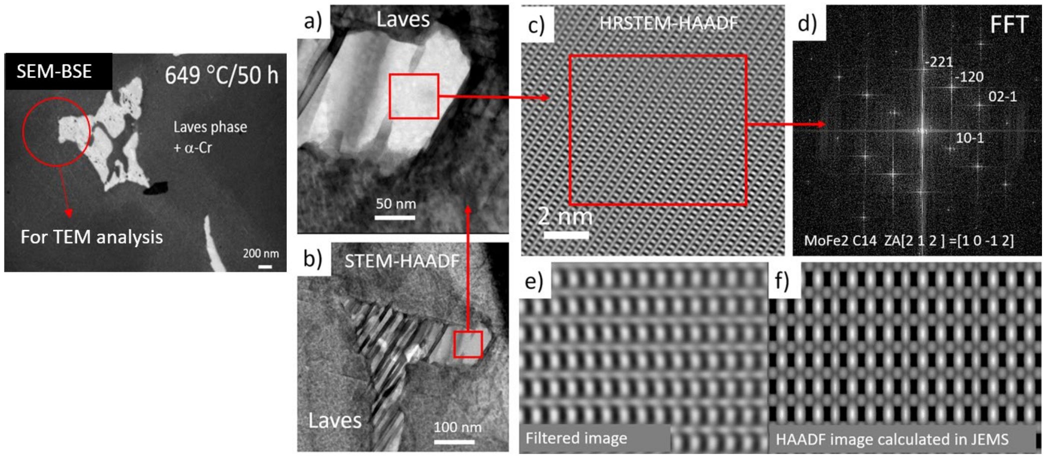


3D visualization of the tomographically reconstructed volume of 2.94 μm³ of the IN718/718Plus weld area heat-treated for 50 h at 649 °C, containing the Laves phase precipitate and primary carbides of the welded joint. The voxel size: 1.5 x 1.5 x 3 nm

Adam Kruk, Grzegorz Cempura, ***Decomposition of the Laves Phase in the Fusion Zone of the Inconel 718/ATI 718Plus® Welded Joint During Isothermal Holding at the Temperature of 649 ℃ .*** **Materials Characterization 2023, 196, 112560**

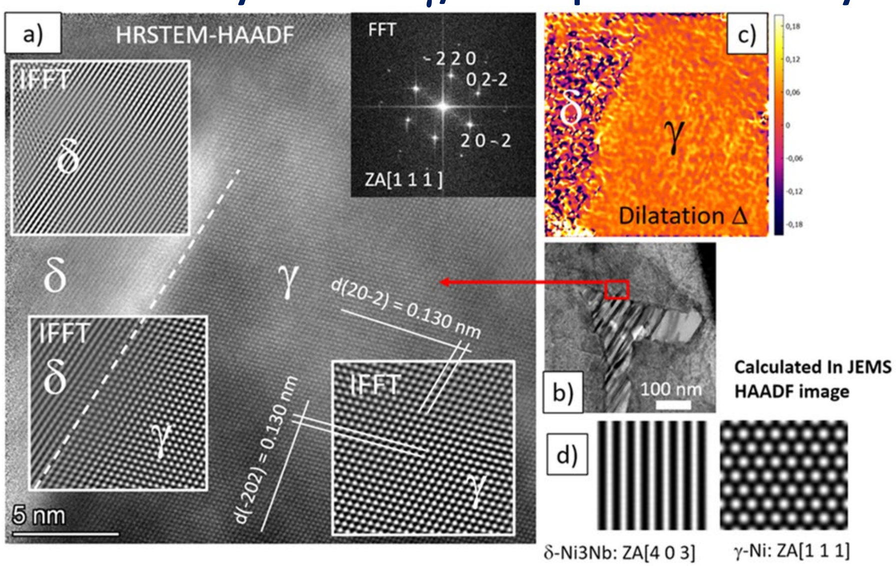
Task 10.2: Materials for Aeronautics/Aerospace

High-resolution analysis of the Laves phase decomposition



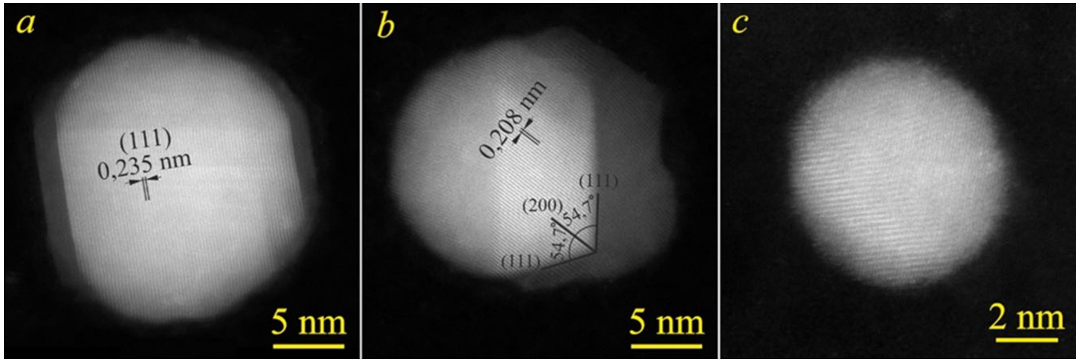
High-resolution analysis of the Laves phase from the weld area of the IN718/718Plus welded joint after annealing for 50 hours at a temperature of 649 °C. a) Laves phase particle on which high-resolution analysis was performed. b) Precipitate of the Laves phase with the area marked for further analysis. c) HRSTEM-HAADF image after digital filtering using FFT bandpass filtration. d) Image of the FFT transform with the marked reciprocal lattice vectors corresponding to the respective interplanar distance. e) Enlarged fragment of the image from c). f) Simulated HAADF image

High-resolution analysis of the γ/δ interphase boundary



High-resolution analysis of the δ phase plate fragment growing from the Laves phase and growing into the γ matrix in the fusion zone of the IN718/718Plus weld joint after annealing for 50 hours at 649 °C. a) HRSTEM-HAADF high-resolution image showing the δ / γ boundary. b) Precipitation of the Laves phase with the marked area containing the δ phase plate on which the imaging shown in Fig. 10a was performed. c) The result of geometric phase analysis calculating the dilatation between δ/γ phases in the HRSTEM image a) made in the Strain ++ software. d) The simulation of HAADF images were performed in the JEMS software for δ phase with ZA [4 0 3] and for γ phase with ZA [1 1 1], the results are presented on Fig.10d

Task 10.2: Materials for Aeronautics/Aerospace

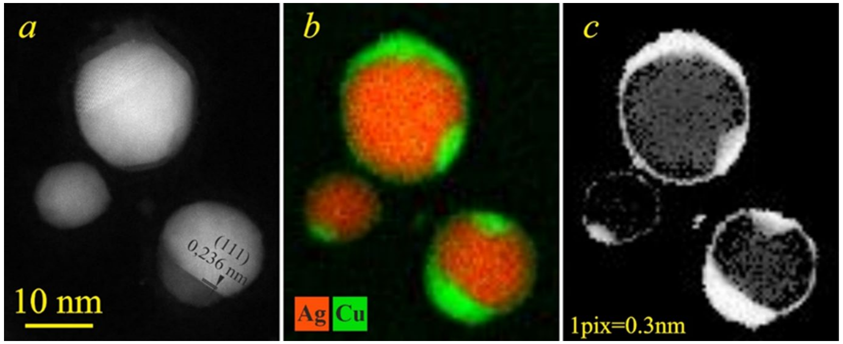


Phase separation mechanism and atomic mixing patterns in AgCu nanoparticles

High-resolution HAADF STEM images of phase-separated (a, b) and single-phase (c) Ag-Cu nanoparticles.

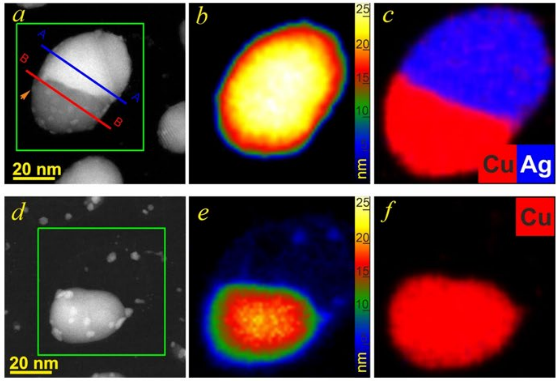
A gradual transition from Janus-like to homogeneous mixing pattern was observed in Ag-Cu nanoparticles (28 wt.%Cu) with decrease of their size.

Revealing surface segregation of Cu in Ag-rich alloy nanoparticles.

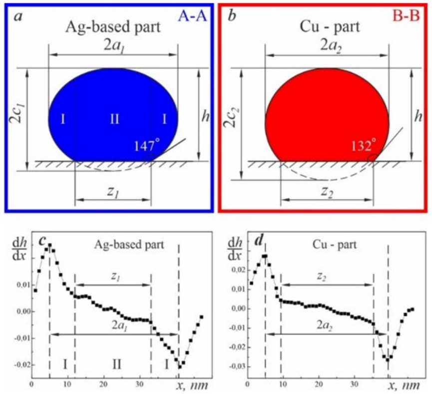


HAADF STEM image of the Ag-Cu binary nanoparticles at 300°C (a), corresponding EELS composite map of Ag and Cu (b), and Cu relative composition map (c).

Evaporation of Janus Ag-Cu nanoparticles. Kinetics and phase composition



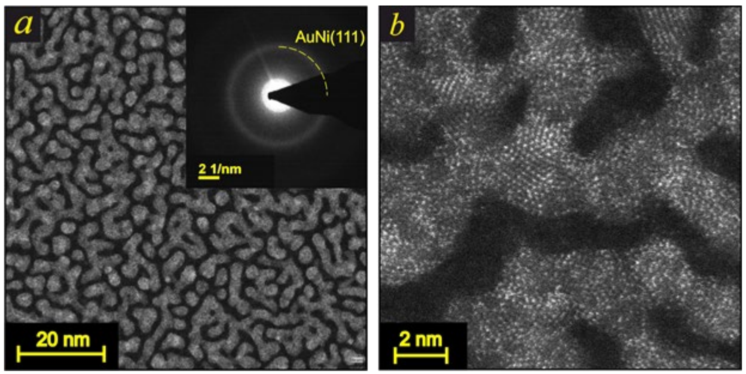
HAADF STEM images of Ag-Cu nanoparticle annealed to 600°C (a) and 750ºC (d) along with false-color thickness maps (b, e), and EDX composite maps (c, f).

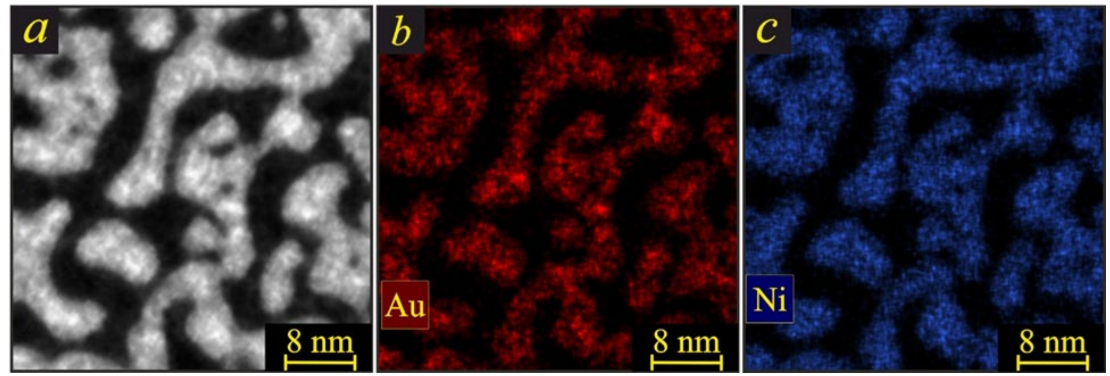


Schematic view of cross sections A-A (a) and B-B (b) of Ag and Cu domains. (c and d) are derivatives of EELS SI data along the corresponding cross-sections.

Task 10.2: Materials for Aeronautics/Aerospace

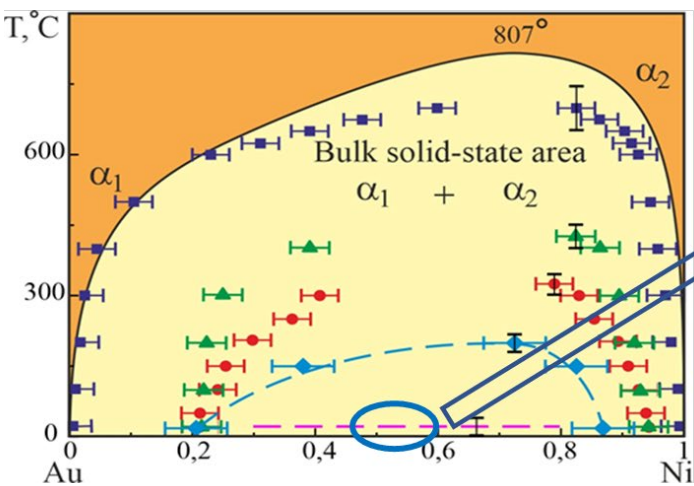
Mixing of Immiscible Components by the Size Effect: A Case Study of Au–Ni Nanostructures

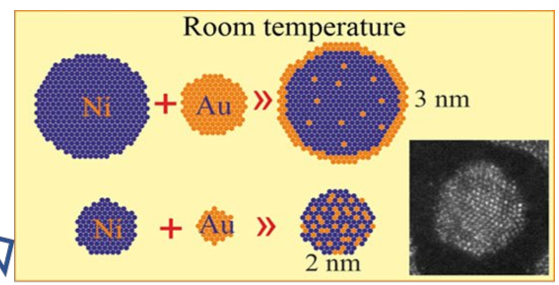




HAADF STEM image of the Au-Ni nanostructures with a size of 2 nm at room temperature and the corresponding EDX component distribution maps

HAADF-STEM image of the Au-Ni film with a thickness of 1.3 nm (overview (a) and HR view (b)) formed on a C substrate at RT. The inset in (a) shows SAED pattern.





Size evolution of the solid-state miscibility gap in Au-Ni phase diagram.

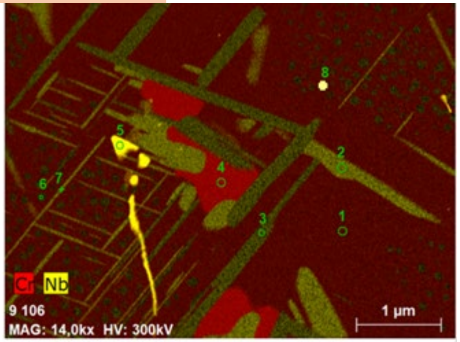
■ – 140 nm, ▲ – 12 nm,
● – 6 nm,
♦ – 3 nm,
▬ - 2 nm

- The curves limiting the solid-state miscibility gap for nm-sized Au-Ni particles was experimentally constructed in the whole concentration range and at all temperatures.
- It was proved the existence of the critical size for the Au-Ni system below which the phase separation does not occur.

Task 10.2: Materials for Aeronautics/Aerospace

- Round robin test (AGH-TRO) for determination of chemical composition of different phases present in investigated area of weld metal.
- Application of STEM – EDX for detailed characterization of the chemical composition of structural elements in fusion zone EBW Inconel 718/ALLVAC® 718PLUS welded joint.

Results from AGH-UST



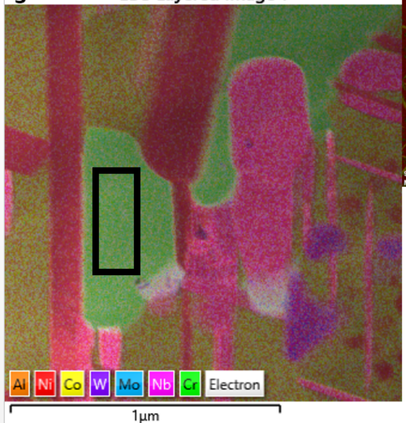
Averaged chemical composition of individual phases.

Atomic percent (%)

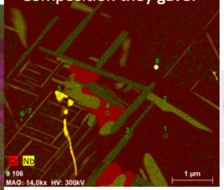
	Cr-KA	Ti-KA	Fe-KA	Co-KA	Ni-KA	Nb-KA	Mo-KA	W-LA	Al-K	
δ	**1.35** 0.63	**1.57** 0.18	**1.82** 0.44	**3.99** 0.26	**67.13** 1.07	**19.15** 0.62	**2.27** 0.35	**0.32** 0.10	**2.40** 0.53	Ni, Nb, Ti
η	**0.77** 0.07	**4.05** 0.26	**1.84** 0.19	**3.57** 0.11	**65.82** 0.59	**11.46** 0.91	**0.75** 0.17	**0.27** 0.08	**11.49** 0.56	Ni, Nb, Al., Ti
σ	**46.14** 0.50	**0.15** 0.05	**12.86** 0.16	**7.61** 0.21	**18.47** 0.34	**0.54** 0.11	**10.93** 0.21	**0.98** 0.08	**2.33** 0.26	Cr, Mo, Co
γ	**23.65** 0.20	**0.25** 0.03	**16.80** 0.07	**6.93** 0.09	**47.00** 0.27	**0.90** 0.05	**1.80** 0.09	**0.30** 0.02	**2.38** 0.04	Cr, Fe, Co, Ni

Results from TRO

3 EDS Layered Image 1



Composition they gave:



Sigma phase:

Point No.	Predicted phase	Chemical composition obtained during STEM-EDX analysis using Titan G3 60-300 TEM, wt%								
		Ni	Cr	Fe	Co	Nb	Mo	Al	W	Ti
4	sigma phase	18.5	40.7	11.8	7.4	0.9	17.0	0.9	2.7	0.1

Data analysis done at NTNU:

Day 1

Elements			Atomic percentage
Al	00,30	Al	0,12
Ti	0,09	Ti	0,16
Cr	50,12	Cr	50,62
Fe	12,61	Fe	12,92
Co	7,67	Co	7,43
Ni	17,76	Ni	17,32
Nb	0,46	Nb	0,12
Mo	10,09	Mo	10,53
W	0,91	W	0,78
Total	100,00	Total	100,00

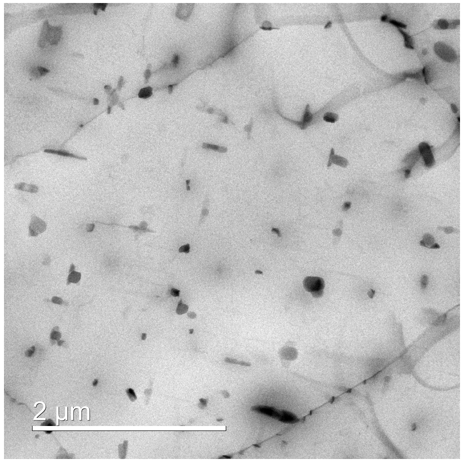
Day 2

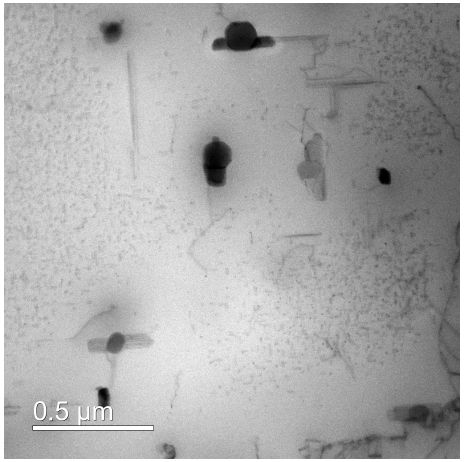
Spectrum 1	wt%	Atomic %
Al	0,19	0,43
Ti	0,06	0,07
Cr	42,46	49,29
Fe	11,93	12,9
Co	7,45	7,63
Ni	17,03	17,51
Nb	0,56	0,37
Mo	17,13	10,78
W	3,18	1,04
Total	100	100

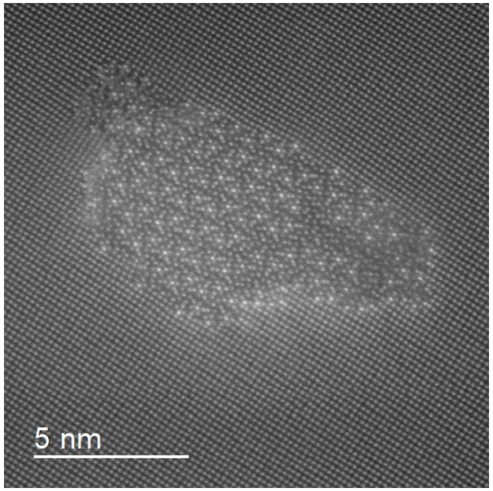
From .cif file:_chemical_formula_sum 'Cr9.32 Fe15.52 Mn0.37 Mo2.21 Ni2.6'

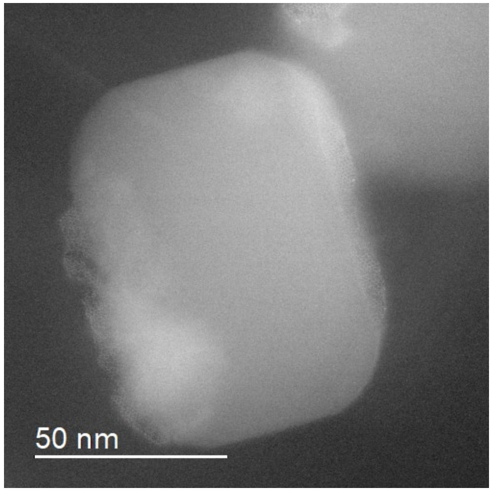
Objective of the study

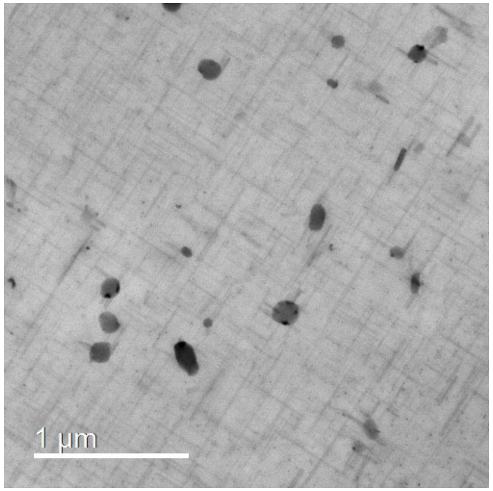
- Qualitative estimation of hardening and non-hardening precipitates in the alloys under different heat-treatment conditions by TEM
- Structural characterization of the precipitates by analyzing atomic resolution images
- Understanding the starting point of nucleation of the secondary precipitates
- Statistical quantification of the volume fraction of the secondary precipitates by analyzing FIB SEM tomography data using particle segmentation method



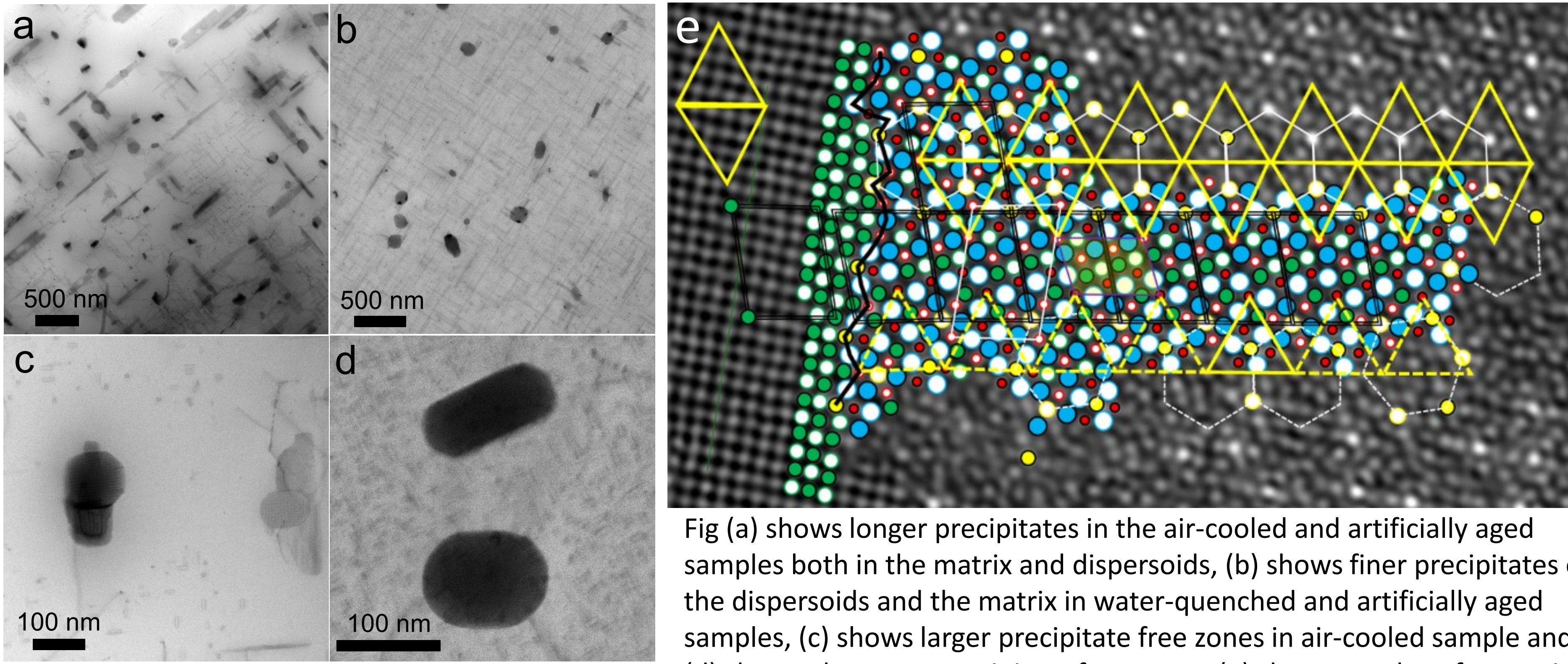








WP10: Heterogeneous Precipitation on α-dispersoids in Al-Mg-Si-Cu alloy at different heat treatments (TRD, KRA)

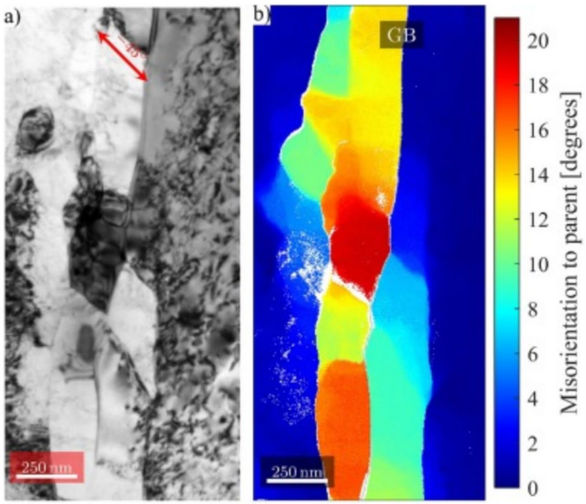


Chatterjee et al. to be submitted

Fig (a) shows longer precipitates in the air-cooled and artificially aged samples both in the matrix and dispersoids, (b) shows finer precipitates on the dispersoids and the matrix in water-quenched and artificially aged samples, (c) shows larger precipitate free zones in air-cooled sample and (d) shows almost no precipitate free zones, (e) shows overlay of atoms in a defective secondary Q' precipitate

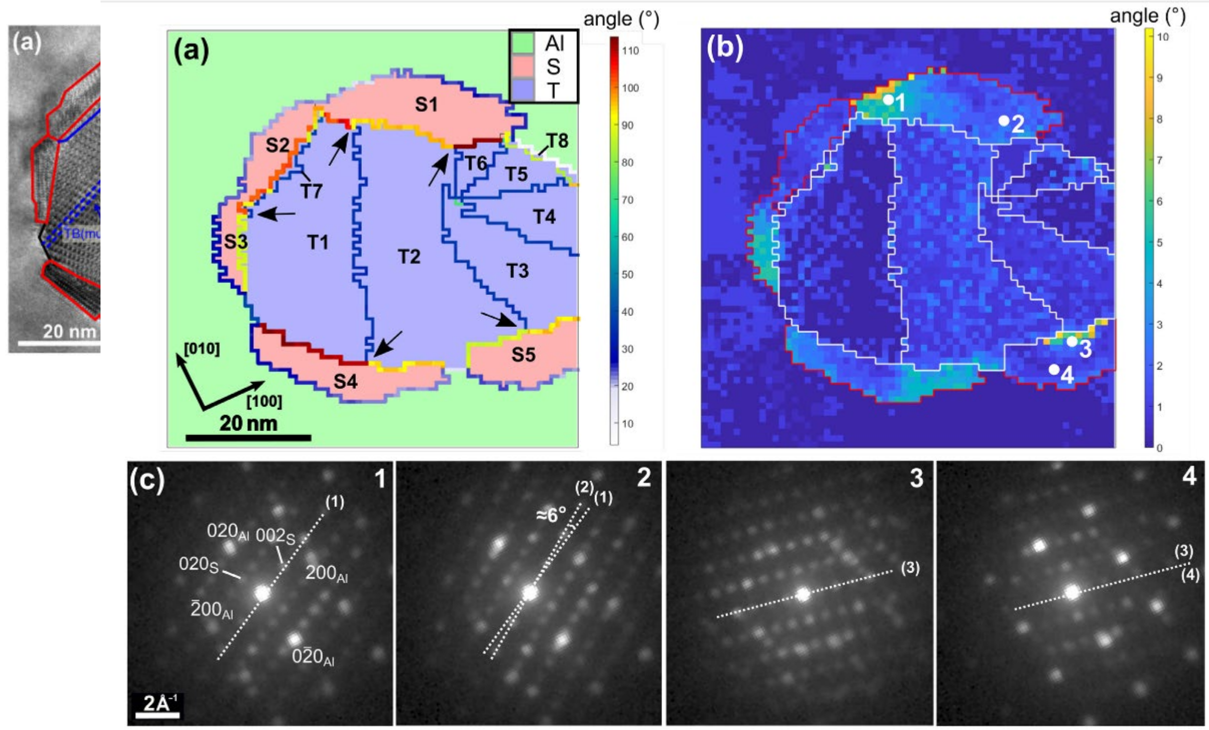
- Comparison of secondary precipitation in the air-cooled and water-quenched samples has been done
- Elucidation of the crystal structure of the defected precipitates is being carried out

Orientation mapping



Misorientations in precipitate free zones of a compressed Al-Mg-Si alloy

Orientation mapping and misorientation analysis of T-/S-phase aggregates



Christiansen et al. Mat. Char. ***144*** *(2018) 522.*

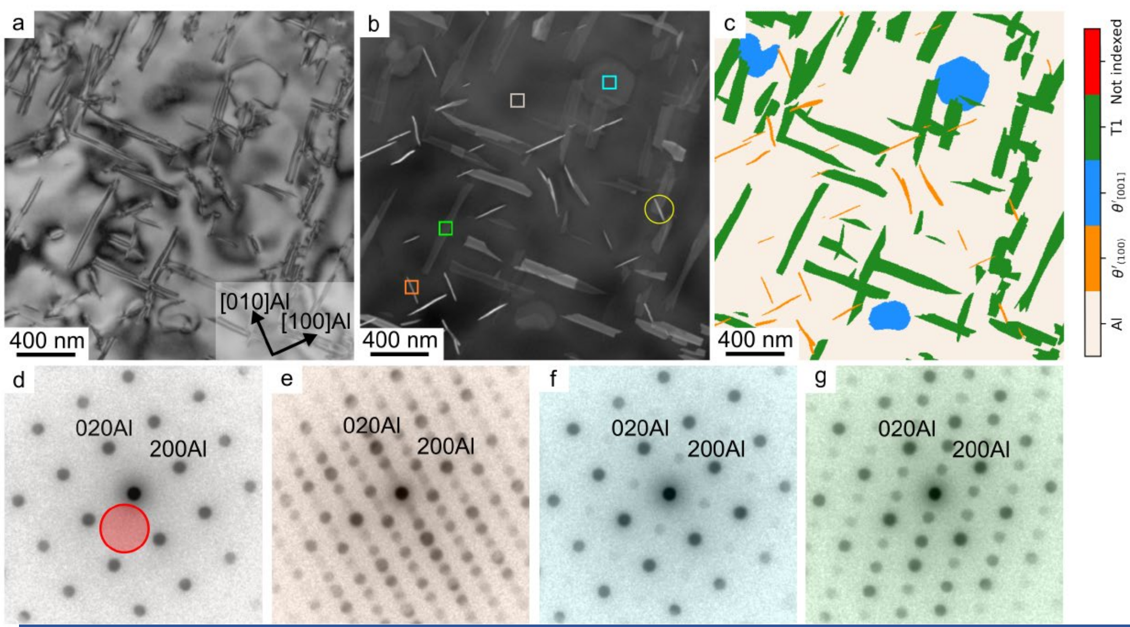
Sunde et al. Acta Mater. ***166*** *(2019) 587-596.*

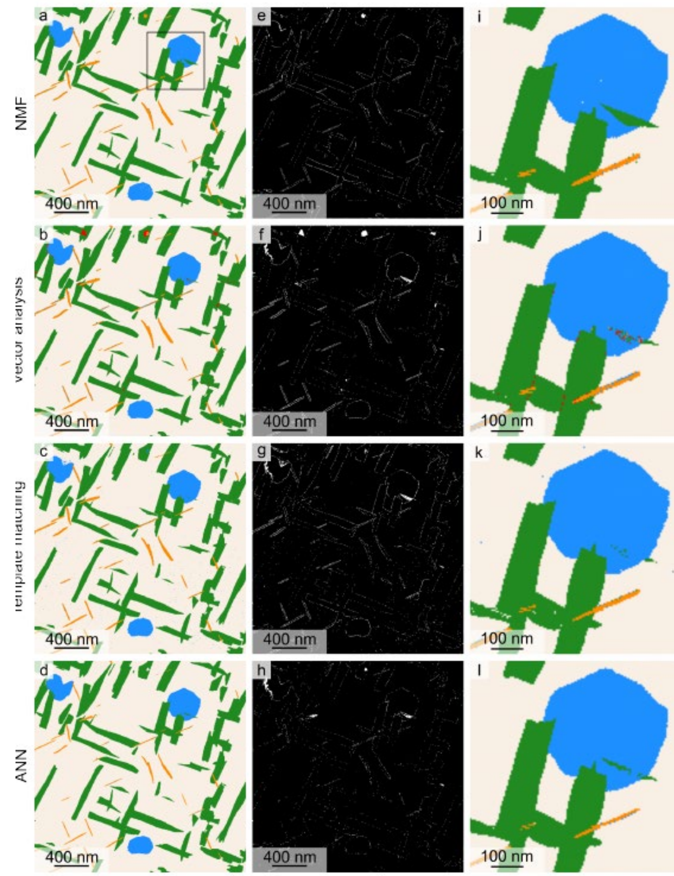
WP10: Analysis of SPED data for phase mapping of precipitates in aluminium alloys (TRD, CAM)

- Scanning precession electron diffraction (SPED) was used to study embedded precipitates in an Al-Cu-Li alloy.
- Four different data analysis approaches for phase mapping were applied on the dataset, containing three different phases.
- These were non-negative matrix factorization (NMF), template matching, vector analysis and artificial neural networks (ANN).
- Resulting phase maps were compared with the ground truth image created from virtual images of the dataset to estimate the accuracy.
- The accuracy was satisfactory and comparable for all approaches.

pyxem

HyperSpy
multi-dimensional data analysis

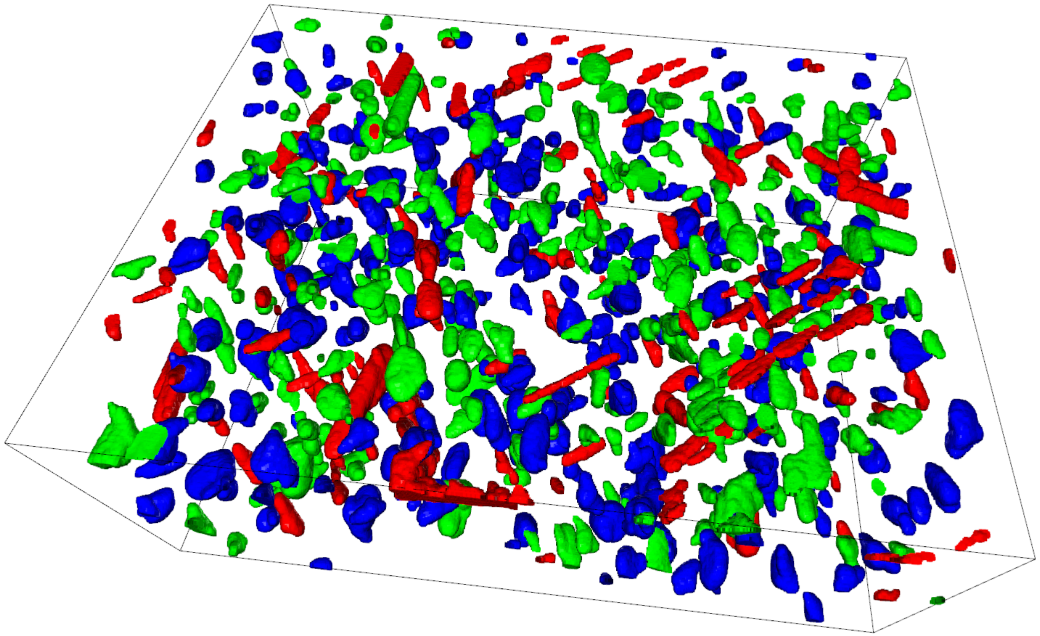




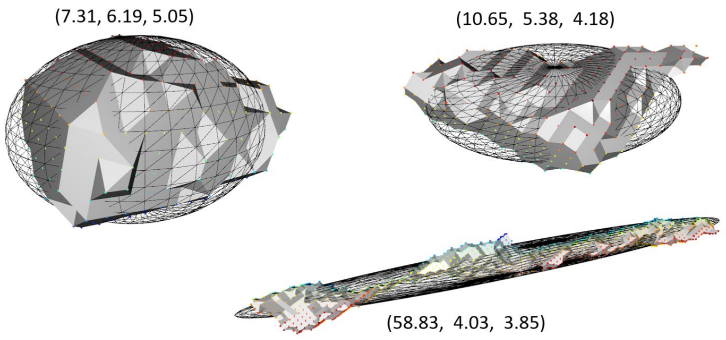
Thronsen, Bergh, Thorsen, Christiansen, Frafjord, Crout, van Helvoort, Midgley & Holmestad; to be submitted

Task 10.3: Materials for Automotive Body and Chassis Structure

Quantification of precipitates in the matrix and on the dispersoids by analysis of FIB SEM tomography data by particle segmentation analysis in air cooled sample, aged for 30 mins



Particle type	Count	Volume fraction [%]
Precipitates (red)	132	0.42
Dispersoids (blue)	278	1.00
Disc shaped	277	1.55



Particles have been classified in terms of the shape:
Further refinement necessary to distinguish the precipitates growing on the dispersoids.

WP10: Materials for Transport

Main achievements

- Procedures for phase identification and microstructural characterization of innovative structural & functional materials for the aerospace and automotive industry.
- Procedures of specimen preparation appropriate for electron microscopy investigations from the above-mentioned materials. We suggest combining FIB-SEM tomographic acquisition of images, used for 3D imaging, with lamella cutting. Such a procedure results in a **Targeted Sample Preparation** (TSP).
- Microstructural characterization of joints in materials for automotive body and chassis structures.
- Implementation of **in-situ straining and heating** (WP6) combined with automated orientation mapping.
- High-resolution analysis of interphase boundary between strengthening particles and matrix in nickel-base superalloys.
- **In-situ TEM investigation** of strengthening particles stability at high-temperatures in nickel-based superalloys.
- High-resolution analysis of strengthening particles in aluminum alloys.

Workpackage 11
Data and Automation

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

Objectives

Within this WP we will develop strategies and capabilities for big data, open data, open science and automation of workflows in TEM for the benefit of the wider user community.

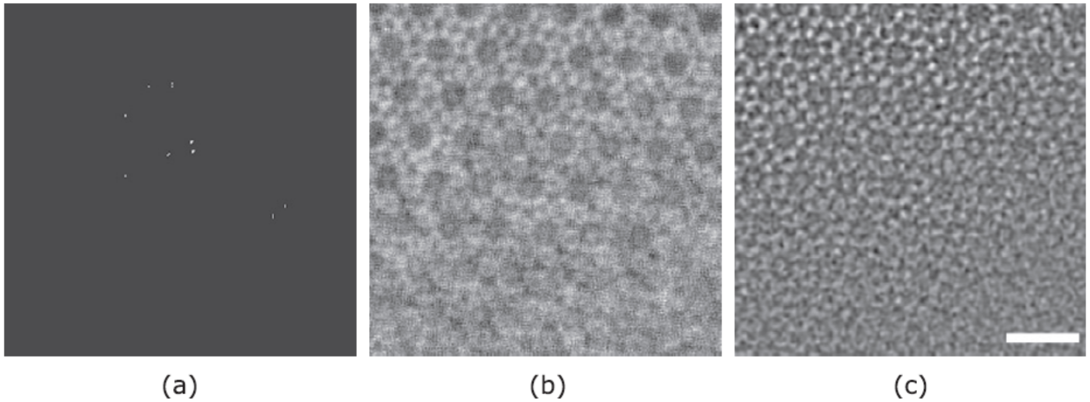
1. The development of optimised hardware and software for handling high data rates and volumes
2. The establishment of automated and smart workflows for optimised instrument control, data acquisition, data processing and machine learning with reduced human intervention
3. The development of software for the design and interpretation of experiments

WP11 highlights UA

- Real time ptychography and ICOM reconstruction: open source: https://github.com/ThFriedrich/riCOM_cpp
- CNN based distortion correction in images: open source: https://github.com/Ivanlh20/r_em
- Advanced model based EELS library: pyEELSMODEL, open source release soon
- Advanced CPU/GPU multislice Multem: open source https://github.com/Ivanlh20/multem
- Open source library of EELS/EDX cross sections: https://zenodo.org/record/6599071#.ZF4AoM5ByUk
- Python electrostatic simulation code

WP11 highlights

- OXF: Low-dose binary ptychography:
  https://doi.org/10.1063/1.5143213



a) Example CBED pattern sequence, (b) iCoM reconstruction, and (c) SSB reconstruction for ZSM-5 using a cumulative electron dose of 200 eA˚ 2. The scale bar for the phase reconstructions is 2 nm.

WP11 highlights

TRO, CAM: HyperSpy-based packages

pyxem 0.15.1 documentation

Search

USER GUIDE

- Importing MIB Datasets
- Data Inspection- Preprocessing - Unsupervised ML
- Phase/Orientation Mapping
- Calibrations
- Introduction
- Contents
- Nanocrystal Segmentation
- Azimuthal Integral
- PDF Analysis Tutorial
- Angular Correlations of Amorphous Materials
- Analysing magnetic materials using STEM-DPC

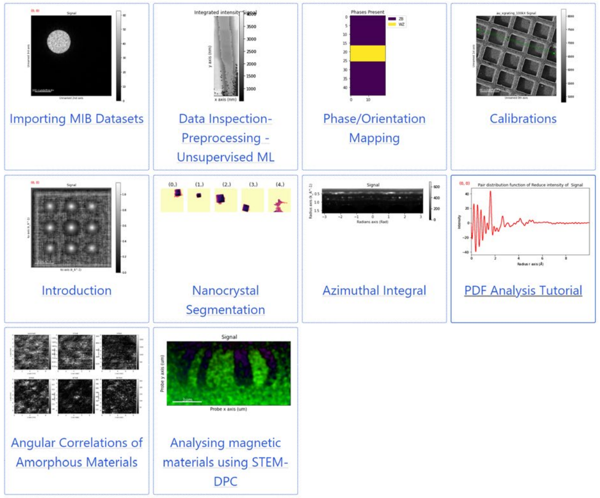
HELP

- API reference
- Bibliography

v: stable

User Guide and Workflows

USER GUIDE



diffsims

build passing | coverage 100% | docs passing | pypi v0.5.2 | code style black | DOI 10.5281/zenodo.3337900

diffsims is an open-source Python library for simulating diffraction.

If simulations performed using diffsims form a part of published work please cite the DOI at the top of this page. You can find demos in the diffsims-demos repository. See the documentation for installation instructions, the API reference, the changelog and more.

diffsims is released under the GPL v3 license.

Welcome to LumiSpy's documentation!

Azure Pipelines succeeded | Tests passing | codecov 100% | CodeQL passing | python 3.7 | 3.8 | 3.9 | 3.10 | pypi v0.2.2 | Anaconda.org 0.2.2 | License GPLv3 | DOI 10.5281/zenodo.4640445 | docs passing

LumiSpy is a Python package extending the functionality for multi-dimensional data analysis provided by the HyperSpy library. It is aimed at helping with the analysis of luminescence spectroscopy data (cathodoluminescence, photoluminescence, electroluminescence, Raman, SNOM).

Check out the Installation section for further information, including how to start using this project.

Complementing this documentation, the LumiSpy Demos repository contains curated Jupyter notebooks to provide tutorials and exemplary workflows.

orix | User guide | API reference | Contributing | Changelog

Section Navigation

Getting started

- Installation

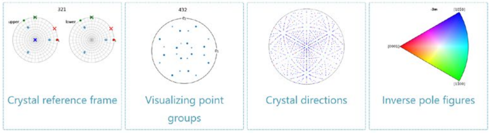
Usage

- Tutorials
  - Crystal reference frame
  - Visualizing point groups
  - Crystal directions
  - Inverse pole figures
  - Uniform sampling of orientation space
  - Stereographic projection
  - Sampling unit vectors
  - Visualizing Crystal Poles in the Pole Density Function
  - Clustering across fundamental region boundaries
  - Clustering orientations
  - Clustering misorientations

Read The Docs | v: stable

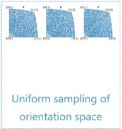
Crystal geometry

These tutorials cover conventions for the unit cell, symmetry operations and relevant reference frames, and also show how to operate with vectors in the crystal and sample reference frames.



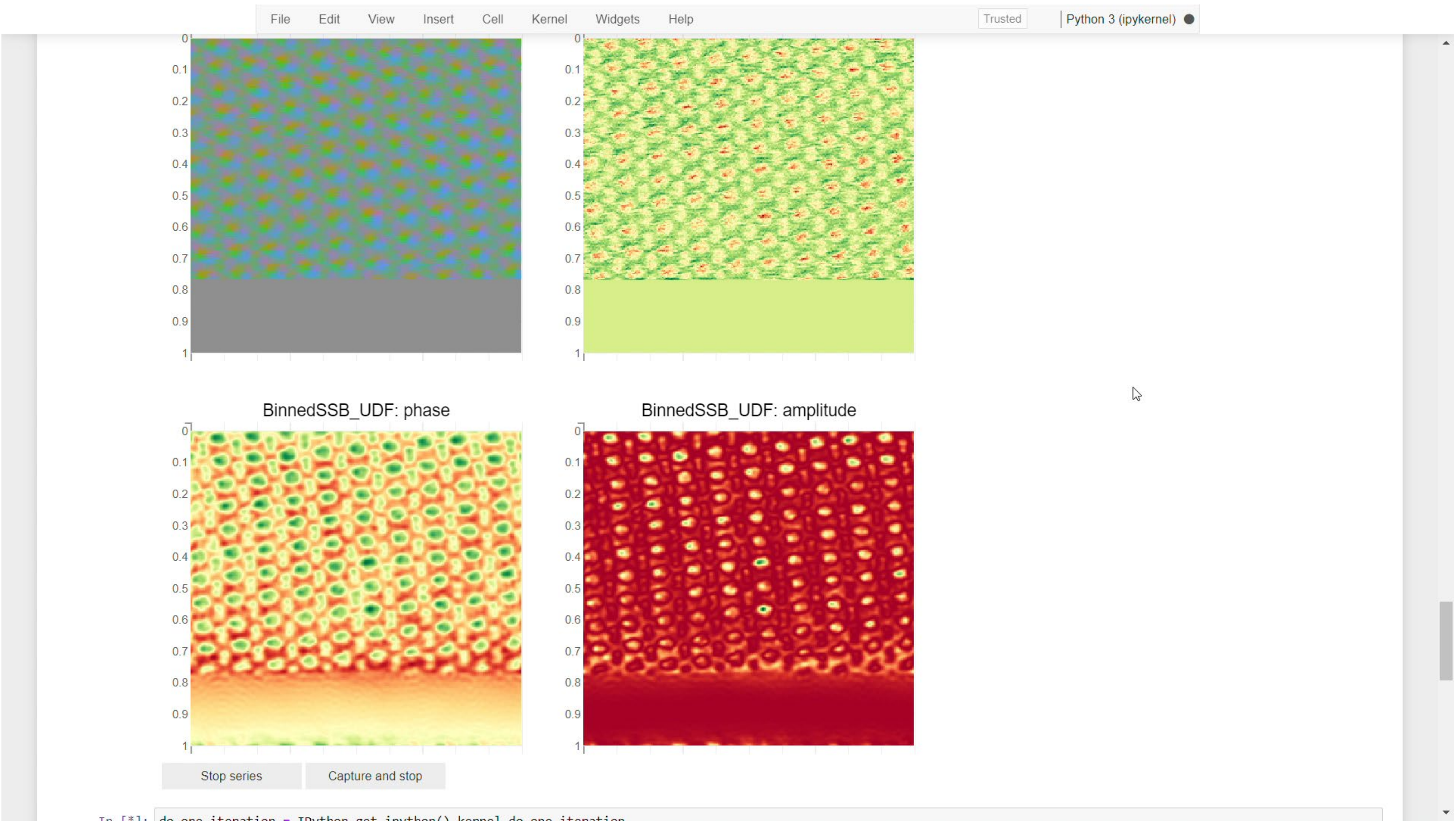
Orientations

This tutorial covers how to sample orientation space of the proper point groups.



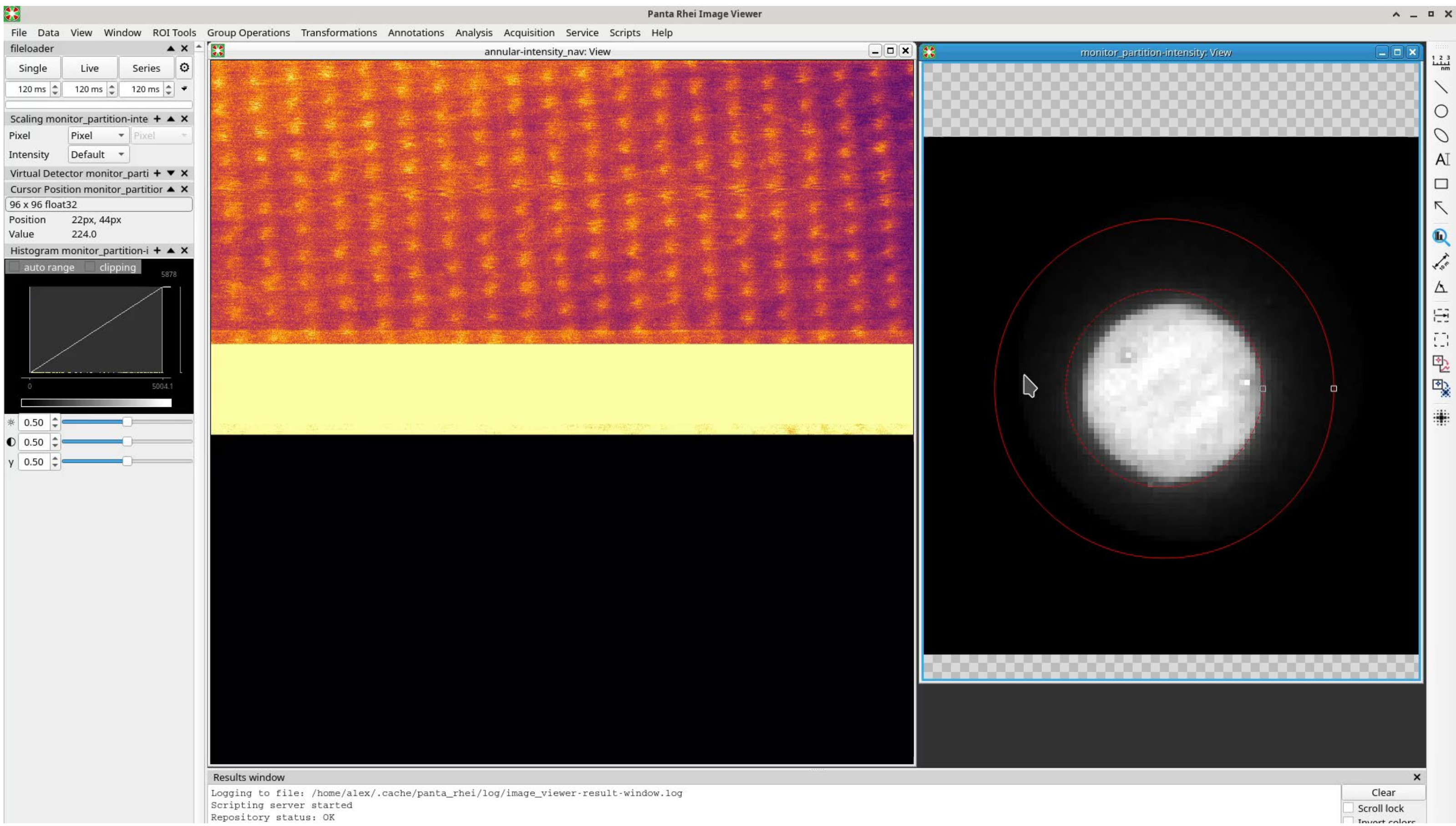
WP11 highlights

PSI, Ptychography 4.0, JUL:
Fast live ptychography



WP11 highlights

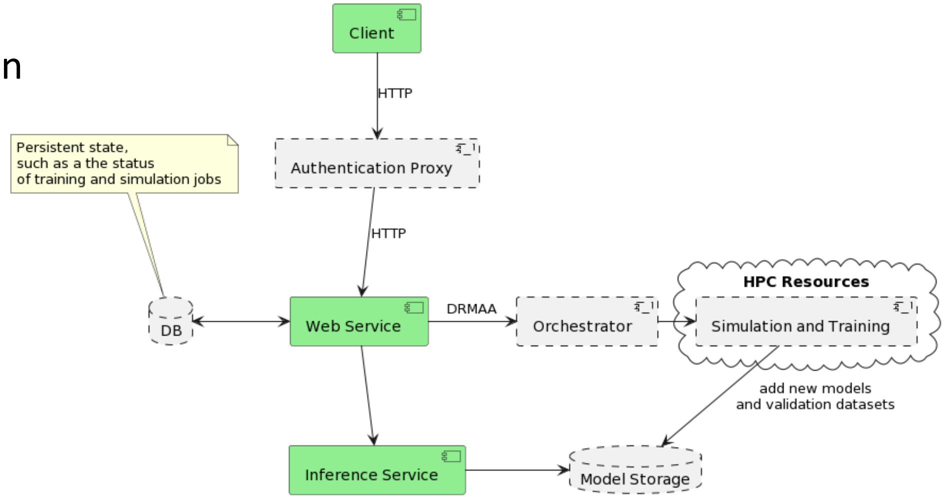
CEOS, JUL: Fast interactive live processing with LiberTEM and PantaRhei

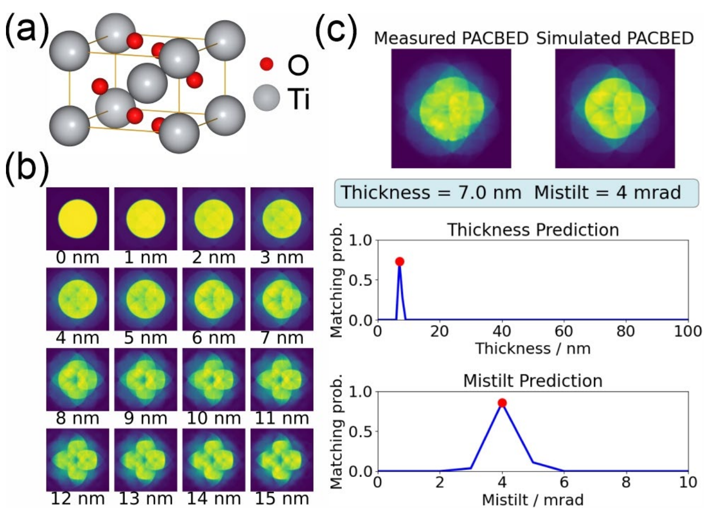


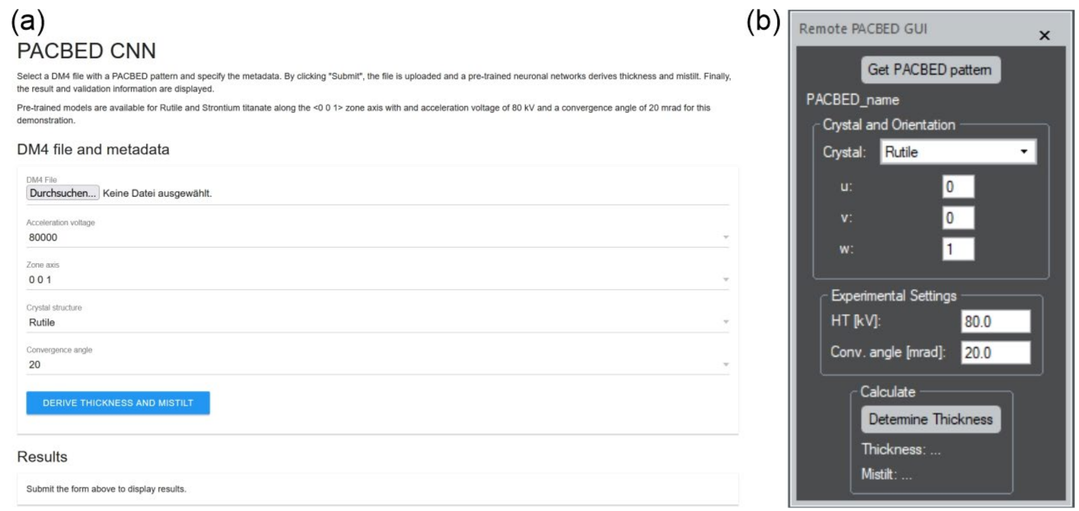
WP11 highlights

Online Thickness Determination with Position Averaged Convergent Beam Electron Diffraction using Convolutional Neural Networks (Graz, JUL)

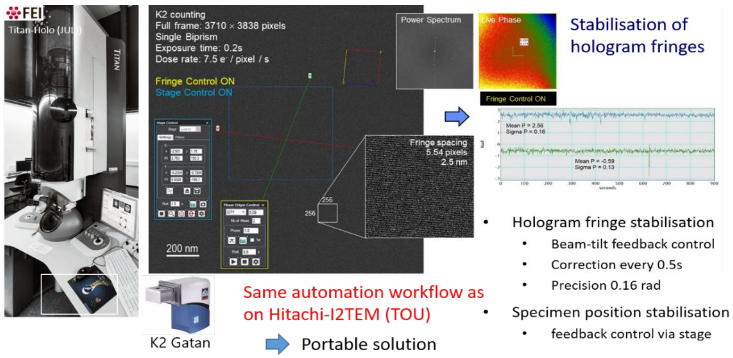
Michael Oberaigner et al.,
https://doi.org/10.1093/micmic/ozac050
https://github.com/MichaelO1993/PACBED-CNN



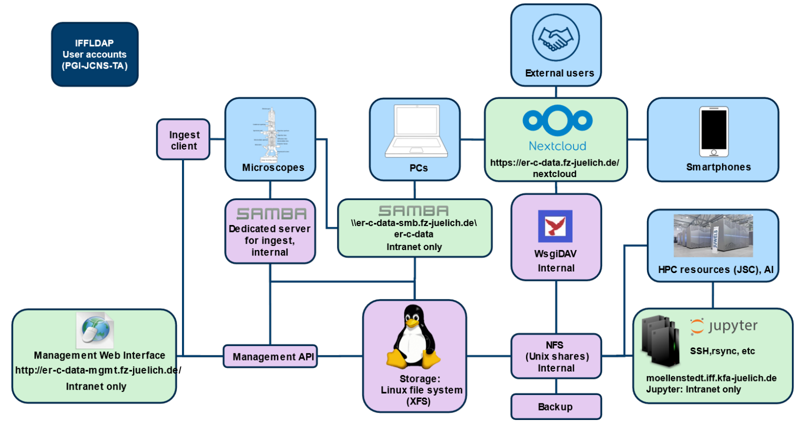




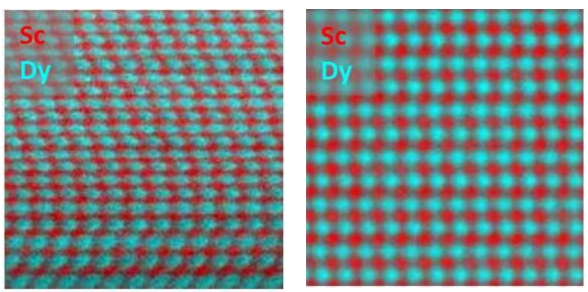
WP11 highlights



TOU: Hologram stabilization



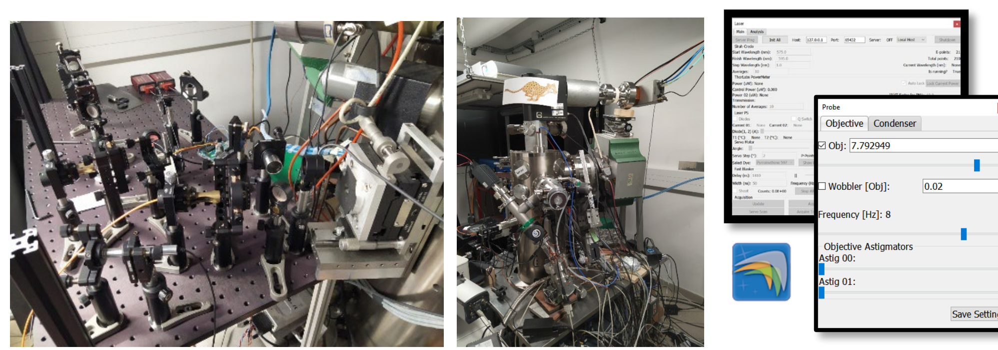
JUL: Data management



1: conventional acquisition

2: acquisition with drift correction

ORS: Real-time EELS drift correction



ORS: Electron energy gain spectroscopy controlled with Nion Swift, VG Lumière, 2021.

COFFEE BREAK

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

ESTEEM3
NETWORKING ACTIVITIES
ACHIEVEMENTS: WP1,WP2,WP3

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NA Work Packages

WP No	Work Package Title	Lead Participant No	Lead Participant Short Name	Person-Months	Start Month	End month
1	Integration and Sustainability	1	STU	**60**	1	48
2	Education and Training	8	LJU	**31**	1	48
3	Outreach	1	STU	**69**	1	48
4	Imaging, Diffraction and Metrology	5	OXF	**86**	1	48
5	Spectroscopy	3	CNRS (ORS)	**77**	1	48
6	In-situ TEM	3	CNRS (TOU)	**50**	1	48
7	Materials for ICT	7	GRA	**64**	1	48
8	Materials for Energy	10	CAD	**46**	1	48
9	Materials for Health	1	STU	**33**	1	48
10	Materials for Transport	11	KRA	**28**	1	48
11	Data and Automation	2	JUL	**44**	1	48
12	Transnational-Access	1	STU	**200**	1	48
13	Management of the Project	1	STU	**30**	1	48
				818		

NA Work Packages

Networking activities KPIs

Activity	WP N°	Category	Target	Status – April. 2023
NA	1-3	ESTEEM3 staff trained through staff exchange	50	31
		Schools & workshops	**11**	**14**
		Participants at schools/workshops	**600**	
		Other educational & training events including presentations to industrial users - Schools, webinars, seminars	**25**	**83**

WP1: Integration and Sustainability

Work package number	1	Lead beneficiary				OXF, STU
Work package title	Integration and Sustainability					
Participant number	*1*	*2*	*3a*	*3b*	*4*	*5*	*6*
Short name of participant	STU	JUL	TOU	ORS	ANT	OXF	CAM
Person months per participant:	6	3	4	3	4	4	4
Participant number	*7*	*8*	*9*	*10*	*11*	*12*	*13*
Short name of participant	LJU	GRA	ZAR	CAD	KRA	CHA	TRO
Person months per participant:	2	1	2	2	1	2	2
Participant number	*14*	*15*	*16*	*17*	*18*	*19*	*20*
Short name of participant	CAT	ATTO	CEOS	DENS	NM	QD	EUR
Person months per participant:	2	2	2	2	2	2	8
Start month	1			End month	48		

Objectives

1. To increase the quality and integration of the TA service provided
2. Set the basis for a legal structure for a European Electron Microscopy Research Infrastructure
3. To define an innovation and impact strategy for advanced TEM

WP1: Integration and Sustainability

Task 1.1: Integration of access procedures and quality of service (led by STU– involvement of all other partners)

Task 1.1a: Management of the Transnational Access Activity (led by **STU**, **EUR**)

Task 1.1b: Staff development (led by **CHA** and **CAT** – participation of all other partners)

Task 1.2: Road mapping (OXF, ORS with participation of JUL, TOU, ZAR, TRO, ANT, LJU, STU)

Task 1.2a: Towards an ESTEEM legal structure

Task 1.2b: Sustainability

Task 1.3: Foresight studies for TEM (JUL, ANT, TOU, CAM, ORS, OXF, ATT, CEOS, DENS, NM, QD, EUR)

Task 1.4: Improving access to the ESTEEM3 infrastructure through standardized data and software approaches in an open data and open science context (TOU, JUL, CAM)

WP1: Integration and Sustainability

Task 1.1: Integration of access procedures and quality of service (led by STU– involvement of all other partners)

Task 1.1a: Management of the Transnational Access Activity (led by STU, EUR)

- **Coordination of TNA projects (TNA presentation)**

Task 1.1b: Staff development (led by CHA and CAT – participation of all other partners)

The online training activities include 31 online training activities of which there are 4 training sessions and 27 webinars.

D1.1a Manual describing best practice for Transnational Access (STU)
D1.1b Report on staff development (CHA)

WP1: Integration and Sustainability

Task 1.2: Road mapping (OXF, ORS with participation of JUL, TOU, ZAR, TRO, ANT, LJU, STU)

Task 1.2a: Towards an ESTEEM legal structure

Well identified by other ARIE RIs, and by the EC (including ESFRI), e-DREAM aims to bring together all TEM infrastructures of the Member States in European initiatives aimed at promoting the creation in the near future of a European RI in Electron Microscopy.

Task 1.2b: Sustainability

- **Presentation on sustainability pending**

D1.2a: Report on a possible legal structure (TOU, 52)
D1.2b: Report on the joint actions with national EM networks (ORS, 46)
D1.2c: Report on possible business model for sustainable funding (ANT, 46)

WP1: Integration and Sustainability

Task 1.3: Foresight studies for TEM (JUL, ANT, TOU, CAM, ORS, OXF, ATT, CEOS, DENS, NM, QD, EUR)

- **Deliverables submitted**

Task 1.4: Improving access to the ESTEEM3 infrastructure through standardized data and software approaches in an open data and open science context (TOU, JUL, CAM)

The positive developments for software and data in electron microscopy in an open science and open data context continue.

D1.3: Innovation strategy white paper (OXF, 52)
D1.4b: Final report on a data and software strategy for TEM (JUL, 52)

NA Work Packages

WP No	Work Package Title	Lead Participant No	Lead Participant Short Name	Person-Months	Start Month	End month
1	Integration and Sustainability	1	STU	**60**	1	48
2	Education and Training	8	LJU	**31**	1	48
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12	Transnational-Access	1	STU	**200**	1	48
13	Management of the Project	1	STU	**30**	1	48
				818		

WP2: Education and Training

Work package number	2	Lead beneficiary			LJU, GRA	
Work package title	Education and Training						
Participant number	*1*	*2*	*3a*	*3b*	*4*	*5*	*6*
Short name of participant	STU	JUL	TOU	ORS	ANT	OXF	CAM
Person months per participant:	2	2	2	2	3		
Participant number	*7*	*8*	*9*	*10*	*11*	*12*	*13*
Short name of participant	LJU	GRA	ZAR	CAD	KRA	CHA	TRO
Person months per participant:	4	4		3	2	3	2
Participant number	*14*	*15*	*16*	*17*	*18*	*19*	*20*
Short name of participant	CAT	ATTO	CEOS	DENS	NM	QD	EUR
Person months per participant:	2	*	*	*	*	*	
Start month	1			End	48		

Objectives

This activity is dedicated to the education and training in advanced TEM techniques. Important objectives of the work will be:

1. Organising schools and workshops in order to transfer knowledge about TEM techniques to the scientific community with an emphasis on attracting scientists in the early stages of their careers
2. Organising webinars for specific and emerging TEM techniques
3. Providing webcasts related to TEM via the ESTEEM3 website to a broader scientific community

Task 2.1.: Schools and Workshops

ESTEEM3 Schools and Workshops

School/workshop name	Date	Participants
TEM and STEM Imaging		
EMAT workshop on transmission electron microscopy (ANT)	June 11 to 21, 2019	67
Transmission electron microscopy of nanomaterials, European Summer Schoole TEM-UCA (CAD)	September 12 to 16, 2022	36
European School on 4D STEM Imaging (AdSTEM3) (LJU)	May 14 to 18, 2023	61
QEM 2022 (TOU+ORS)	May 8 to 20, 2022	154
The 6th Stanisław Gorczyca European school on electron microscopy and tomography (KRA)	July 12 to 15, 2022	45
Sample preparation		
Workshop on Advanced TEM Specimen Preparation (STU)	October 24 to 28, 2022	15

Task 2.1.: Schools and Workshops

School/workshop name	Date	Participants
Spectroscopy		
ESTEEM-spectroscopy school (GRA)	February 4 to 7, 2020	16
Conventional and counting EELS spectroscopy school (CAT)	July 22 to 25, 2019	27
Diffraction		
Workshop on electron diffraction for solving engineering problems (TRO)	June 21 to23	42 (+13 online)
In situ		
European workshop on advanced electron microscopy of ICT structures and quantum device materials structures (CHA)	May 8 to 9, 2023	58
Workshop on TEM Characterization Techniques: Focusing on In-situ and EELS (JUL)	March 22 to 24, 2023	30 (+162 online)

Task 2.1.: Schools and Workshops

ESTEEM3 Additional Schools and Workshops

Title of the school/workshop	Partner	Dates	Participants
Workshop on Electrochemistry in Liquid TEM and on Orientation/Phase Mapping in Liquid	Amiens	May 27 to 29, 2019	23
Advanced Direct Detection EELS Workshop	Graz	September 25 to 26, 2019	12
European EELS & EFTEM School	Graz	February 7 to 10, 2023	11

Task 2.2.: Webinars

- ✓ Low-loss EELS modelling and tomography (GRA)
- ✓ Preparations and considerations for in-situ microscopy of ICT structure (CHA)
- ✓ Open software for TEM image simulation (ANT)
- ✓ Computer-assisted electron crystallography (CAD)
- ✓ Industrial webinars by the SME partners (NanoMEGAS, DENSsolutions)
- In-situ techniques (LJU): Mid June2023

D2.2: Second report on training and education for the period M17 to M40 (M40 –GRA)
D2.3: Third report on training and education for the period M40 to M52 (M52 – LJU)

WP3: Outreach

Work package number	3	Lead beneficiary			CAM, STU	
Work package title	Outreach						
Participant number	*1*	*2*	*3a*	*3b*	*4*	*5*	*6*
Short name of participant	STU	JUL	TOU	ORS	ANT	OXF	CAM
Person months per participant:	6	2	3	3	3	3	6
Participant number	*7*	*8*	*9*	*10*	*11*	*12*	*13*
Short name of participant	LJU	GRA	ZAR	CAD	KRA	CHA	TRO
Person months per participant:	3	3	3	3	3	3	5
Participant number	*14*	*15*	*16*	*17*	*18*	*19*	*20*
Short name of participant	CAT	ATTO	CEOS	DENS	NM	QD	EUR
Person months per participant:	3	1	1	1	1	1	12
Start month	1			End month	48		

WP3: Outreach

1

Objective 1

- Promotion of ESTEEM3 activities and of TA offers in particular, including dissemination to industry and non-specialist scientific communities and general public

2

Objective 2

- Increase in collaborations and service provision

Task 3.1: Dissemination and communication

(MPG, EUR with participation of all other partners)

Dissemination and communication plan

Task 3.2: Industrial engagement

(led by CAM and TRO with participation of all academic partners)

Task 3.1: Dissem. and Comm.

Task 3.1: Dissemination and communication

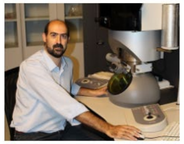
(MPG, EUR with participation of all other partners)

Academic and non-academic research community	• Communities interested in the ESTEEM3 project's developments, results and innovation. • Scientific contributions of ESTEEM3 are particularly interesting for them.
Industrial sector	• Address and trigger the active involvement of companies in the TA scheme.
Government bodies, professional assoc., and policy makers	• Regional authorities • Professional associations • Public administrations at regional and national level
EU technology clusters	• Shared interests with European Materials Characterisation Council (EMCC)
EU projects working in similar domain	• EUSMI provides an interdisciplinary infrastructure for soft matter research • NFFA sets out a platform to carry out comprehensive projects for multidisciplinary research at the nanoscale. • Q-SORT is a research project focusing on employing TEM as a so-called Quantum Sorter
TEM manufacturing companies	• TEM and component manufacturers are obvious stakeholders of the project.

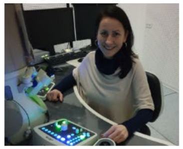
Task 3.1: Dissem. and Comm.

Main achievements

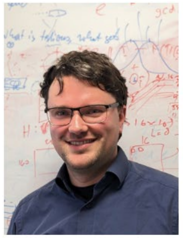
- Updating **different sections of the website** with
  - **58 Deliverables,**
  - **Protocols and softwares (update ongoing)**
  - **General news and events**
  - **Schools, workshops, webinars (3 recorded webinars available online)**
  - **9 interviews,**
  - **13 TA success stories**,
  - **24 job positions** etc.
  - Key figures from project start : **>22K visitors, >79K views**
- **Promotional videos :**
  - stop motion video displayed on the homepage of the project (M8) : **864 views**
  - **Final video will be presented during the final event**
- Creation of **visual identity** of the project (M3), comic strip



ESTEEM3 interviews : Raul Arenal from LMA-Universidad de Zaragoza



ESTEEM3 interviews : Vesna Srot from Stuttg Center for Electron Microscopy



ESTEEM3 interviews : Dieter Weber from Forschungszentrum Jülich

ESTEEM3 inteviews : Susana Trasobares Llorente from University of Cádiz

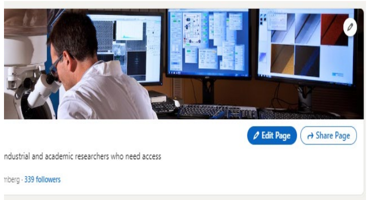
Task 3.1: Dissem. and Comm.

- Production of **bi-annual newsletters**
  - **7** editions so far: Sept 2019, April 2020, Oct 2020, June 2021, Nov 2021, June 2022, Dec 2022
  - Last newsletter will be planned in June 2023
  - **561 contacts (+2% since December 2022) -** (1/3 open it)

- Promotion on **social media**
  - **494 members on LinkedIn (+20% since December 2022)**
  - **524.505 followers on Twitter (+4% since December 2022)**

- **Brochures** (2) explaining the project with a focus on TA (M6, final brochure)

- **Scientific publications**



Task 3.1: Dissem. and Comm.

- **Collaboration** with other EU projects:
  - **EUSMI** – European Soft Matter Infrastructure
  - **Q-SORT** – The Quantum Sorter
  - **TEESMAT** – The Open Innovation Test bed for Electrochemical Energy Storage MATerials

- **Organisation of two TA user forums**
- **Workshops and conferences**: Hosted or participated in professional events to share findings and engage with the wider scientific community.
- **Public Engagement**: Public lectures and engagement to share the importance and impact of the project's work with a wider audience.
- **Online presence**: A project website, social media, webinars, and online resources - useful for reaching a global audience.

Task 3.1: Dissem. and Comm.

Communication

Actions	Metric	Status of KPI M53	Objective	Excellent	Good	Moderate	Weak	Comments
Brochure	Number	2	2	≥ 1	1	-	0	2nd Brochure planned for M52
	Prints	850	1200	≥ 1150	≥ 850	≥ 550	< 350	200 additional final brochures will be printed before the end of the project
Website	Visitors (monthly)	884	500	≥ 450	≥ 350	≥ 250	< 150	Excellent
	News posts	139	200	≥ 150	≥ 100	≥ 50	< 25	Publication pace has been accelerated by the increase of activities and results in the last months. The last 3 months of the project will generate additional news.
Newsletter	Subscribers (S)	560	500	≥ 450	≥ 350	≥ 250	< 150	Excellent
	Number	7	8	≥ 8	≥ 6	≥ 3	< 3	1edition is upcoming (June 2023)
	Openers (%) / O%	43%	40%	≥ 40	≥ 30	≥ 20	< 10	It has been accelerated & achieved by the increase of activities and results in the last months
Twitter	Followers	514	400	≥ 350	≥ 250	≥ 150	< 50	Excellent
LinkedIn	Members	451	400	≥ 350	≥ 250	≥ 150	< 51	Excellent
Videos	Number	1	2	≥ 1	1	-	0	1 final video upcoming at M52 will be presented for the final event
	Views	740	500	≥ 450	≥ 350	≥ 250	< 150	Excellent

Task 3.1: Dissem. and Comm.

Dissemination

Journal articles (peer-reviewed and open access)	Number	520	500	≥ 450	≥ 350	≥ 250	< 150	Excellent
Public deliverables	Number	58	57	≥ 50	≥ 30	≥ 10	< 5	It will be accelerated & achieved by the submission of a wave of public deliverables in April 2023
Conference papers and proceedings	Number	15	10	≥ 10	≥ 6	≥ 3	< 3	Excellent
Schools, workshops, training organised by ESTEEM3	Number	14	11	≥ 10	≥ 6	≥ 3	< 3	2 will be organised by the end of the project (2023)
Exhibitions	Number	8	5	≥ 5	≥ 4	≥ 1	< 1	Excellent
Other educational/training events including actions to industrial users	Number	70	20	≥ 20	≥ 17	≥ 10	< 10	Excellent
Participation in activities organised jointly with other EU project(s)	Number	4	5	≥ 5	≥ 4	≥ 1	< 1	Good, the consortium will seek for collaborations for the final event.
Online presence - media articles	Number	5	5	≥ 5	≥ 4	≥ 1	< 1	. Publications in the EMS yearbook as well as EU research magazine.
Press releases	Number	2	2	>2	2	1	0	There are two press releases issued by the partners of the project. Instead of a press release, the consortium decided to create an additional video to present the project's results.

Task 3.1: Dissem. and Comm.

Summary of Dissemination & Communication activities

Estimated number of persons reached	
Estimated number of persons reached for RP1	36859
Estimated number of persons reached for RP2	104986
Estimated number of persons reached for RP3	179121
Total reach	**320966**

Task 3.2: Industrial engagement (led by CAM and TRO with participation of all academic partners)

Activities

- Discussions with other infrastructure projects (See WP 3.1).
  - TEESMAT (Open Innovation Test Bed (OITB) for Electrochemical Energy Storage Materials).
  - EUSMI (European Soft Matter Inrastructure).
- Webinar: Transnational Access within the Projects ESTEEM3, EUSMI and TEESMAT 9 December 2020.
- Online meeting 15 Jan 2021 – presented ESTEEM3 to TEESMAT.
  - Potential service-provider, TEM not included in portfolio.

WP3: Outreach, Task 3.2: Ind. Engag.

- Organised ‘‘Microscopy in Industrial Applications” session at EMC2020.
  - Cancelled due to Covid, considering EMC2024.
- “TNA for industry users” online discussion session arranged by DG Research & Innovation Research Infrastructures group, 2nd Dec 2022.
  - Input gathered from a range of consortia invited to discuss industrial TNA openly.
  - Feedback has been collated and shared with participants.
  - Session in ESTEEM3 Innovation Strategy Workshop, Jan 2023.

WP3: Outreach, Task 3.2: Ind. Engag.

Deliverables

- D3.2: Initial list of industrial collaborations (M4 TRO)
- D3.3: Initial plan for establishing industrial awareness and uptake of TA (M6 CAM)
- D3.4: Report on engagement with industry (M20 CAM, update at M40)
- D3.4: Update of the report on engagement with industry (M46).

ESTEEM3
TRANSNATIONAL ACCESS ACHIEVEMENTS

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

WP12: Transnational Access

Transnational Access procedure

Application procedure

- **TNA applicants were able to :**
  - Apply directly online via ESTEEM3 website (centralised system)
  - Apply at any time (no call for applications with deadline). Applications were open from 2019 to early 2023

TNA applicants needed **to submit one administrative form + one proposal form (description of the work)**

Transparent process : TNA User charter available online,

Document explaining the condition of access, the application process (including submission and review processes), the processes when the TNA project is accepted

TNA application procedure

Steps of the TNA application procedure

1. Processing the applications that come through the ESTEEM3's website.
2. Checking applications according to eligibility criteria.
3. Proposal is sent to two members of the evaluation committee (TAPEC) for review
4. The requested laboratory is contacted to ensure they can provide access and get confirmation their expertise is aligned with the project
5. Official notification to the project leader and all the participants
   - If accepted : formal notification (Approval letter)
   - If discarded, recommendations provided for improving the project and resubmission invitation
6. Project ongoing
7. At the project's end, a report form is sent to the project leader to assess the project and ESTEM3 process.

TNA selection procedure

Transnational Access Proposal Evaluation Committee members (TAPEC)

Composition

- The TAPEC is composed of **49 members**, including **22 females** and **27 males**.
- Peer review process: **renowned** scientists in microscopy and materials science.

Rules

- The evaluating scientists are **not associated with the project** to ensure transparency.
- To avoid potential conflicts of interest and to ensure impartiality, the TAPEC members reviewing proposals will **neither come from the country of the applicant nor of the TA provider.**
- NDA signed with TAPEC members

Selection criteria

- **Scientific quality** of the proposal

(rank: weak: 0 – outstanding: 10)

- Demonstration of the **need for the use of the advanced infrastructure**

(rank: weak: 0 – outstanding: 5)

- Potential **impact for academic or industrial innovation**

(rank: weak: 0 – outstanding: 5)

Proposals with a total ranking less than 10 were rejected

During the implementation

- **Application** : Each project can request a maximum of **20** units (sample prep, TEM and DA included)
- **Implementation** : there is some flexibility

Compared to the requested TA, you can provide maximum the following additional units without notifying the coordinator/ Euronovia (without amendment)

- Prepare **two more samples**
- Provide **three more days in total on TEM and/or data analysis**

- If the changes are more significant, TNA project **Amendment** can be considered :
  - If the project does not exceed 25 units, we will redistribute the requested TA units
  - If the number of units to be provided is more than 25, a new project will have to be submitted

TNA promotion

Communication & Dissemination activities are focused on the TNA application and TNA project results :

The ESTEEM3 website is currently updated with the latest news on Transnational Access and all activities are detailed.

Weekly news are published on social networks, including any information related to the Transnational Access activities.

For example:

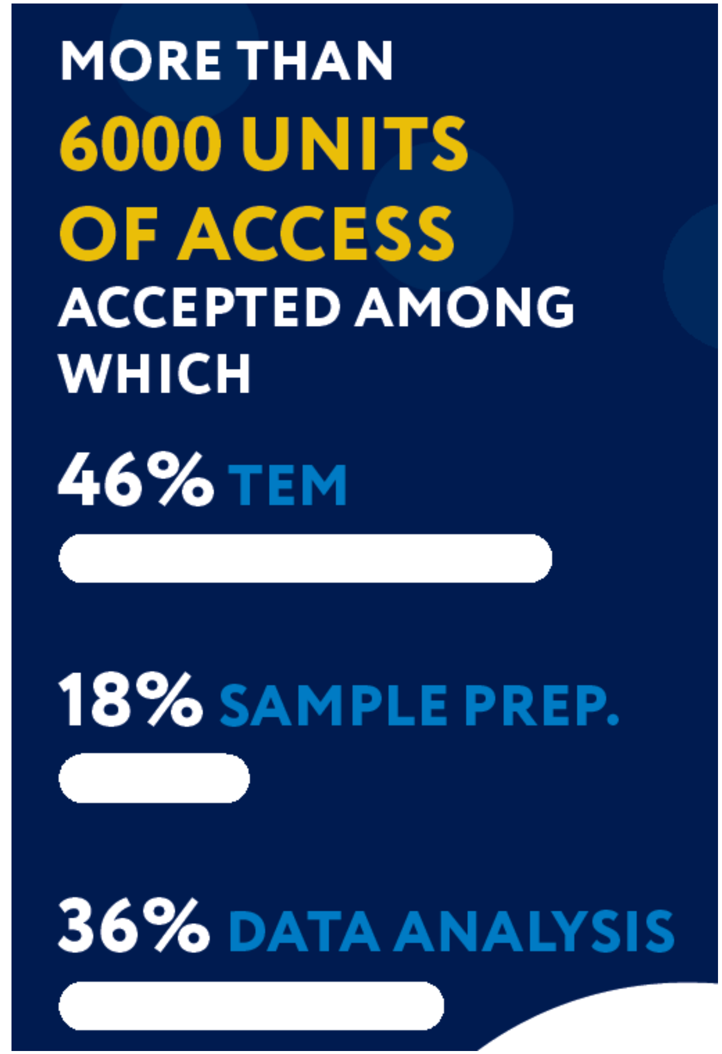
- **13 TA success stories** are already published on the website and shared on our social media.
- **10 articles** will be available end of June 2023!
- **2 TNA Users forum organised**

Overview of the TNA Progress

General overview

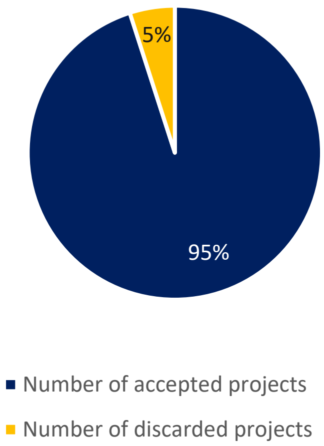
Approx. **500**

TNA projects accepted

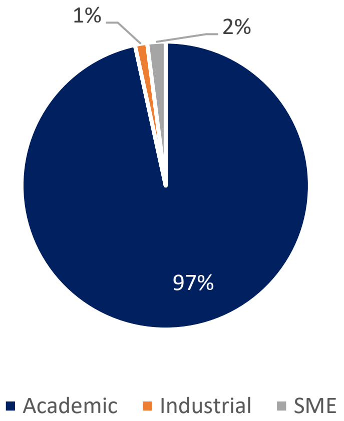


General overview

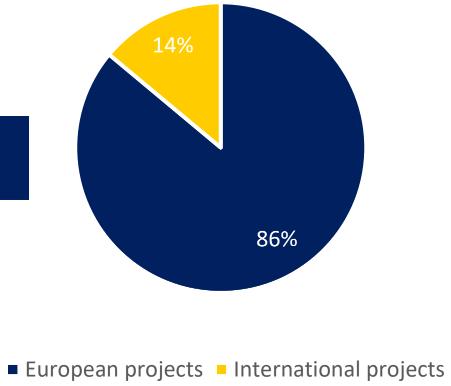
Success rate



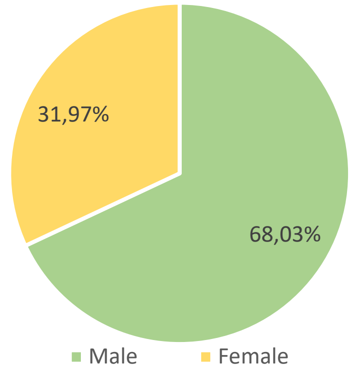
Sector



Country origin



Gender Balance



ROUND TABLE:
TNA FOR INDUSTRY IN THE CONTEXT OF EU GRANTS

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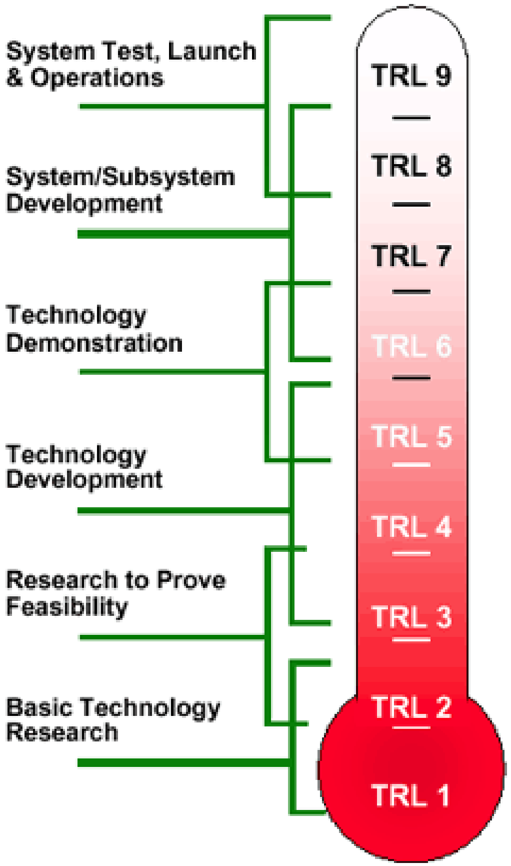
TNA – ESTEEM3

- Industry uptake in ESTEEM3
  - Level below ambitions.
- Direct discussion with other projects
  - TEESMAT (battery), EUSMI (soft materials) – multi technique programmes.
- Policy officers EC RI programme – meetings end of 2022
  - Industrial TNA uptake challenging for many projects and overall.
  - Range of requirements apparent, fast access, consultancy, insight, establishing networks.
  - Larger companies may prefer to pay to avoid publication requirement.
  - Transnational requirements vs local networks less attractive, particularly SMEs.
  - Notes have been distributed.

Feedback from ESTEEM3?

- Accommodate range of access levels?
  - Basic imaging to advanced techniques.
- Consulting role?
  - TNA to Technology Readiness Levels.
- Best way to make contact?
  - Build networks, open calls, events (conferences, webinars)….
- Invited to provide input to RI officers.
  - Framework for future TEM TNA provision?
  - Industry in future TEM initiatives?
    - eDREAM, ARIA?



Participation to TNA
an industrial view

Dogan Ozkaya, Scientific Consultant EM,
Johnson Matthey Technology Centre

Presentation available only during the online event

LUNCH BREAK

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TNA USER FORUM

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esteem3 TNA user feedback

Nano-gold on hierarchically structured gadolinium-doped ceria as an active oxidation catalyst: synergistic effect of chemical composition and structural hierarchy

Piotr Woźniak
Institute of Low Temperature and Structure Research

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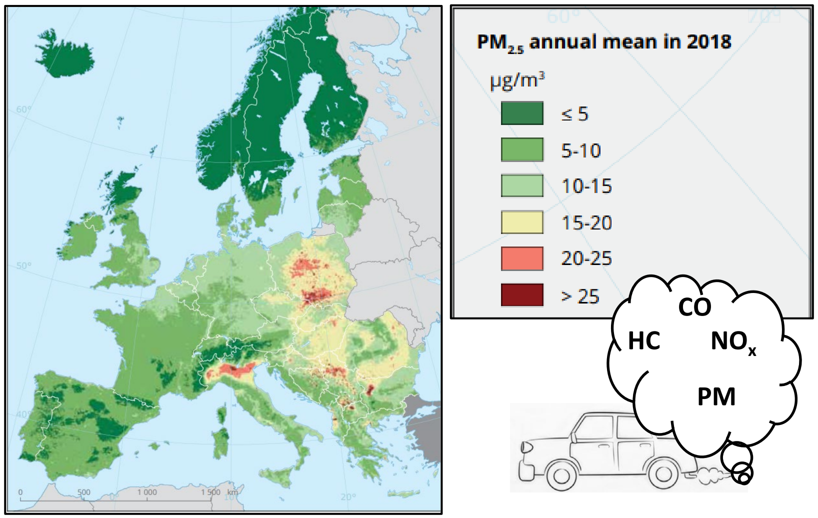
HSMCO project

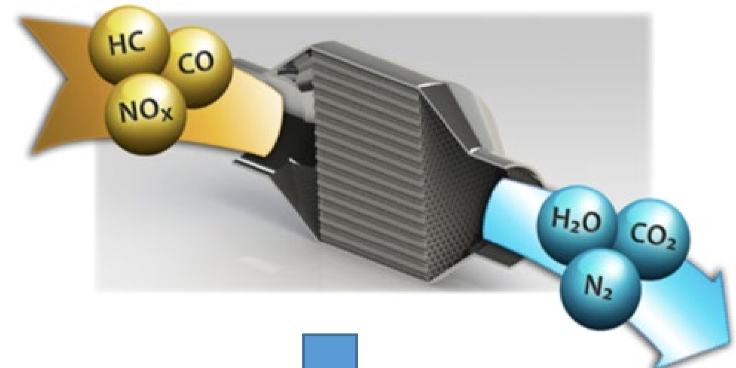
Hierarchically Structured Mixed Cerium Oxides

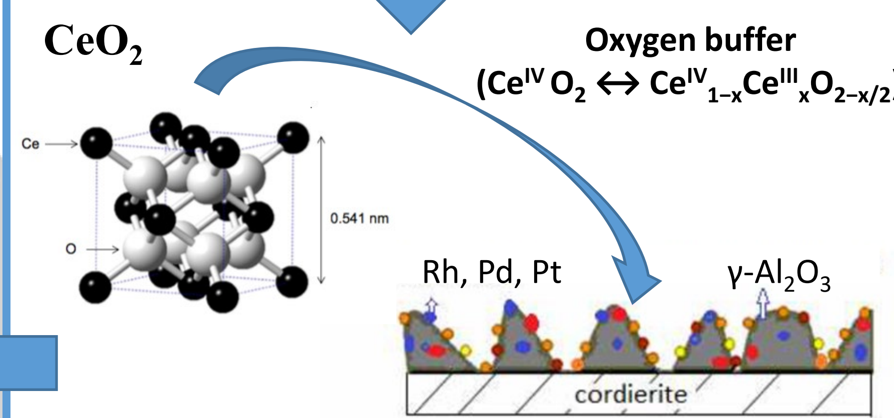
Motivation



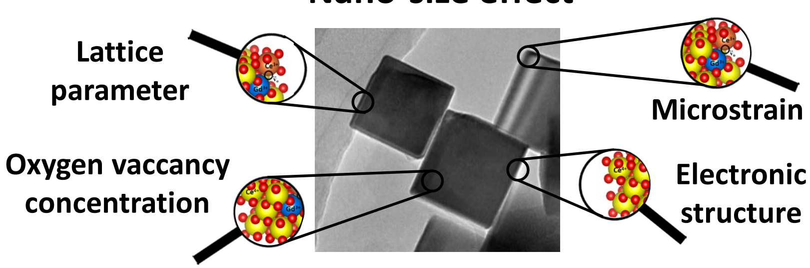
EEA/Air quality in Europe – raport No 9/2020

Automotive catalyst



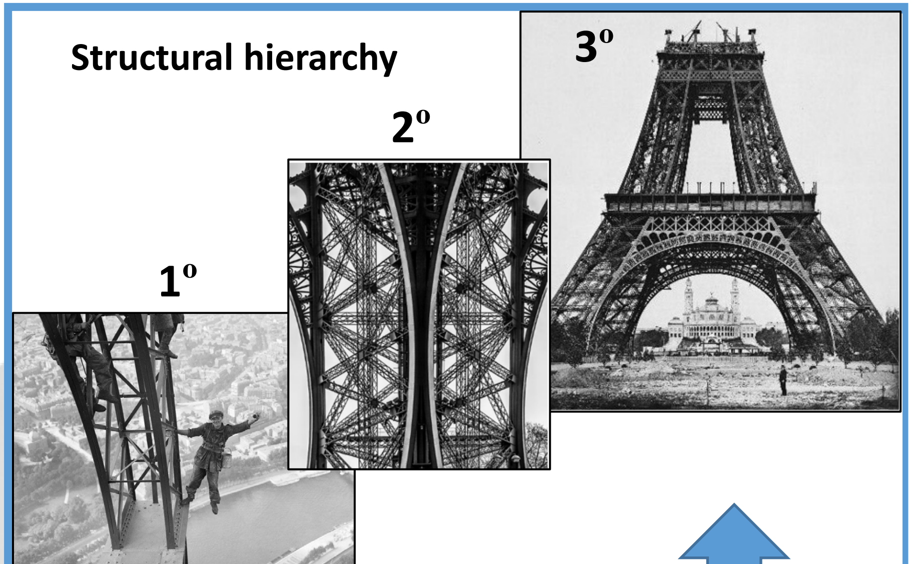


Nano-size effect

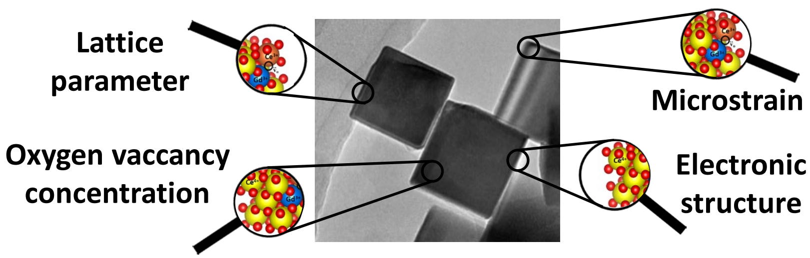


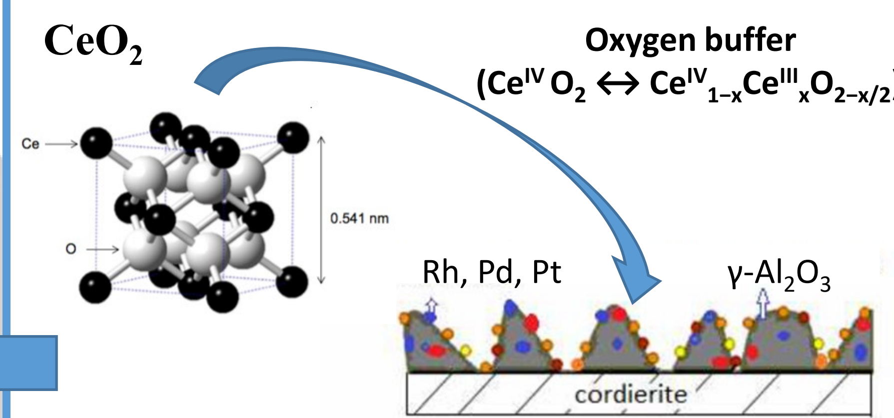
Motivation

Structural hierarchy



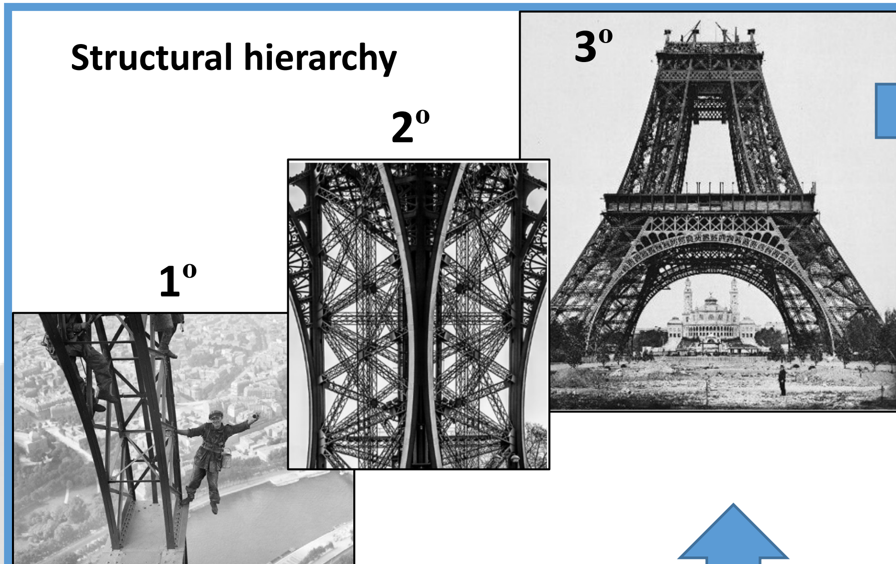
Nano-size effect



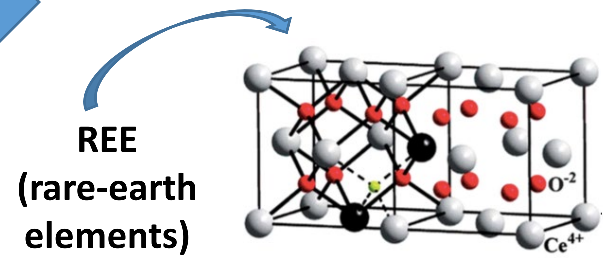


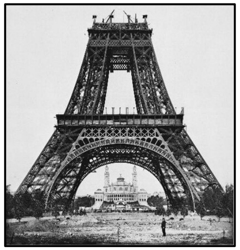
Motivation

Structural hierarchy



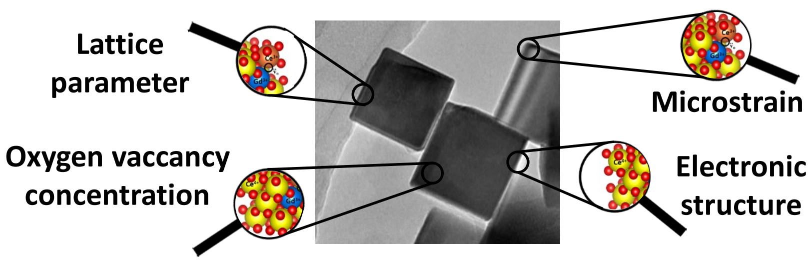
Doping



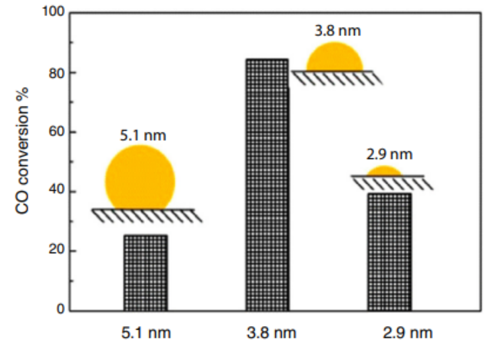


?

Nano-size effect



Active phase deposition



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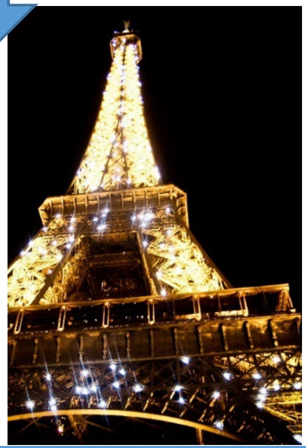


Motivation

Determination of structure-activity relationship

HSMCO project goals

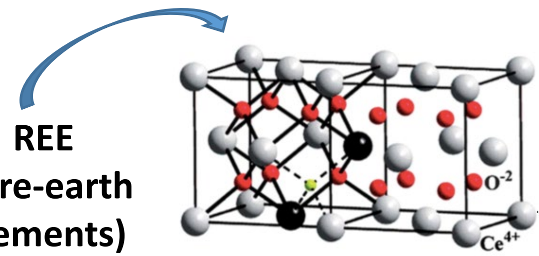
1. Description of the structural hierarchy of active catalytic supports and gold-decorated catalysts.
2. Determination of the Gd^{3+} dopant distribution throughout the system.
3. Determination of the distribution and location of Au NPs.

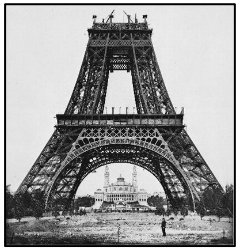
EM techniques used

HREM, EDX, EELS, HAADF-STEM, ET-HAADF

Doping

REE (rare-earth elements)





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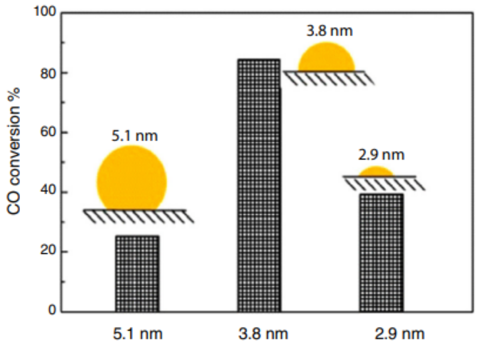
Active phase deposition



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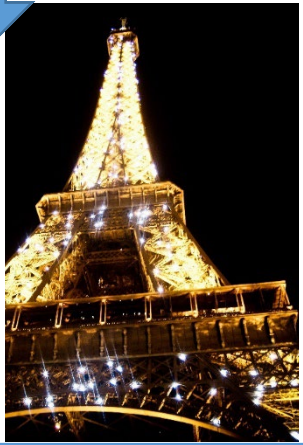
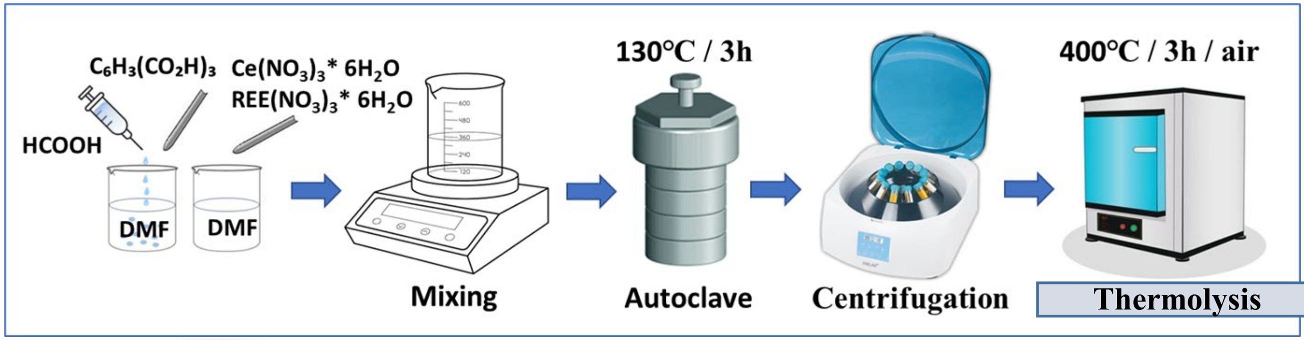
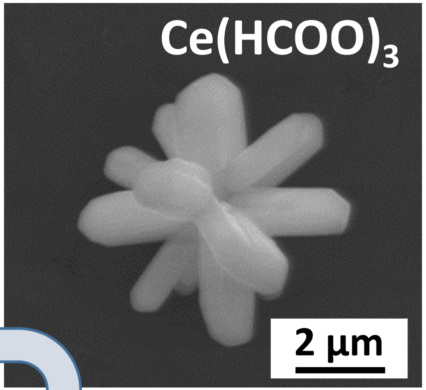


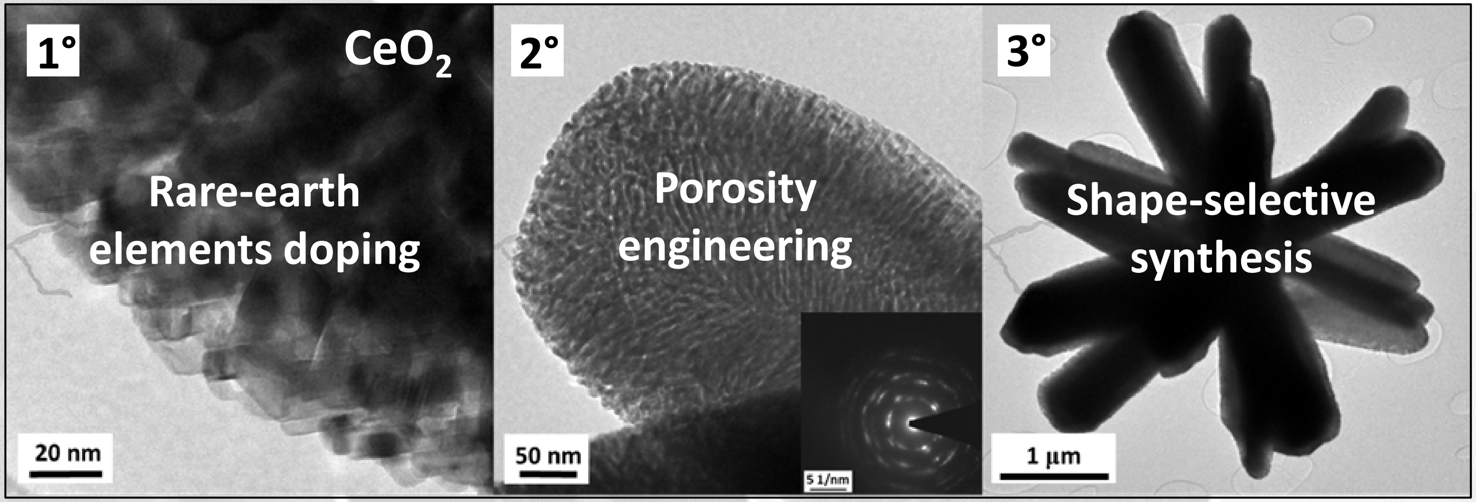
Structural hierarchy

Synthesis

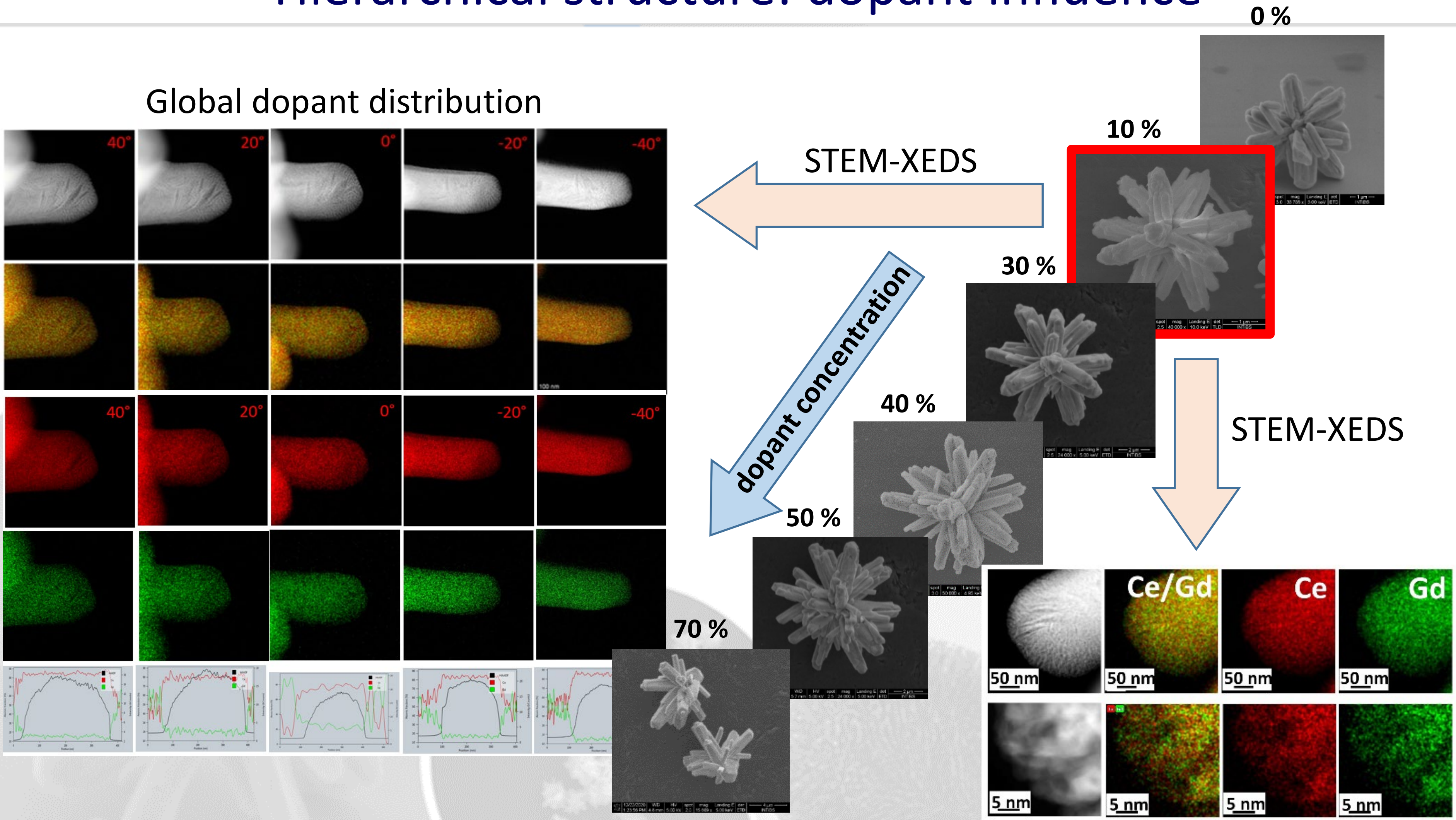




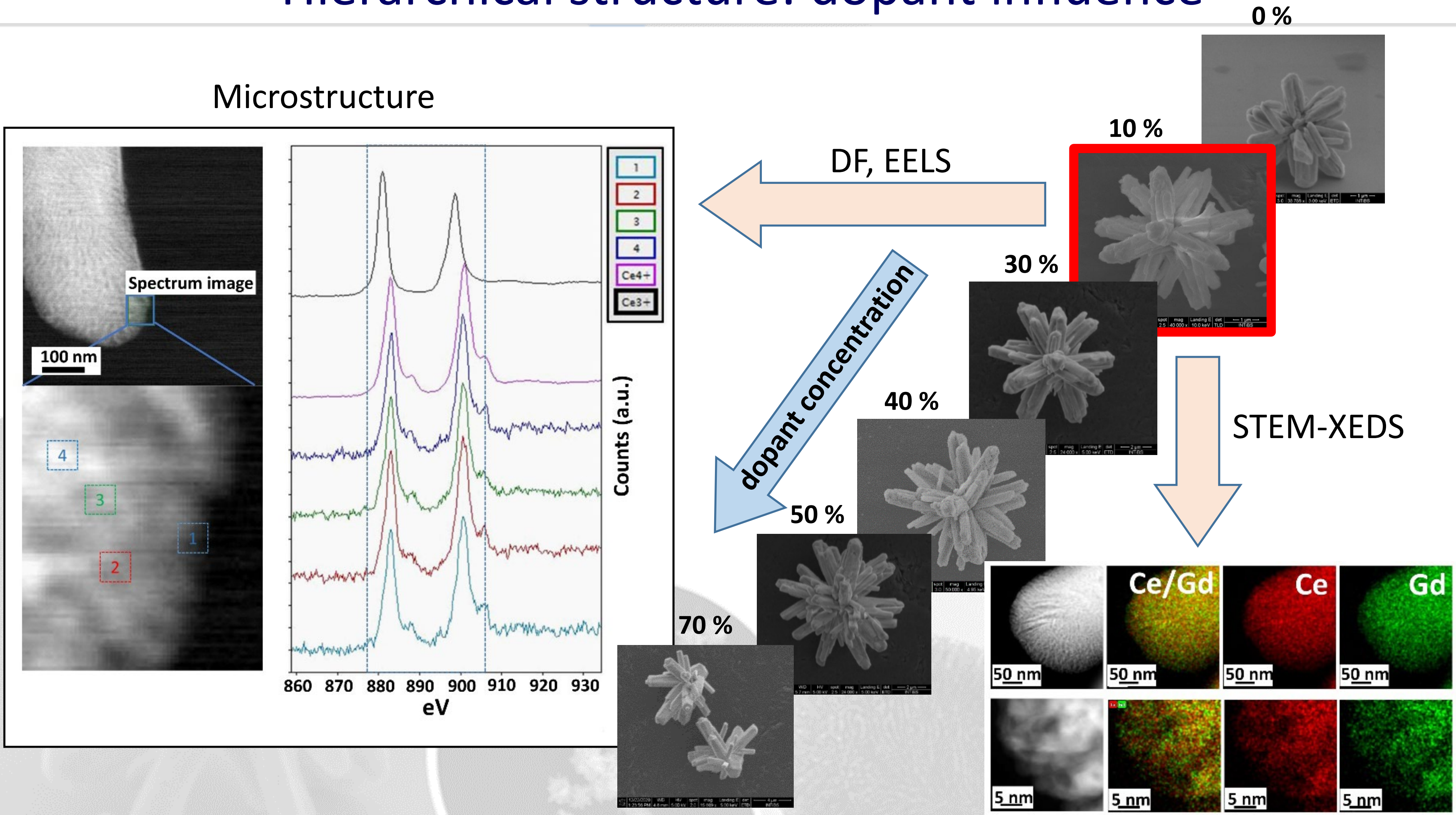
Nanoparticles | Porous rods | Star-shaped particles



Hierarchical structure: dopant influence

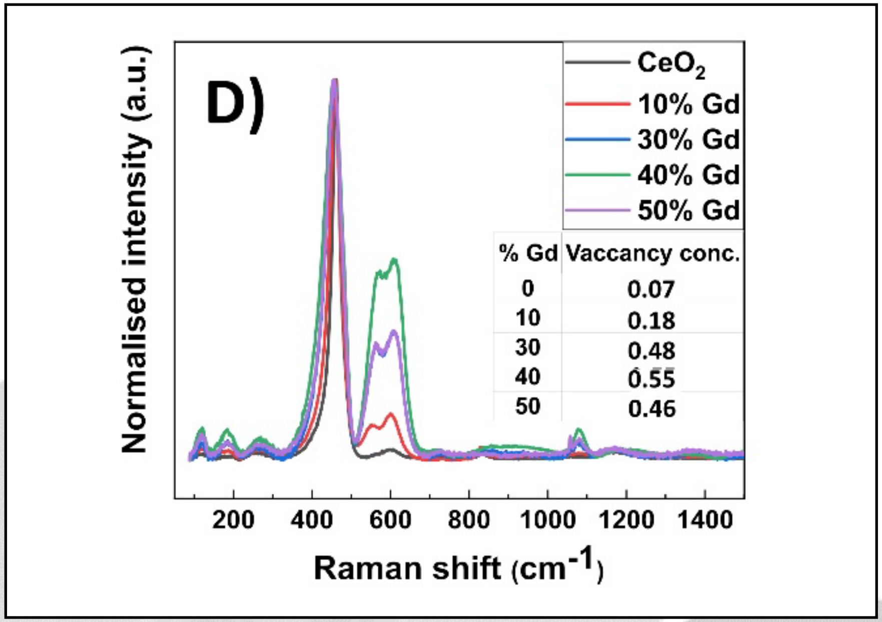


Hierarchical structure: dopant influence

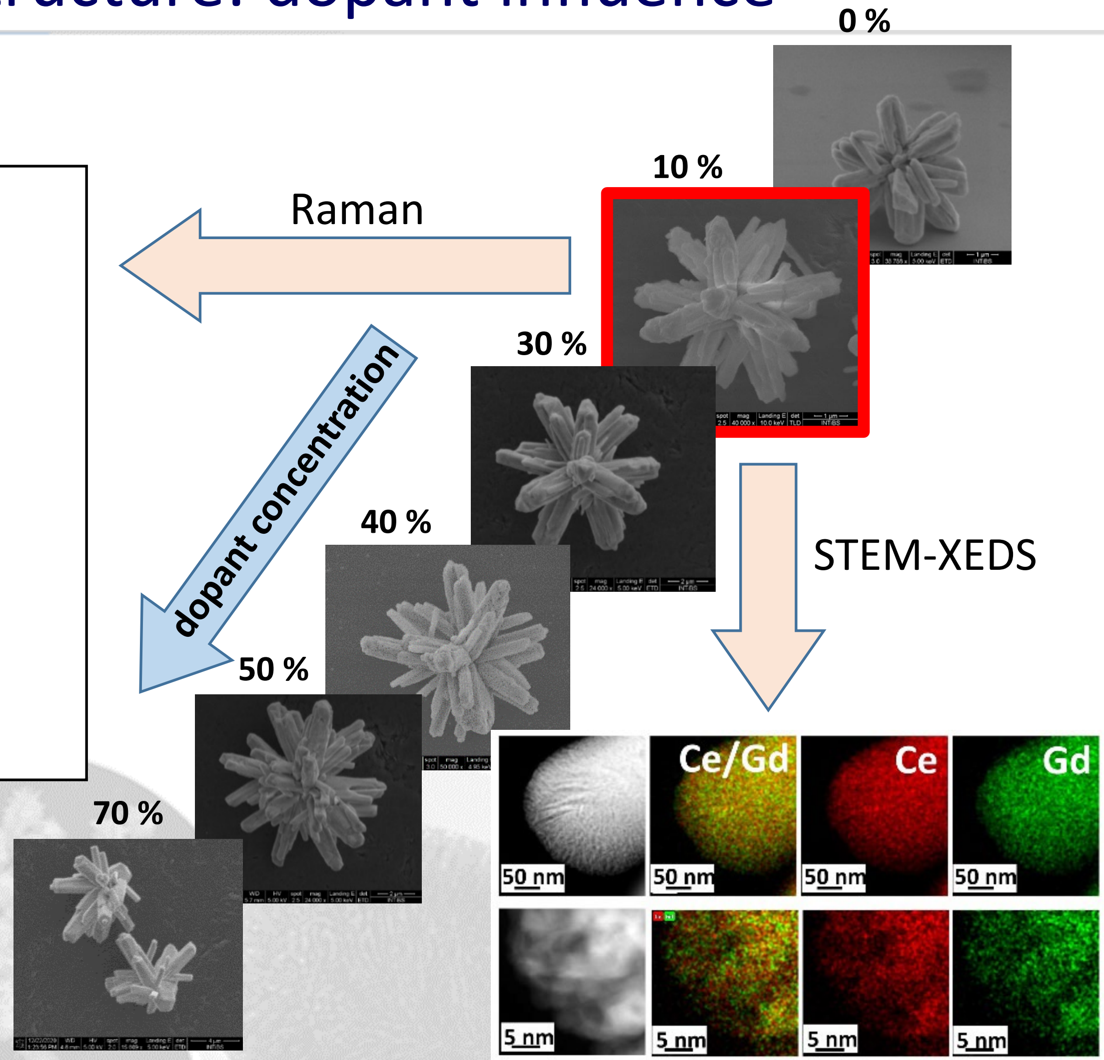


Hierarchical structure: dopant influence

Microstructure

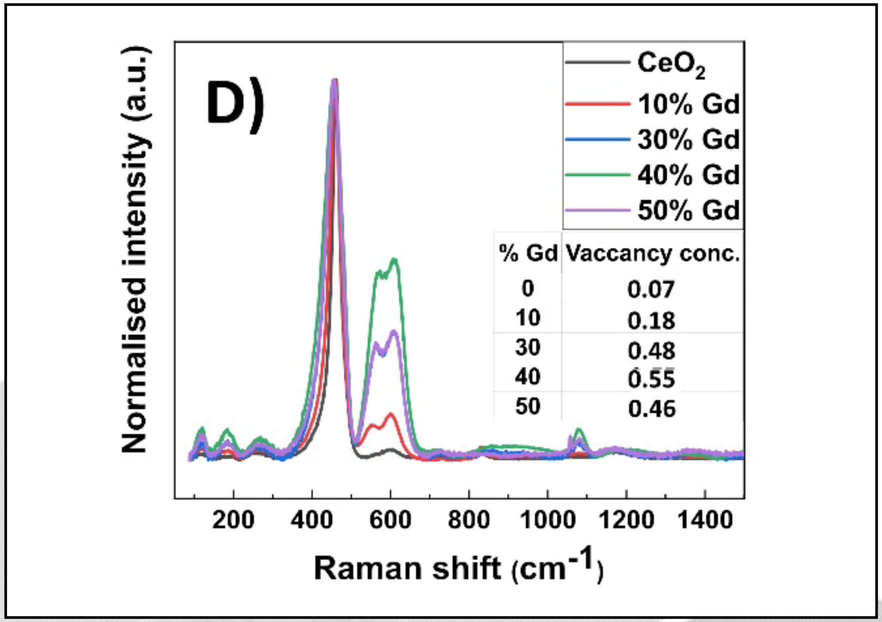


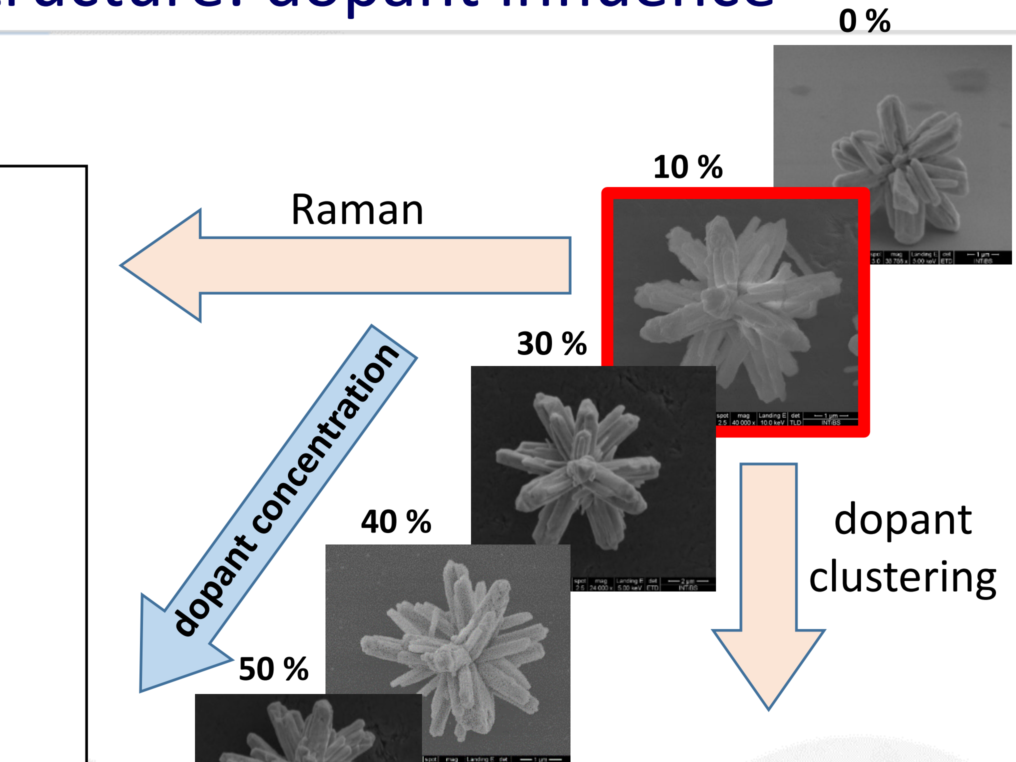
% Gd	Vaccancy conc.
0	0.07
10	0.18
30	0.48
40	0.55
50	0.46

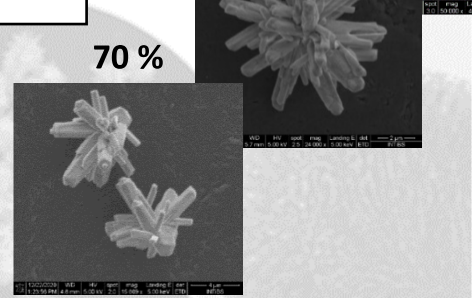


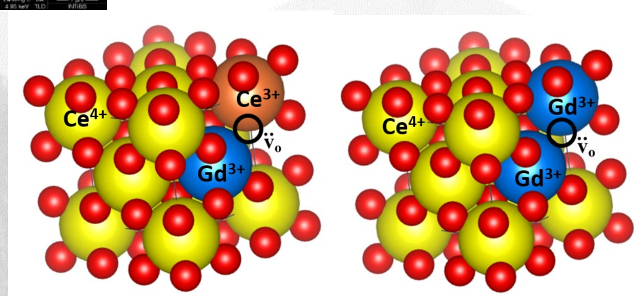
Hierarchical structure: dopant influence

Microstructure



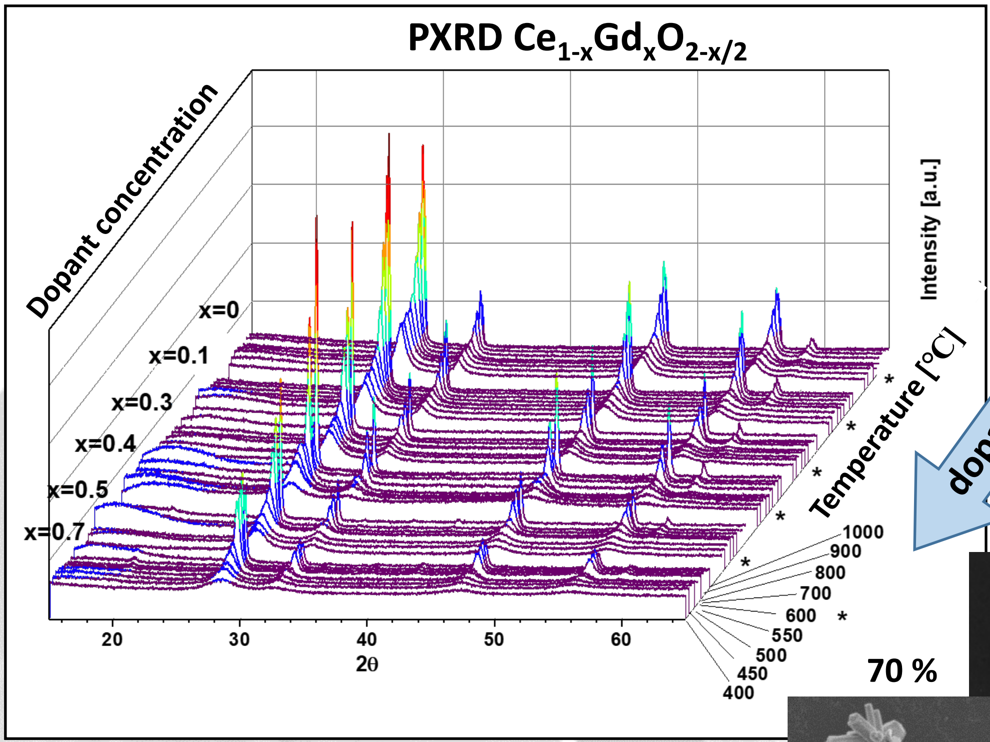


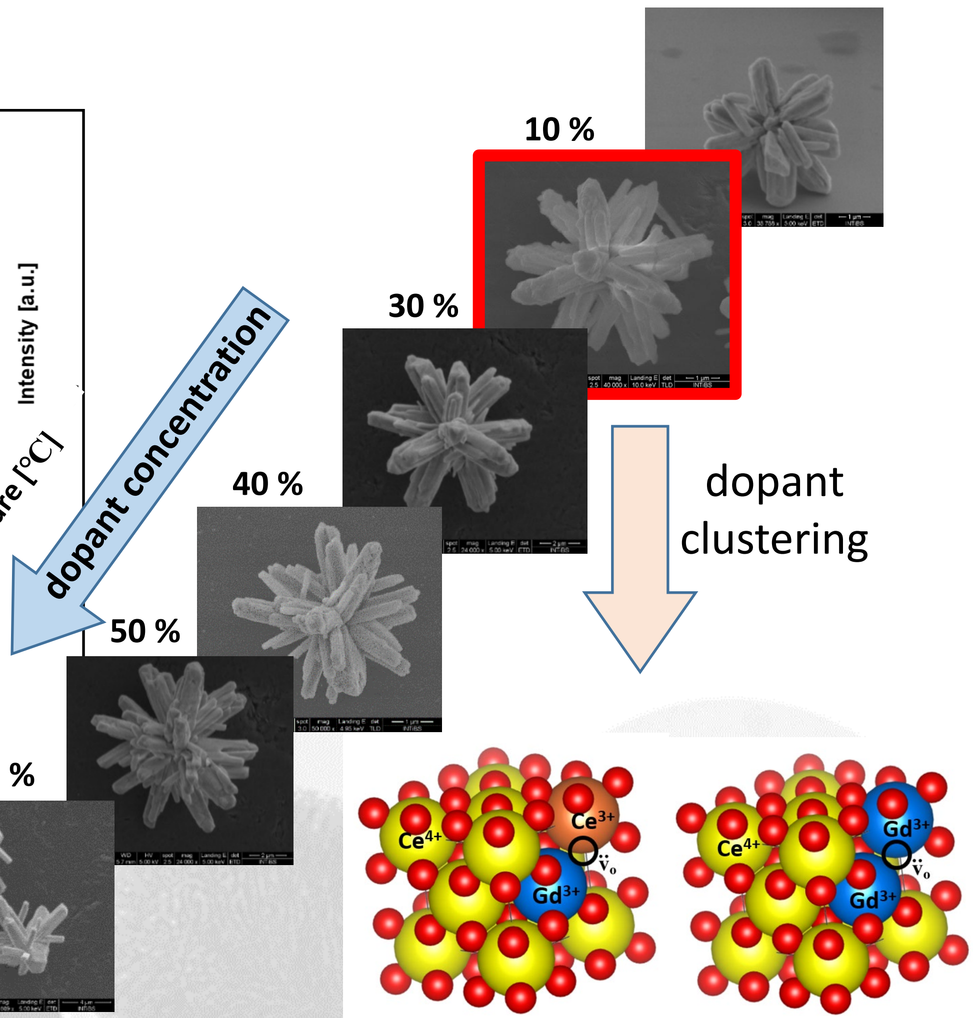




Hierarchical structure: dopant influence

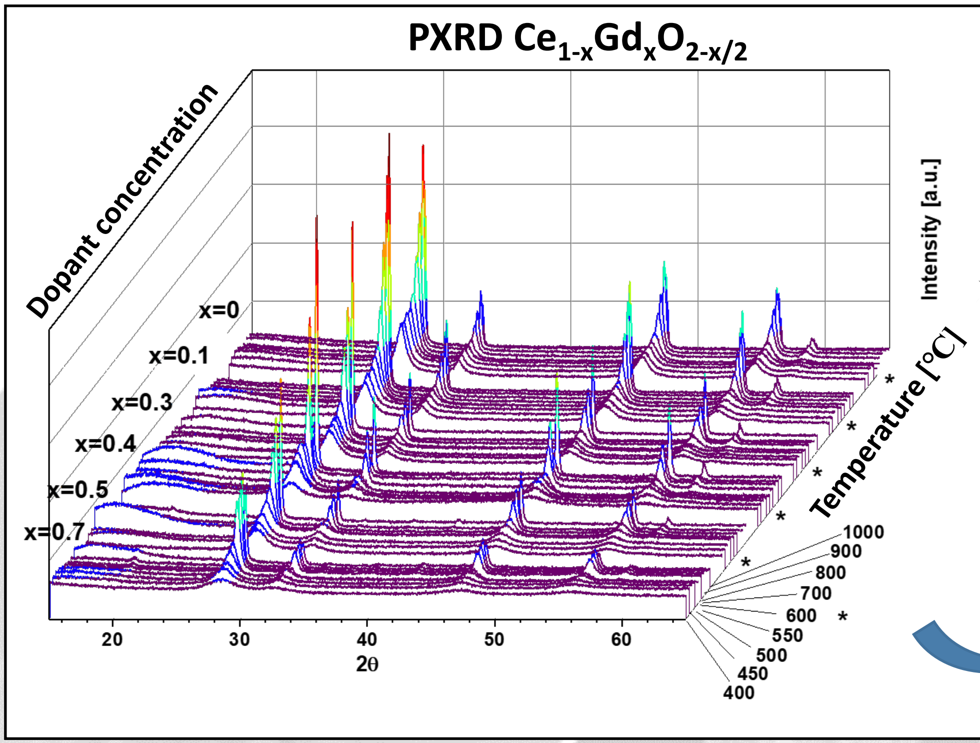
Crystal structure

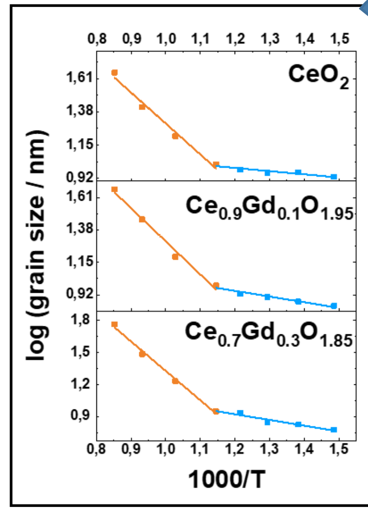




Hierarchical structure: dopant influence

Crystal structure

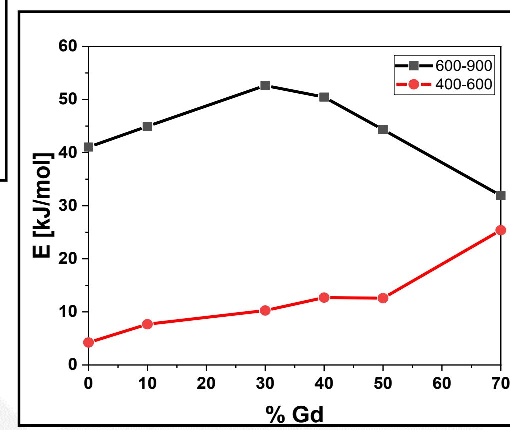




$$lnD = Ae^{-\frac{E_a}{RT}}$$

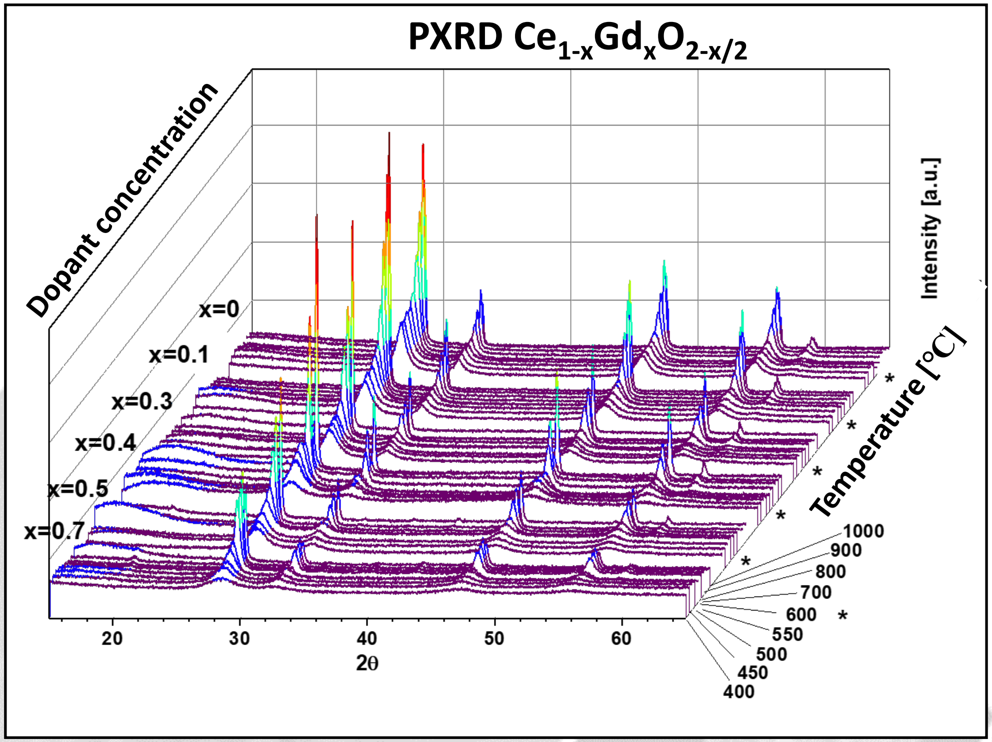
$$D \approx \frac{\lambda}{\beta_{2\theta} cos\theta}$$

Activation energy of NPs growth

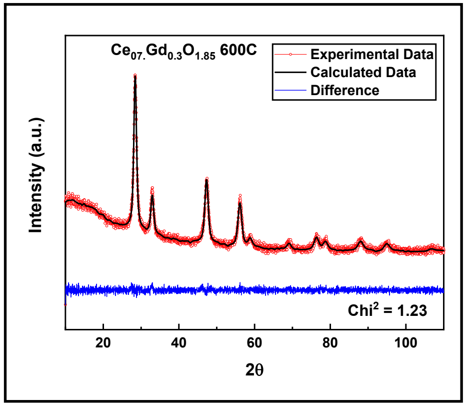


Hierarchical structure: dopant influence

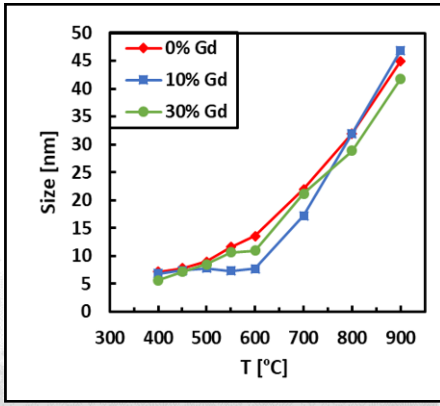
Crystal structure



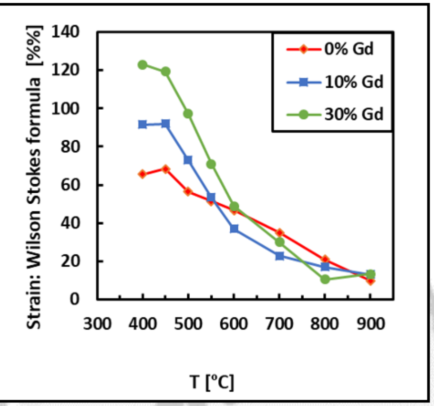
Rietveld refinement



NPs size

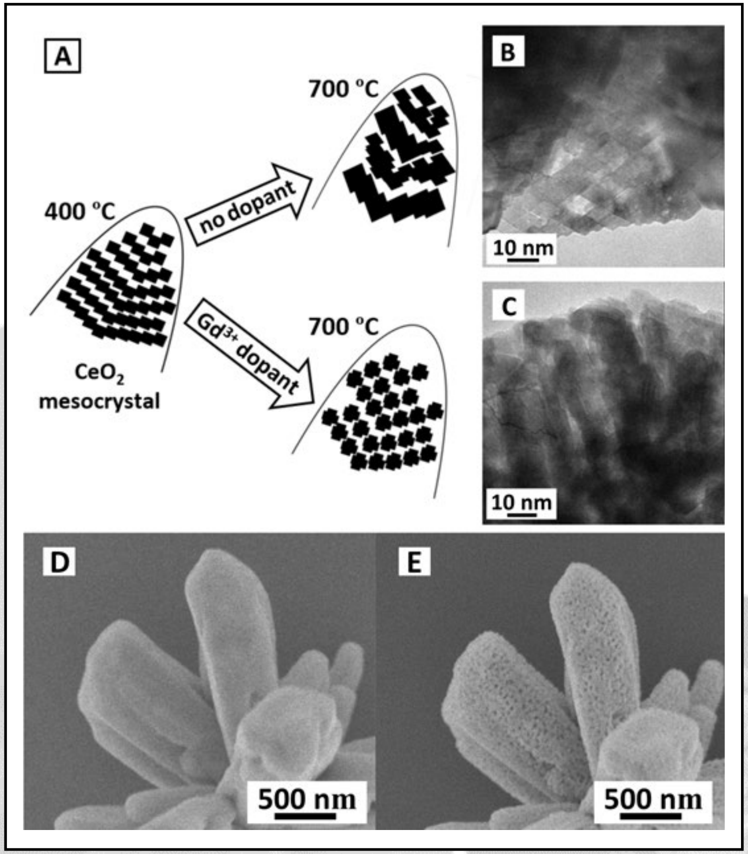


NPs strain

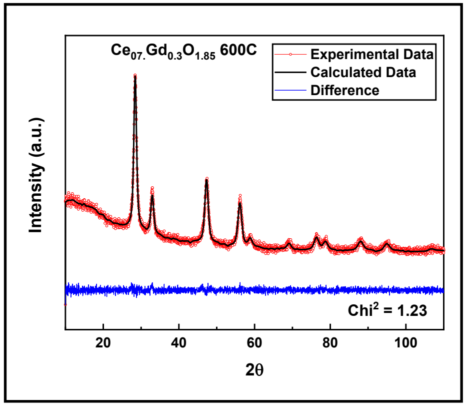


Hierarchical structure: dopant influence

Evolution of the support architecture

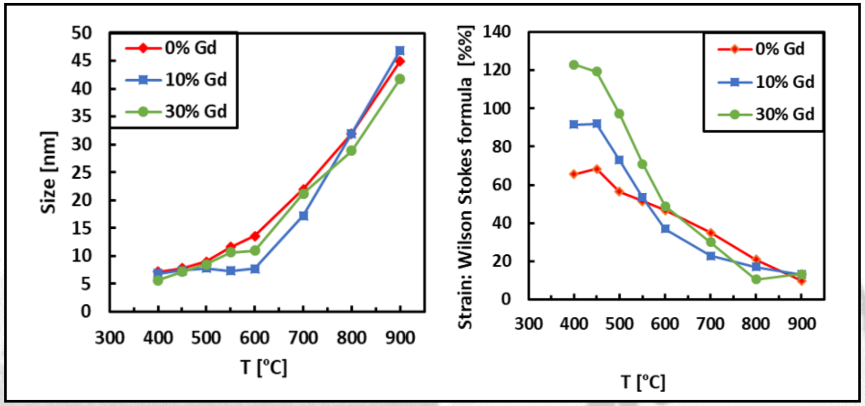


Rietveld refinement



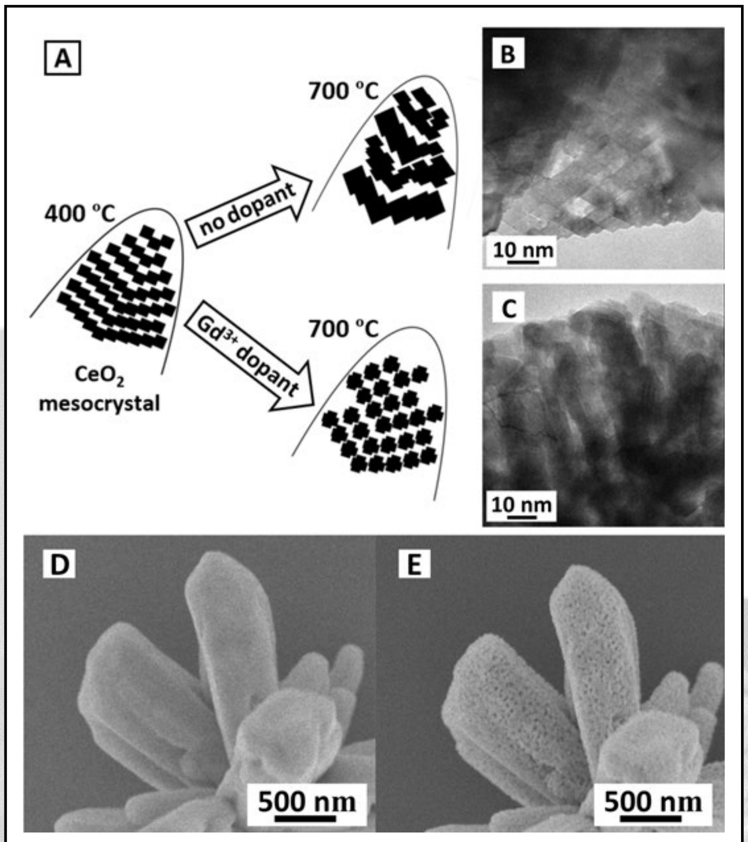
NPs size

NPs strain

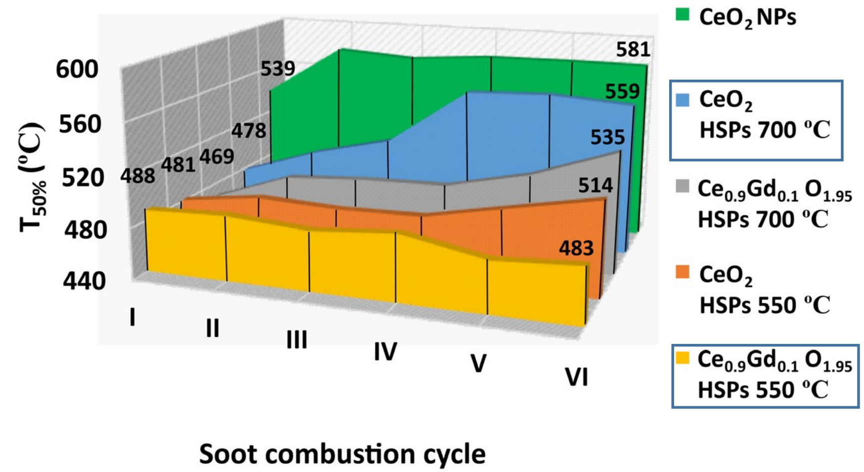


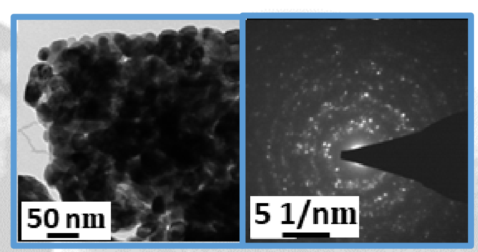
Hierarchical structure: dopant influence

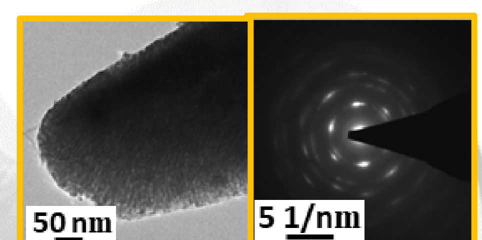
Evolution of the support architecture



Stability of the architecture in soot combustion

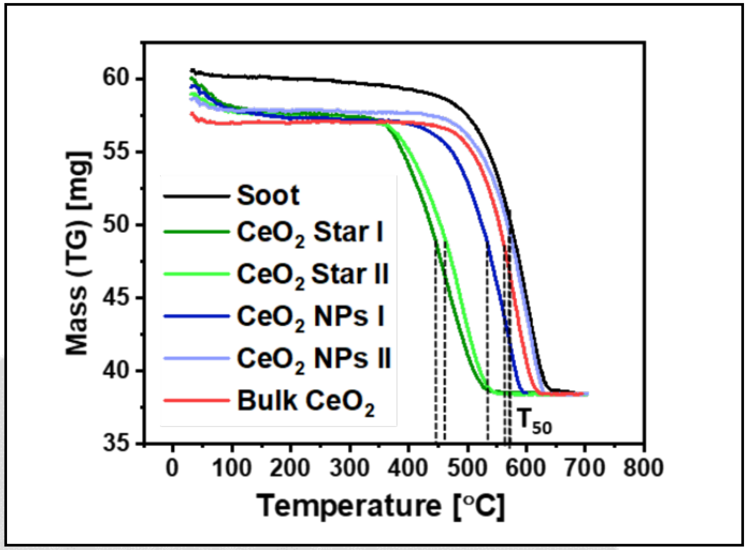




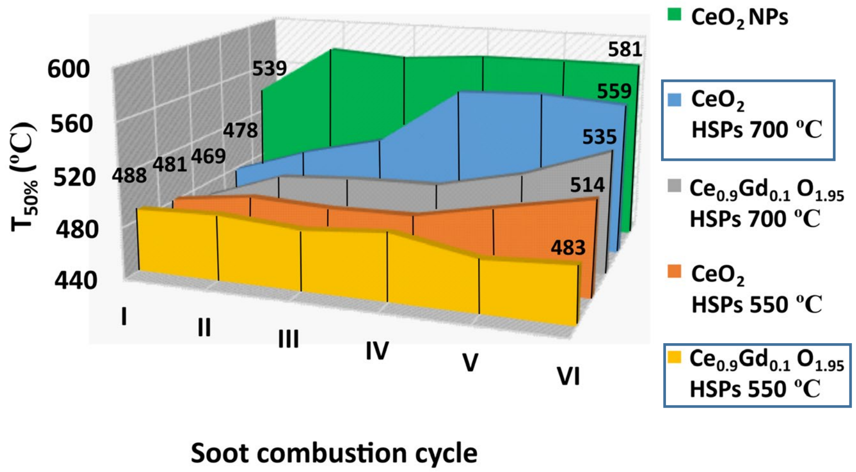


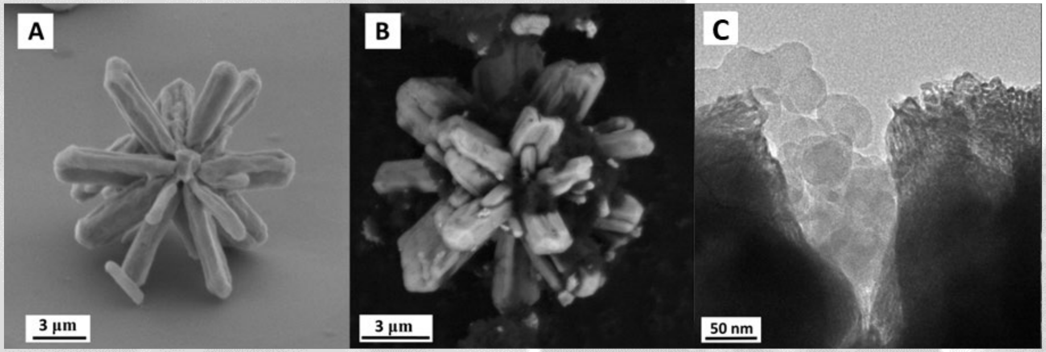
Catalytic activity

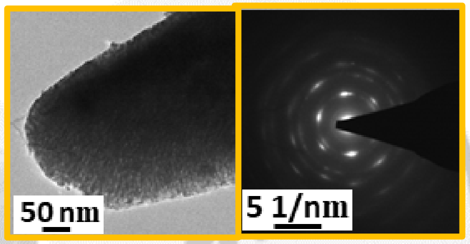
Soot combustion



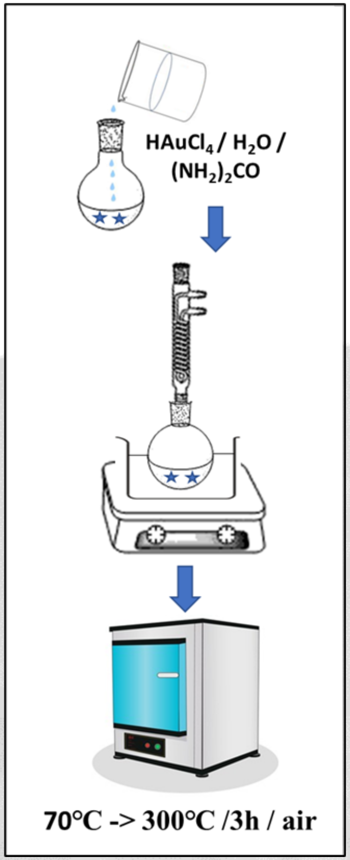
Stability of the architecture in soot combustion

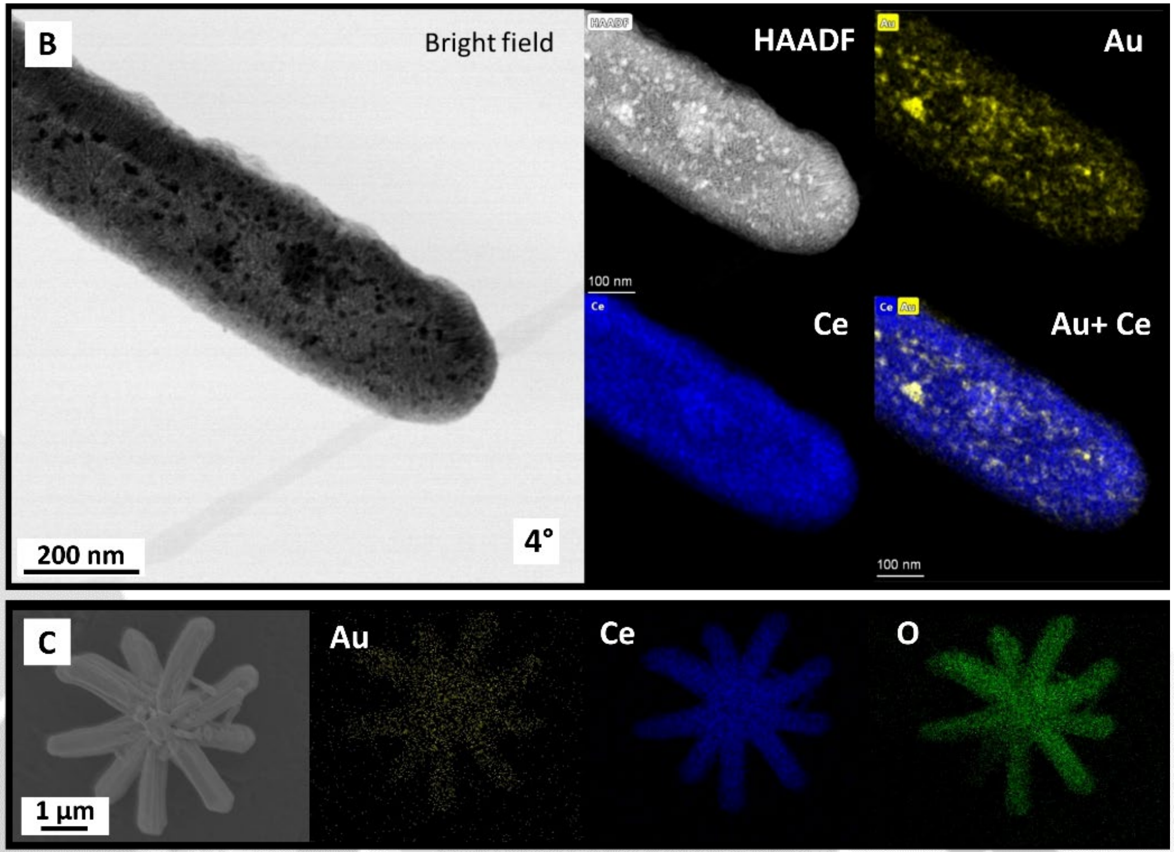




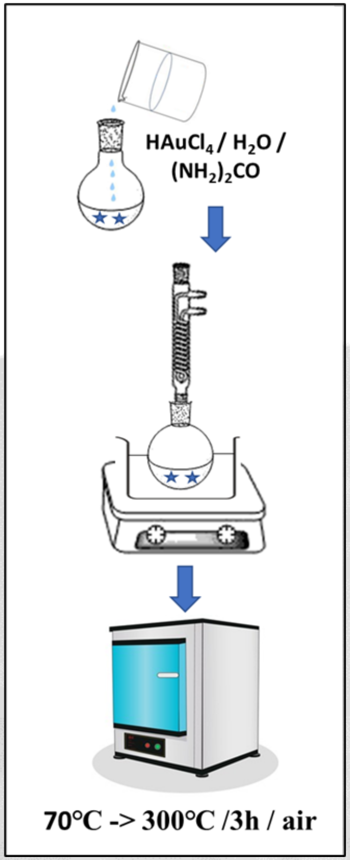


Active phase deposition



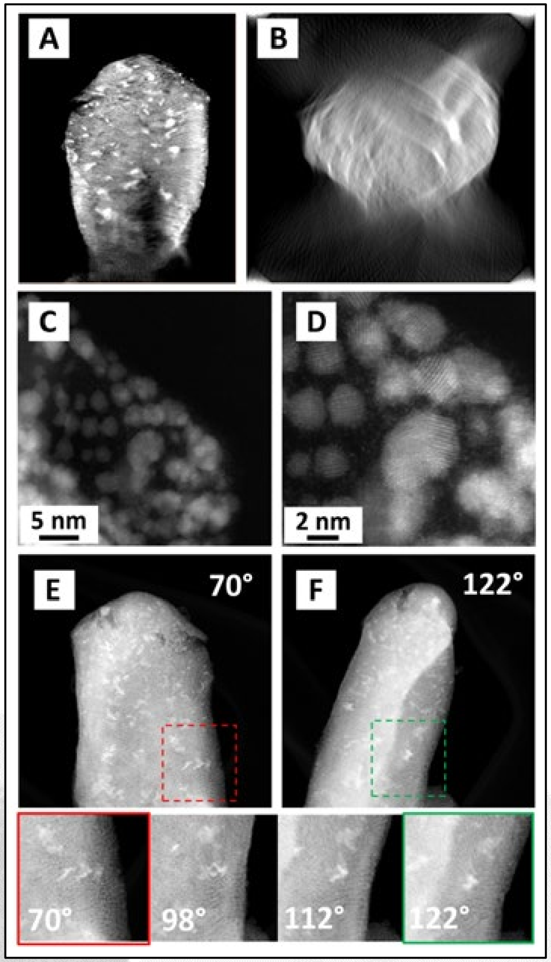


Active phase deposition



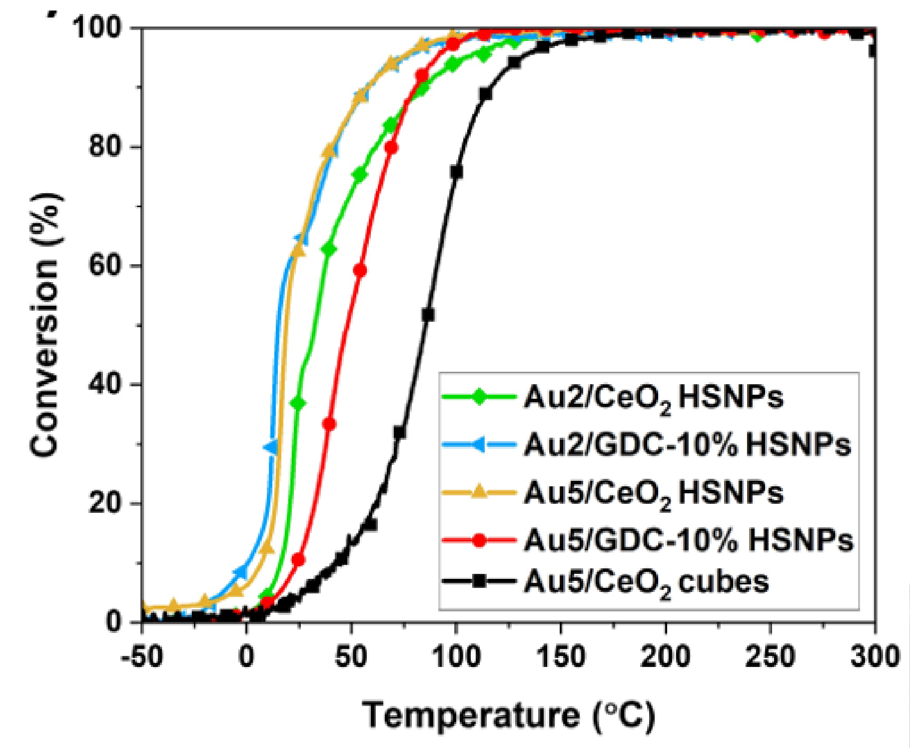
Electron Tomography

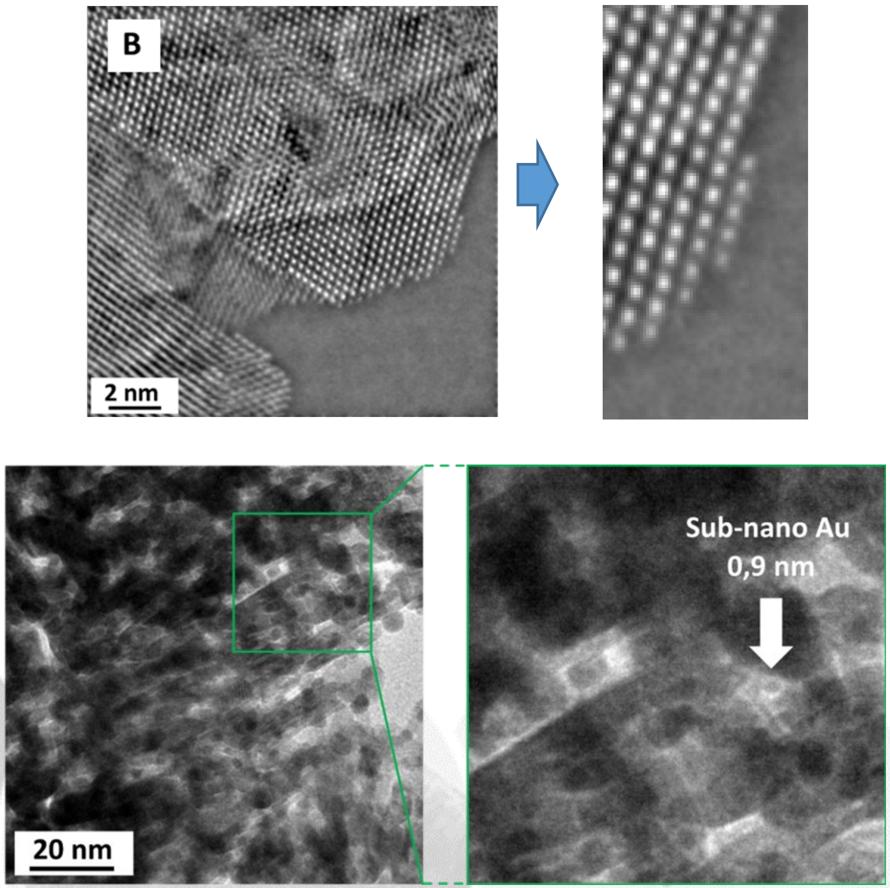
Au5/ CeO_2 HSNPs



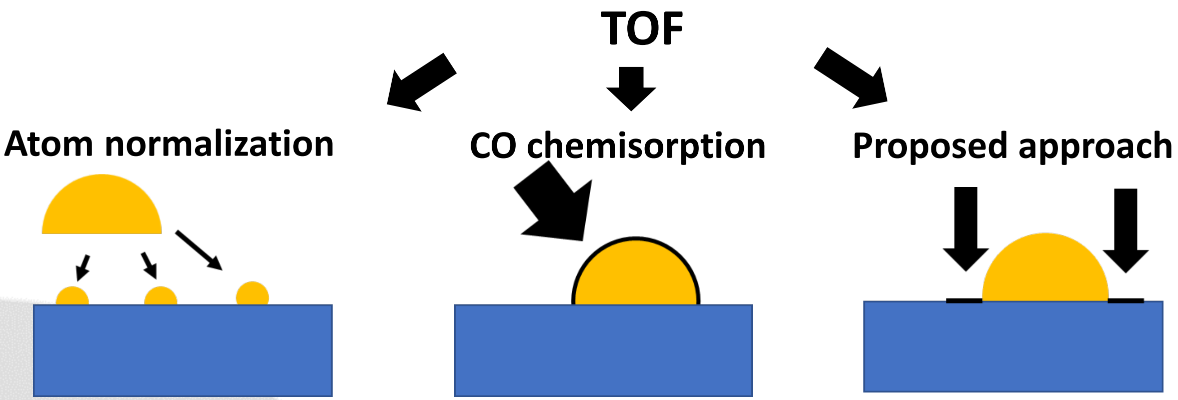
Catalytic activity

CO oxidation

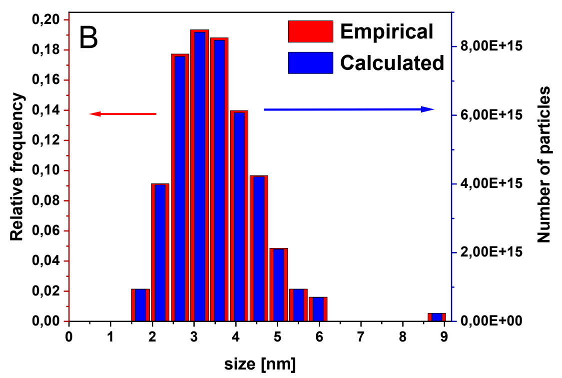




Synergistic effect of chemical composition and structural hierarchy



TEM data + simulation



$$TOF = \frac{reaction\ rate}{number\ of\ active\ sites}$$

Synergistic effect of chemical composition and structural hierarchy

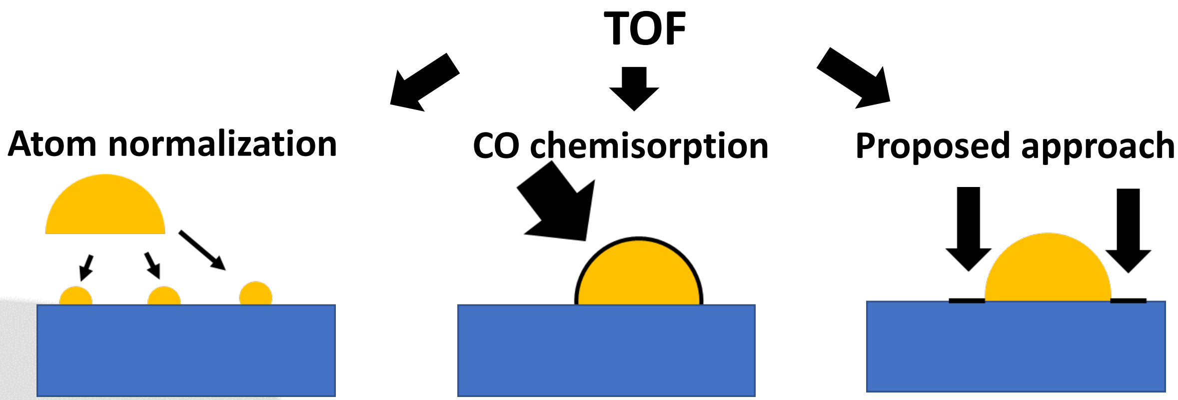


Table 2 Kinetic data for propane oxidation

Catalyst	$r_{300°C} \times 10^2$ ($mol_{CO}\ g_{Au}^{-1}\ h^{-1}$)	E_a (kJ/mol)	$TOF_{300°C} \times 10^2$ [a] (s^{-1})	$TOF_{300°C} \times 10^2$ [b] (s^{-1})	Reference
Au0.5/CeO_2 HSNPs	1.07	63	2.18	-	This work
Au2/CeO_2 HSNPs	0.77	49	0.43	0.49	This work
Au5/CeO_2 HSNPs	0.61	55	0.14	0.43	This work
Au5/CeO_2 cubes	0.76	57	0.78	0,26	This work
Au5/CeO_2 NPs	0.04	65	0.01	-	This work
Au0.5/GDC-10% HSNPs	1.13	52	2.14	-	This work
Au2/GDC-10% HSNPs	1.85	49	1.05	1.18	This work
Au5/GDC-10% HSNPs	0.72	51	0.17	1.11	This work
Au5/GDC-10% cubes	0.96	47	1.04	0,61	This work
Au5/GDC-10% NPs	0.24	61	0.11	-	This work

Conclusions

1. Hydrothermal synthesis + thermolysis + deposition-precipitation -> catalyst with structural hierarchy and superior activity

2. Gd^{3+} increases activity and stability of the architecture of catalyst

3. Gd^{3+} influences the temperature-dependent evolution of the architecture

4. Synergistic effect of structural hierarchy and doping in propane oxidation

Thank you for your attention!

Literature

Woźniak, P., Małecka, M.A., Chinchilla, L., Trasobares, S., 3D hierarchically structured $Ce_{1-x}Gd_xO_{2-x/2}$ mixed oxide particles: the role of microstructure, porosity and multi-level architecture stability in soot and propane oxidation, Mater. Res. Bull., 2022, 151, 111816.

Woźniak, P., Małecka, M.A., Kraszkiewicz, P., Miśta, W., Bezkrovnyi, O., Chinchilla, L., Trasobares, S., Confinement of nano-gold in 3D hierarchically structured gadolinium- doped ceria mesocrystal: synergistic effect of chemical composition and structural hierarchy in CO and propane oxidation. Catal. Sci. Technol., 2022, 12, 7082-7113.

Woźniak P., Miśta W., Małecka M.A., Function of various levels of hierarchical organization of porous $Ce_{0.9}REE_{0.1}O_{1.95}$ mixed oxides in catalytic activity, CrystEngComm, 2020, 22, 5914-5930.

TNA user feedback

Advanced microscopy characterization of innovative ceramic coatings for hydrogen technologies

Elisa Zanchi
Politecnico di Torino, Italy

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

Outline

- Network and collaborations around **ESTEEM3 project**
- The **CeramECS** project: “*Glass-ceramic and ceramic materials for energy conversion and storage*”
- Motivation: **Solid Oxide Cell** technologies for **hydrogen energy**
  - Ceramic protective **coatings**
  - Electrophoretic deposition (**EPD**)
  - Advanced **TEM** characterization
- Further activities: the **GlaMater** project
- Concluding remarks

Network and collaborations

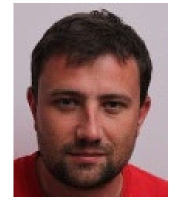
EPD processing

Politecnico di Torino

F. Smeacetto, E. Zanchi

Department of Applied science and Technology, Institute of Physics, GLANCE Group, Turin, Italy

AGH



G. Cempura

Advanced microscopy characterization

International Centre of Electron Microscopy for Materials Science at the AGH-UST, Krakow, Poland

KMM.VIN

FAU FRIEDRICH-ALEXANDER UNIVERSITÄT ERLANGEN-NÜRNBERG TECHNISCHE FAKULTÄT

Electrophoretic deposition expertise

Erlangen, Germany

A.R. Boccaccini

sunfire

H. Javed

Industrial partner for EU projects Stack testing

Dresden, Germany

IREC Shaping Energy for a Sustainable Future

Barcelona, Spain

EPD upscaling

A.G. Sabato

POLITECHNIKA GDAŃSKA

Gdansk, Poland

Testing & characterisation

S. Molin, J. Ignaczak

GrInHy Green Industrial Hydrogen

FCH FUEL CELLS AND HYDROGEN JOINT UNDERTAKING

GA # 700300

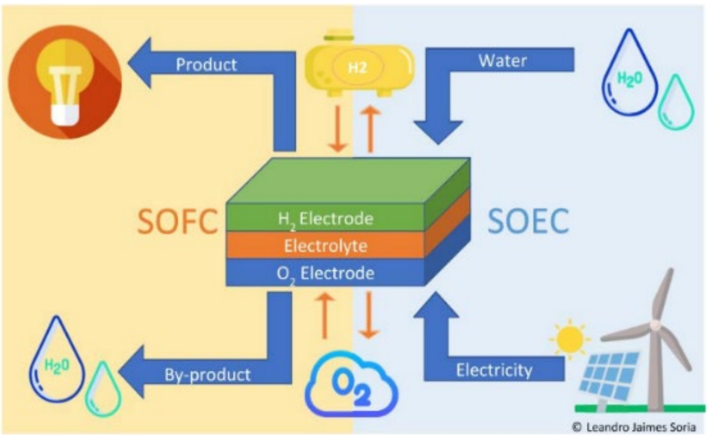
NewSOC

Clean Hydrogen Partnership

GA # 874577

Motivation: H_2 technologies

Solid oxide cells (SOCs) are electrochemical **energy conversion** devices operating at temperatures in the range of **600–850 °C**

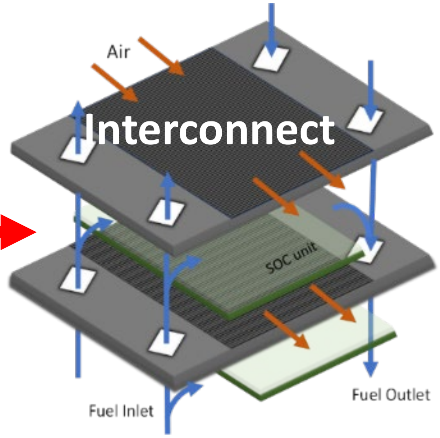


SOEC: Electrical energy ➔ Chemical energy (**H_2**)
SOFC: Chemical energy (**H_2**)➔ Electrical energy
rSOC: integration in renewable energy systems

Why SOCs?

✓**Efficient** energy conversion technology
✓**Clean**: Low GHGs emissions
✓Reduction of **CO_2** fingerprint

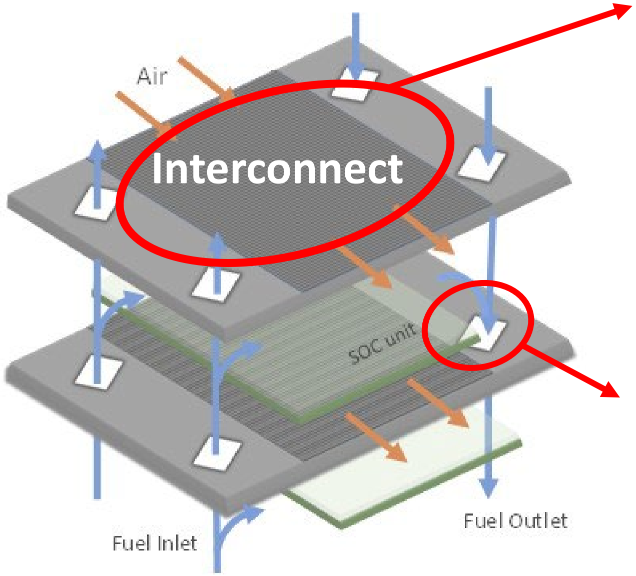
30 cells stack,
Sunfire GmbH



SOC stack

Ferritic stainless-steels are widely used as **interconnects** SOC **stacks**

- Electrical connection
- Separation between oxidizing/reducing atmosphere



HT corrosion

Progressive oxidation limits the electric conductivity

Volatile Cr-compounds degrade the cell performance

I) Deposition of a ceramic protective **coating**

CeramECS

Gas tightness

Need to completely avoid gas leakage

Suppress undesired reactions

NewSOC Clean Hydrogen Partnership

Grant # 874577 - NewSOC

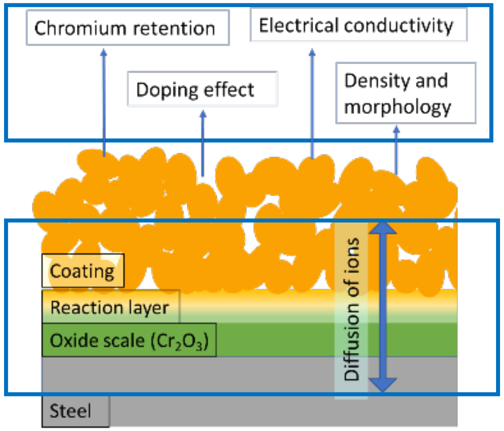
II) Design of **innovative interface** concepts

HT corrosion

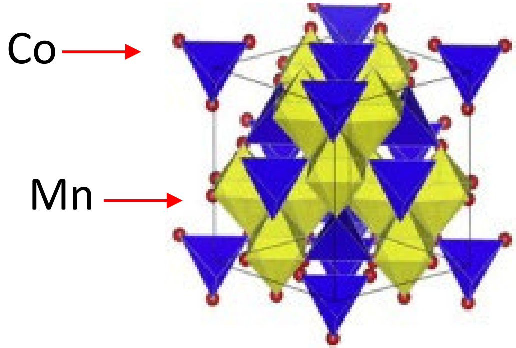
Applying a **protective coating** on the steel is a promising approach to extend the interconnect life and avoid cathode poisoning

The coating must:

- Reduce the oxidation rate of the steel
- Reduce Cr-evaporation
- Ensure high electrical conductivity
- Compatible thermal expansion coefficient (11-13 10^{-6} K^{-1})



E. Zanchi et al., ***J. Power Sources.*** 455 (2020) 227910.



Xu et al, **Science** 21 (2019) 19

The **Mn-Co spinel family** (MCO) has been shown to be particularly promising

The functional properties can be improved by

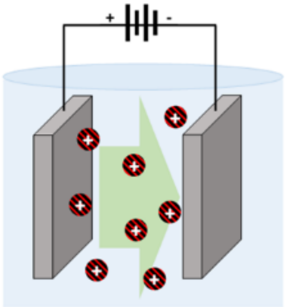
✓ Modifications with additional elements – **doping**

✓ Optimized **deposition and sintering**

Electrophoretic deposition

Electrophoresis : motion of charged particles in a suspension under the influence of an electric field

Deposition: coagulation of particles to a dense mass



E. Zanchi, et al., ***Mater. Lett.*** 286 (2021) 129229

Hamaker's equation:

$$m = C_s \mu S E t$$

m= mass deposited at time t on surface S
Cs = concentration in the suspension
μ = electrophoretic mobility
E = electric field applied

$$\mu = \frac{\varepsilon\zeta}{4\pi\eta} = \frac{\nu}{E}$$

ε = dielectric constant of the liquid.
η = viscosity of the liquid.
ζ = Zeta potential.
ν = particle velocity.

Experimental challenges:

- Stability of the suspension
- Deposition parameters
- Sintering process

Compared to other techniques, **EPD**

- Has a simple and **adaptable set-up**
- Is a **fast** and **versatile** technique
- Reduces processing **time and cost**

EPD set-up @ POLITO

1) **Upscale** of the electrophoretic deposition set-up

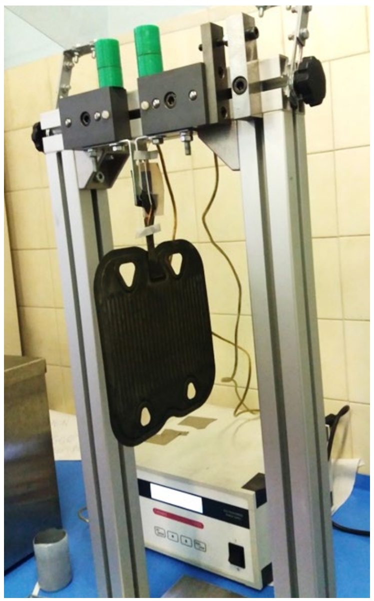
2) Modification of the spinel by **electrophoretic co-deposition**

CeramECS

Glass-ceramic and ceramic materials for energy conversion and storage

EPD apparatus upscale

…to validate EPD for real dimension devices



EPD adapted to a 3-electrode set-up

~20x20 cm surface successfully deposited

Journal of the European Ceramic Society 41 (2021) 4496–4504

Contents lists available at ScienceDirect

Journal of the European Ceramic Society

journal homepage: www.elsevier.com/locate/jeurceramsoc

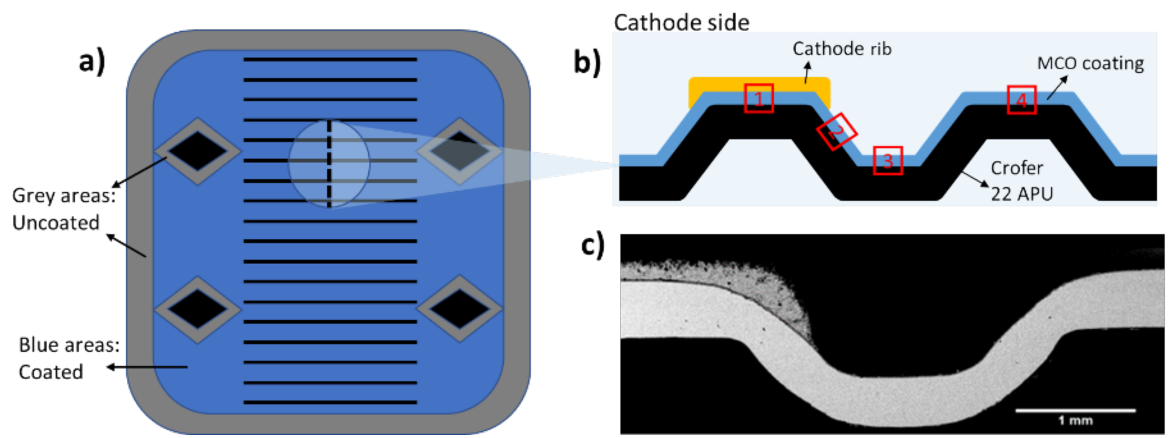
Mn-Co spinel coatings on Crofer 22 APU by electrophoretic deposition: Up scaling, performance in SOFC stack at 850 °C and compositional modifications

A.G. Sabato[a], E. Zanchi[b], S. Molin[c], G. Cempura[d], H. Javed[e], K. Herbrig[e], C. Walter[e], A. R. Boccaccini[f], F. Smeacetto[b,*]

GA # 700300

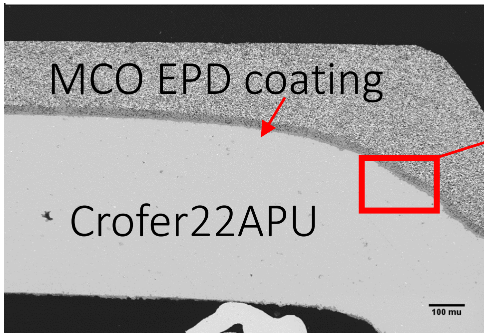
SOC Stack test: 850 °C, 3000 h

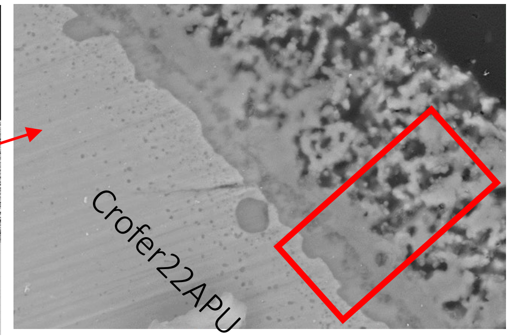
The EPD deposited Mn–Co spinel coating showed improved stability



Mn-Co spinel coating

Advanced TEM post-mortem characterization



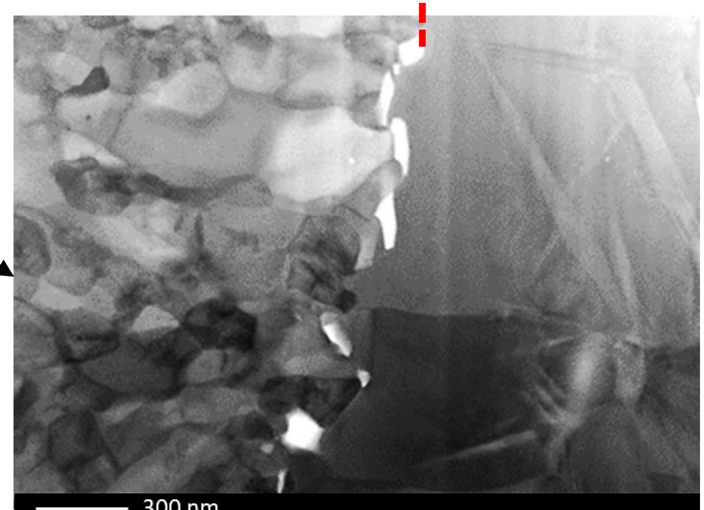


Fib lamella:
to investigate reactions at the **steel-coating interface** and developed **phases**

Crofer22APU | **Oxide Scale** | **Coating**

Overview of the FIB lamella

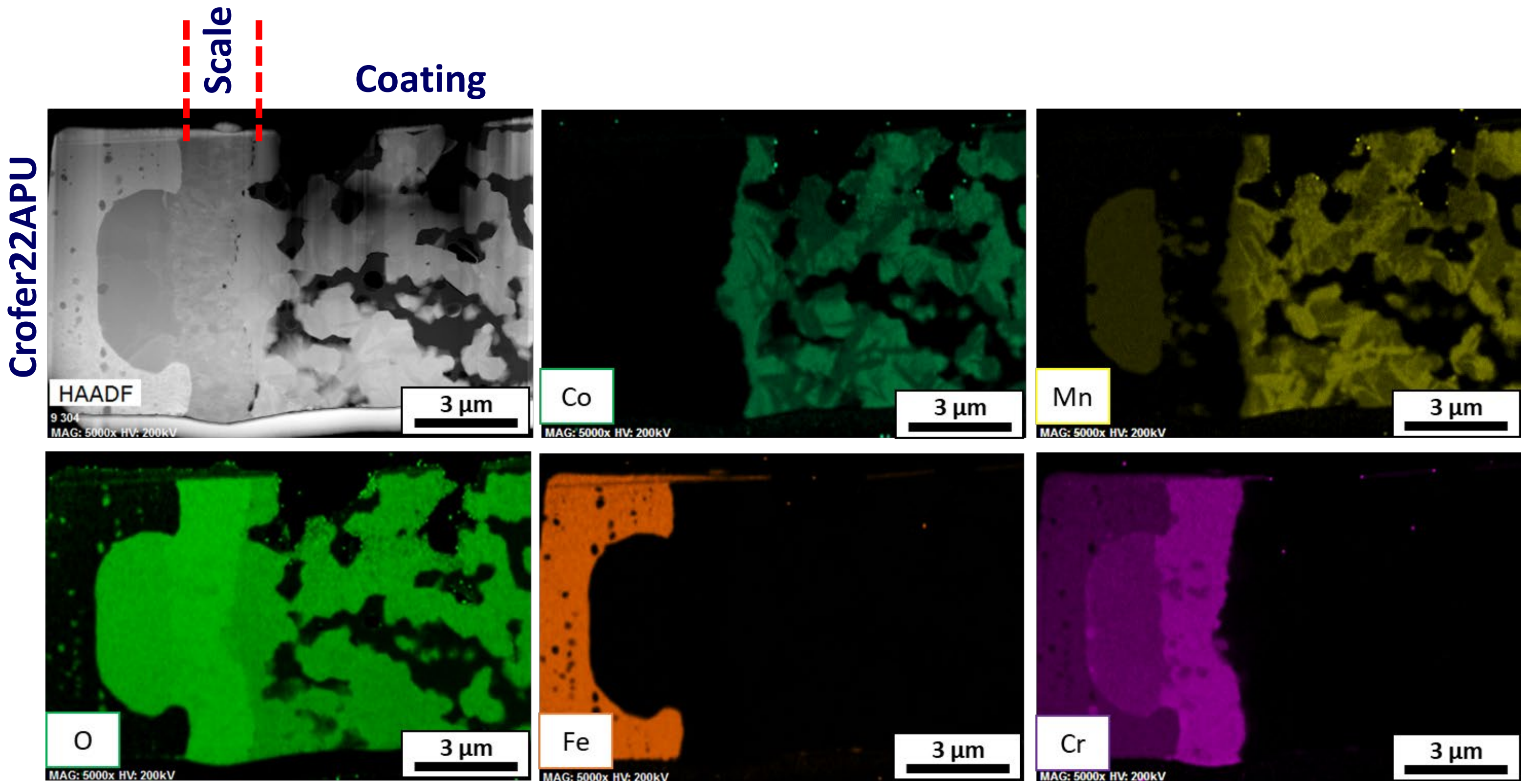
Oxide scale | **MCO coating**



Magnification of **oxide scale-coating** interface

Mn-Co spinel coating

Overview of the FIB lamella from **coated interconnect**



EDS map of the lamella:

Different Mn and Co distribution in the coating

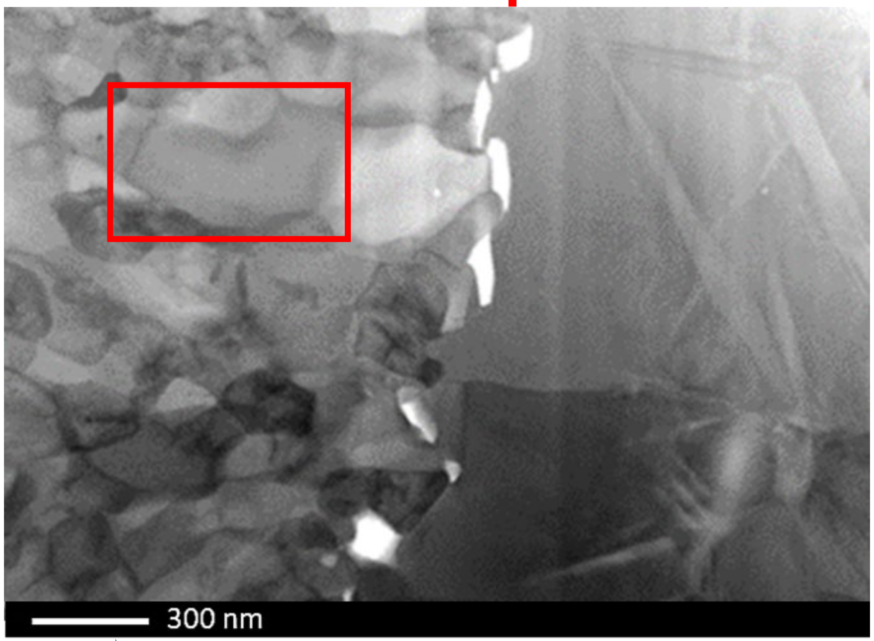
Cr confined in the oxide scale and absence of "reaction layer"

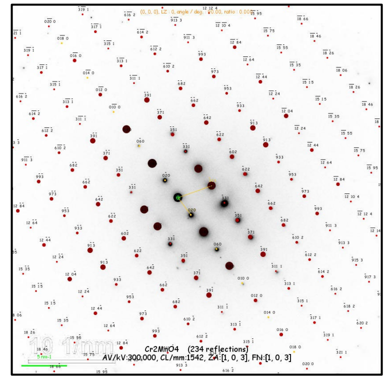
Mn-rich sub scale oxidation

Mn-Co spinel coating

Advanced **TEM** characterisation of **oxide scale–coating interface**

Oxide scale | **Coating**

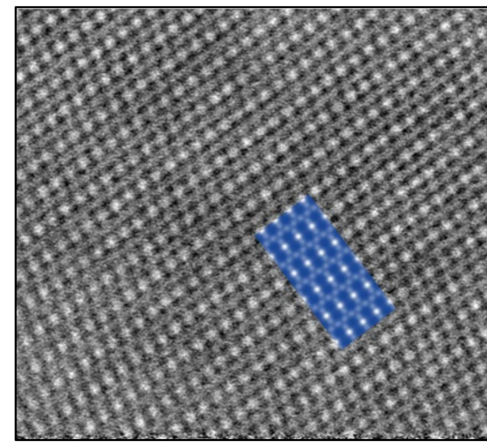




Simulated SAED of **Cr_2MnO_4**, superimposed on experimental one

EDS composition

O	Cr	Mn	Fe
59.2	30.4	10.0	0.2



Experimental HAADF –STEM and Simulated HAADF STEM

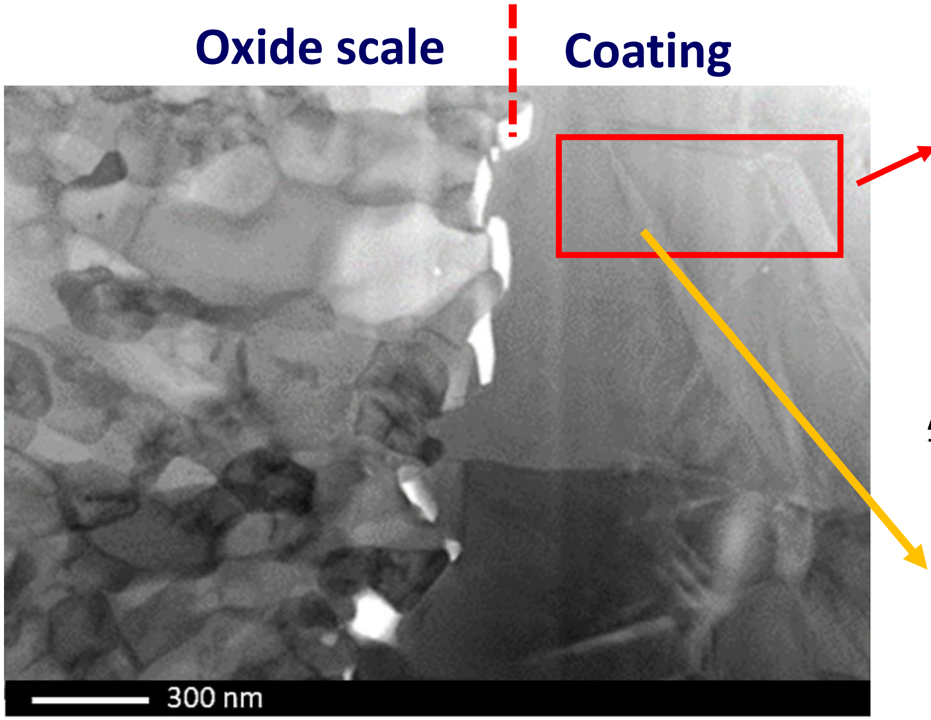
Mn-rich oxide scale grain

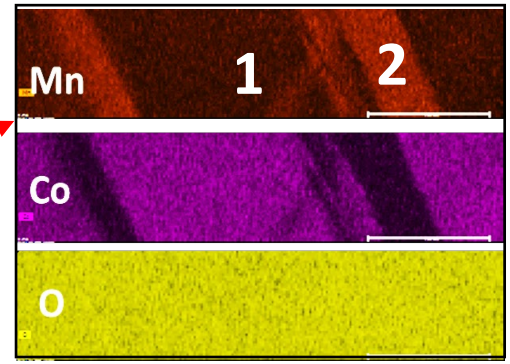
Remarkable diffusion of Mn leading to **Cr-Mn spinel formation**

Increased the electric conductivity and lower Cr evaporation compared to pure chromia scale

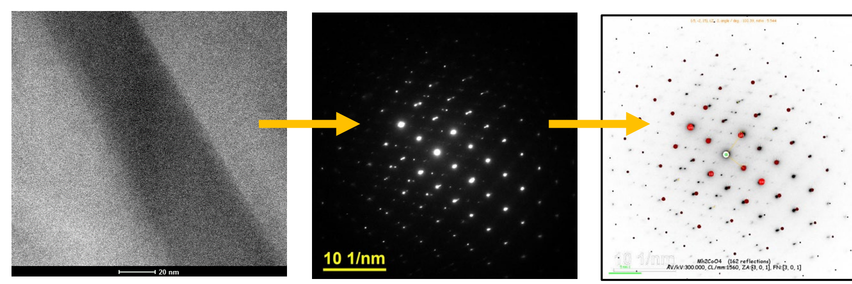
Mn-Co spinel coating

Advanced **TEM** characterisation of **oxide scale–coating interface**





At.%	O	Cr	Mn	Co	Fe
1	53.5	0.6	17.6	27.8	0.6
2	54.5	1.3	29.1	14.8	0.3



Dual phase structure of the Mn-Co spinel coating

Cobalt-rich grains: $MnCo_2O_4$ cubic structure

Mn-rich elongated grains: Mn_2CoO_4 tetragonal structure

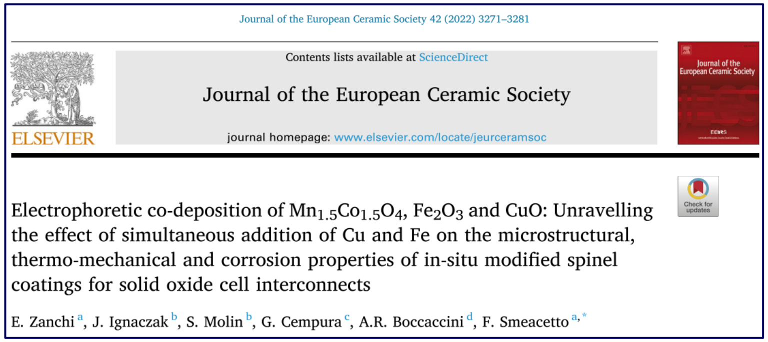
Simulated SAED of Mn_2CoO_4 superimposed on exp. one [ZA 301]

CeramECS

Glass-ceramic and ceramic materials for energy conversion and storage

Fe-Cu co-doping of the Mn-Co spinel coating

…to improve the functional properties of the Mn-Co spinel by addition of transition metals


Journal of the European Ceramic Society 42 (2022) 3271–3281
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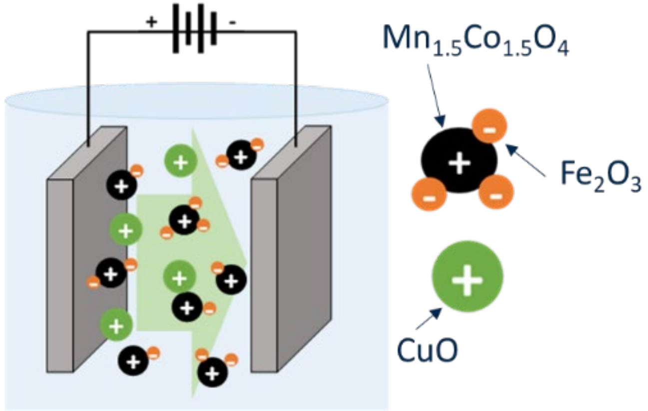
journal homepage: www.elsevier.com/locate/jeurceramsoc

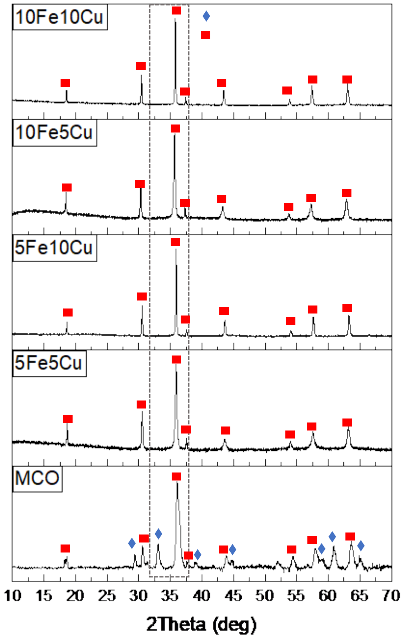
Electrophoretic co-deposition of $Mn_{1.5}Co_{1.5}O_4$, Fe_2O_3 and CuO: Unravelling the effect of simultaneous addition of Cu and Fe on the microstructural, thermo-mechanical and corrosion properties of in-situ modified spinel coatings for solid oxide cell interconnects

E. Zanchi[a], J. Ignaczak[b], S. Molin[b], G. Cempura[c], A.R. Boccaccini[d], F. Smeacetto[a,*]

Electrophoretic co-deposition of 3 precursors: cathodic deposition



- Water-ethanol suspension
- Deposited on Crofer22APU
- Optimised deposition at 50 V for 30 s
- Four different spinel compositions



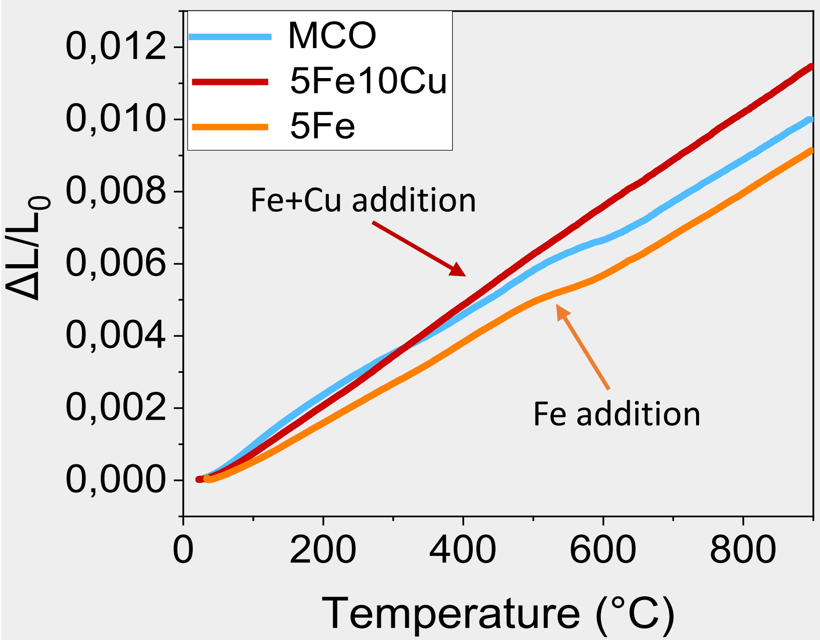
Two-step Reactive sintering:
Reduction
Re-oxidation

Successful **in-situ** doping: Stabilization of the **cubic spinel structure**

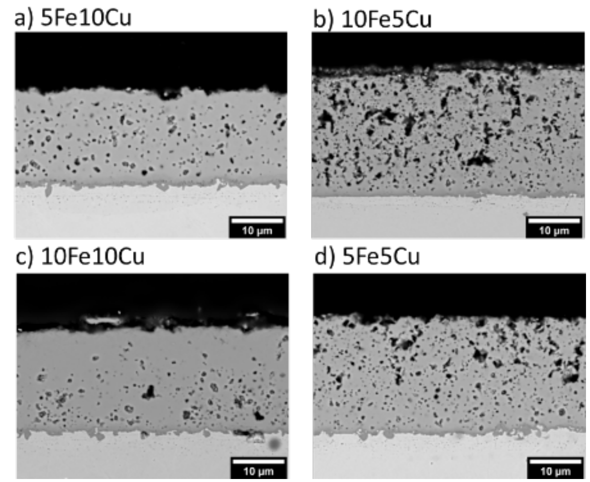
Doped spinel coatings

Characterization of **Mn-Co spinel** coatings with **Fe-Cu addition**

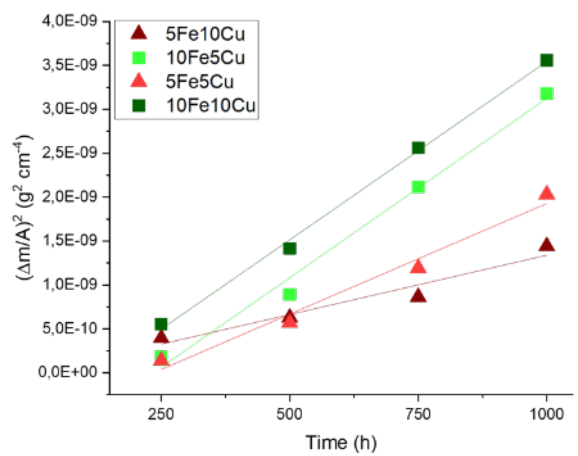
Assessment of dilatometric properties of Fe-Cu doped spinels



- ✓ Improved spinel stability
- ✓ Synergic effect of Cu and Fe



- ✓ Cu addition boosts densification
- ✓ No Cr diffusion during aging

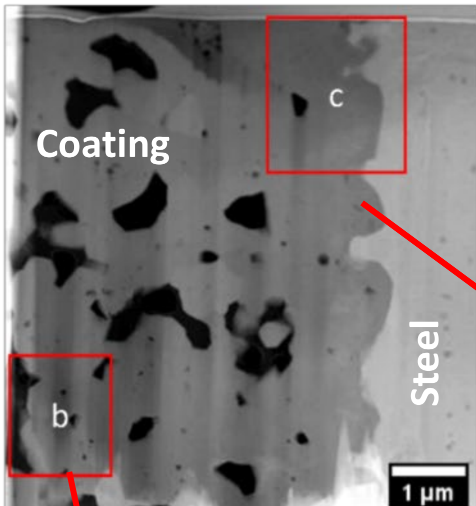


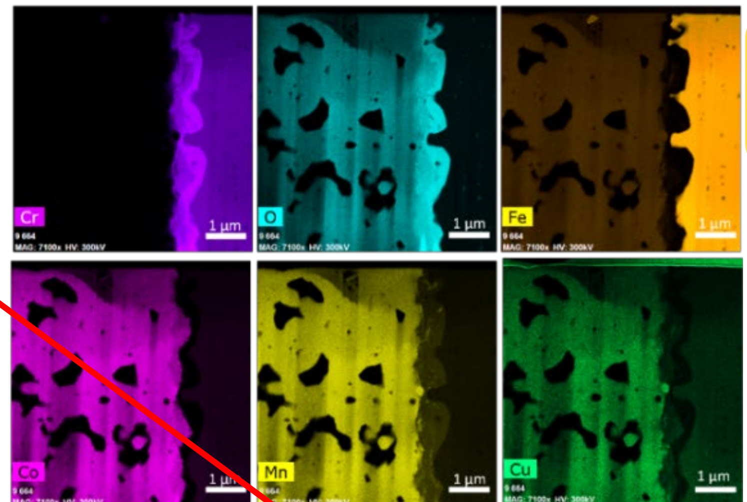
- ✓ Better oxidation resistance of Fe-Cu doped coatings compared to base Mn-Co spinel

Doped spinel coatings

Advanced TEM characterization of doped coatings

a)





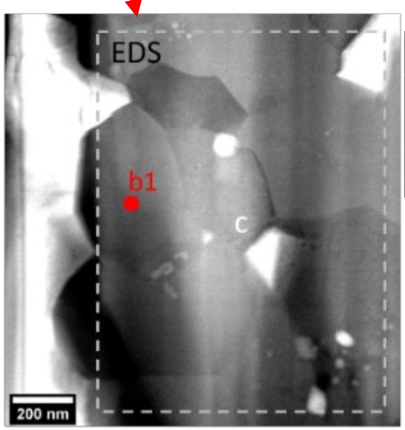
10Fe10Cu sample:
Highest amount of dopants

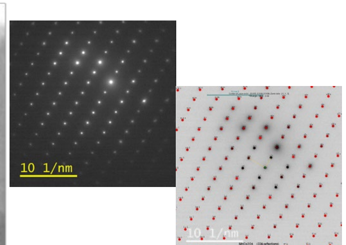
High coating densification

Fe and Cu homogeneously distributed in the coating

Thin oxide scale

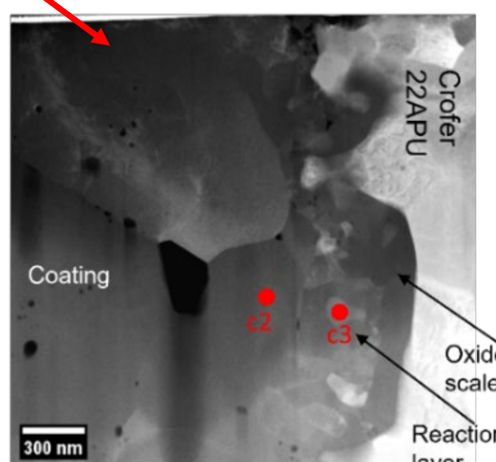
b)

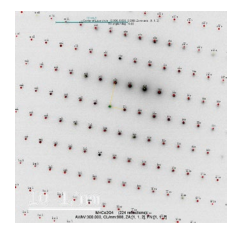




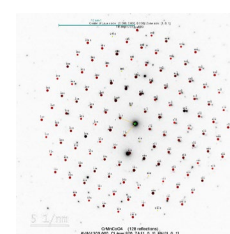
Point b1:
Solution with **$MnCo_2O_4$**
Zone axis 112

c)





Point c2:
Solution with
$MnCo_2O_4$
Zone axis 112

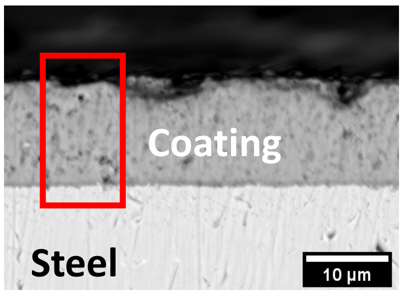


Point c3:
Solution with
$CrMnCoO_4$
Zone axis 101

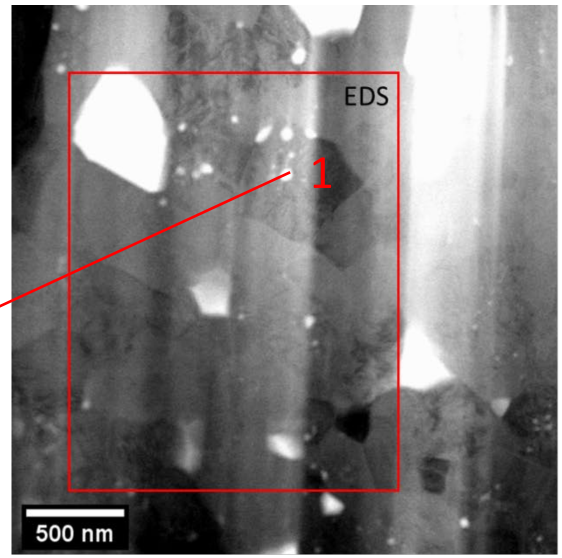
Doped spinel coatings

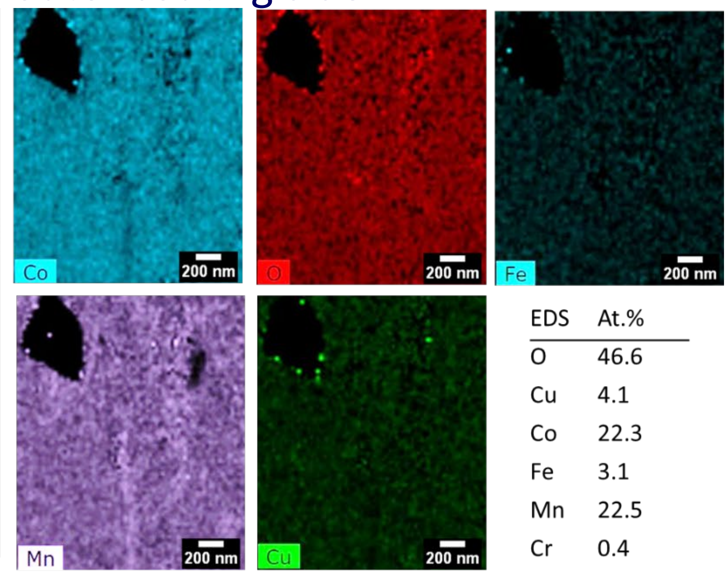
Advanced TEM characterization of **doped coatings**

5Fe5Cu sample: lowest amount of dopants

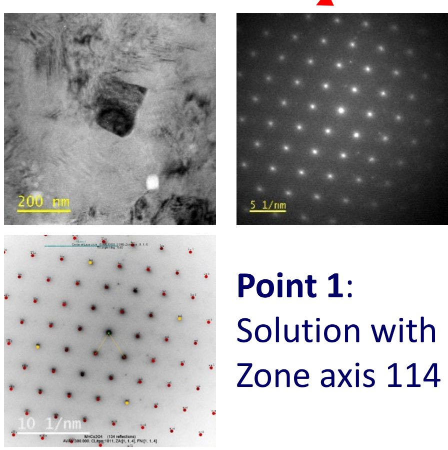


View of the lamella from the outer coating side





EDS	At.%
O	46.6
Cu	4.1
Co	22.3
Fe	3.1
Mn	22.5
Cr	0.4

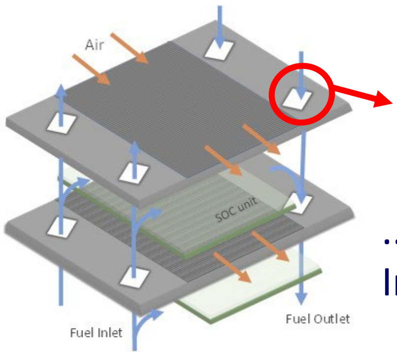


Point 1:
Solution with **$MnCo_2O_4$**
Zone axis 114

Homogeneous distribution of Fe and Cu in the Mn-Co spinel
Fe+Cu addition: synergic effect
in suppressing the double-phase spinel formation

Further activities in ESTEEM3

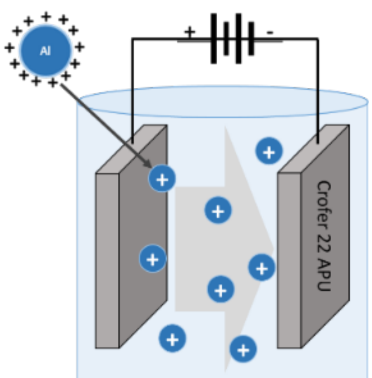
GlaMater “Advanced glass and ceramic materials for energy applications”



Gas tightness

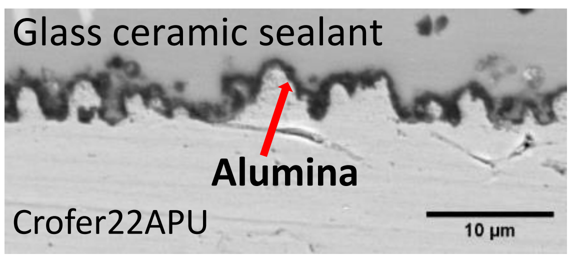
II) Design of **innovative interface** concepts

.. To minimize reactions between steel and the sealant
Improve thermo-mechanical properties of the joint



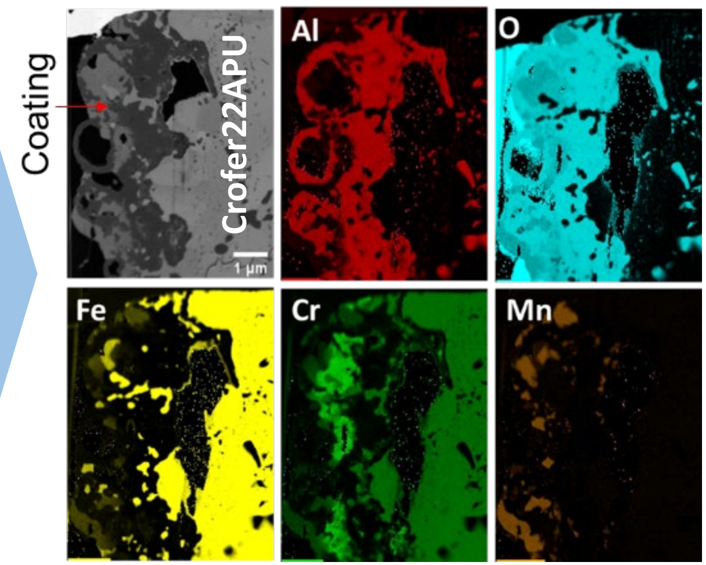
HT (air)

Successful EPD modification:



Alumina layer
with enhanced surface roughness

TEM lamella of alumina coating @1000 °C



NewSOC · Clean Hydrogen Partnership · Co-funded by the European Union · sunfire · AGH

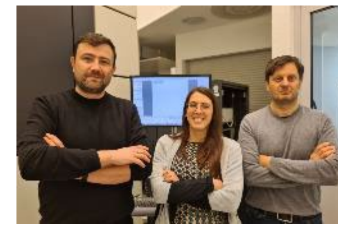
GlaMater (March 2023)

Concluding remarks

The **Transnational Access**
provided by **ESTEEM3 Project** enabled:

- The consolidation of fruitful **networking**
- Strengthening of **AGH-POLITO** collaboration
- **Publications** on high-impact journals
- **Training** on TEM characterization techniques
- Professional (and personal!) experience for **PhD** activity

Krakow, 2019



Krakow, 2023

Results and publications
on the **CeramECS** projects provided:

- ✓ A first in-depth characterization of **Mn-Co dual spinel structure** and oxide scale evolution after stack test at relevant conditions
- ✓ A first comprehensive assessment of **synergic effect of Fe-Cu** addition into the Mn–Co spinel structure
- ✓ Better understanding of coating degradation phenomena

…to improve the performance and durability of SOC devices and boost hydrogen technologies

Acknowledgments

Electrophoretic deposition, FAU, Erlangen, Germany,

This project has received funding from the **Fuel Cells and Hydrogen 2 Joint Undertaking** (**now Clean Hydrogen Partnership**) under Grant Agreement No 874577-NewSOC. This Joint Undertaking receives support from the European Union's Horizon 2020 Research and Innovation program, Hydrogen Europe and Hydrogen Europe Research.

Thank you for the attention

elisa.zanchi@polito.it

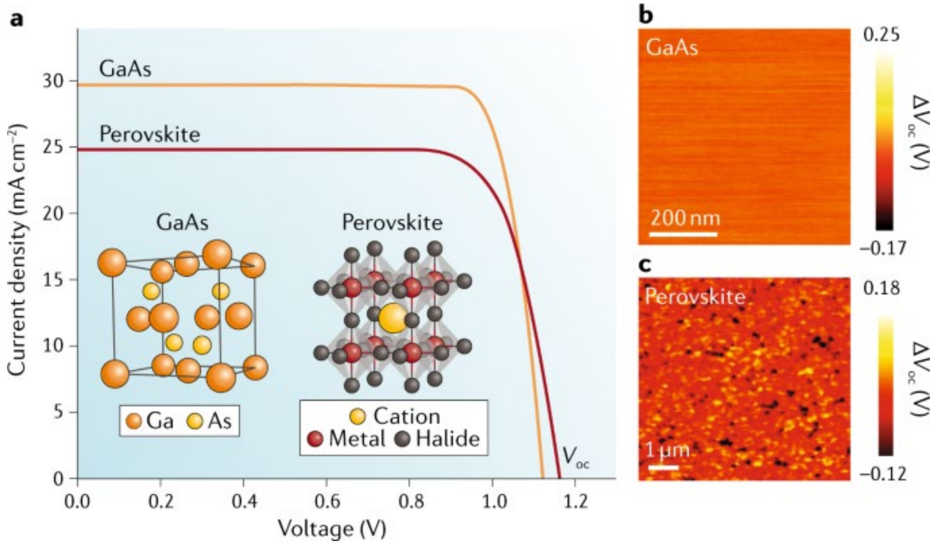
TNA user feedback

Hybrid composites for bright and stable light emission

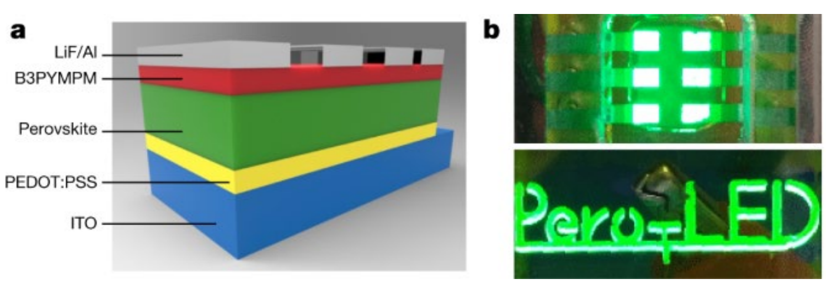
Dr. S.M. Collins – Lumi **University of Leeds**

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

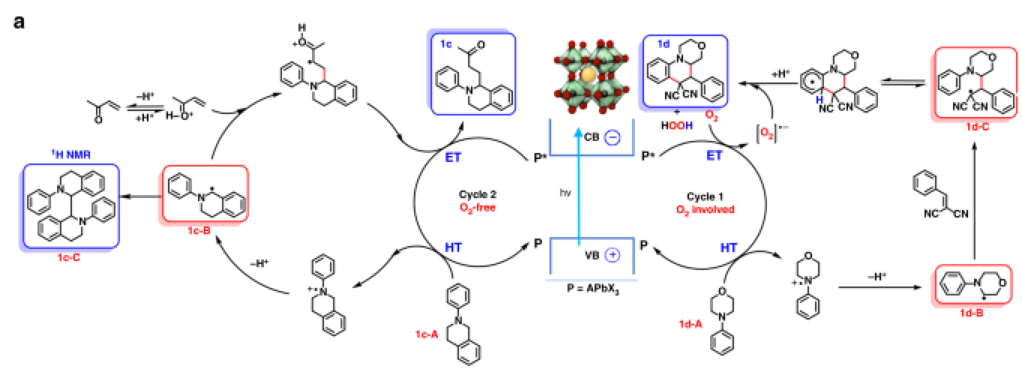
Lead halide perovskites (LHPs)



E.M. Tennyson, T.A.S. Doherty, S.D. Stranks *Nat. Rev. Mater.* **2019**, *4*, 573-587.

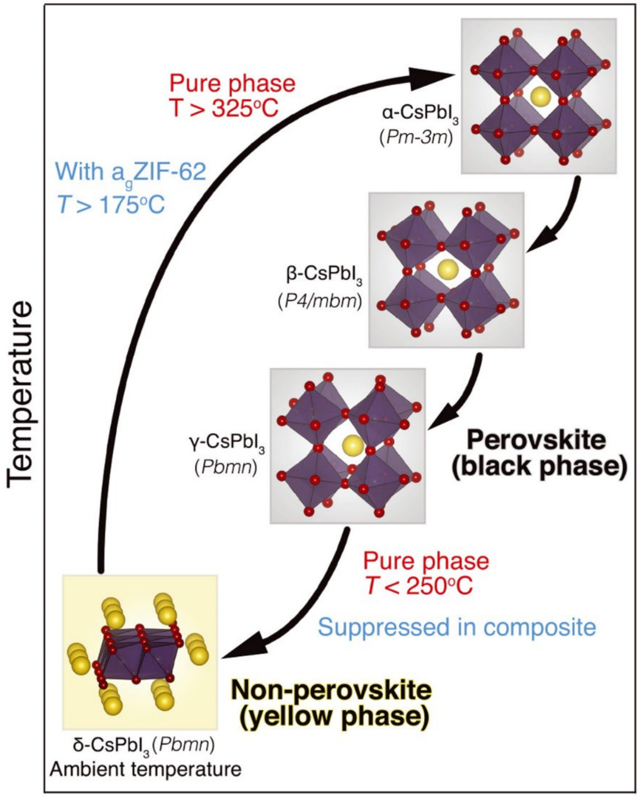


K. Lin et al. *Nature* **2018**, *562*, 245-248.



X. Zhu et al. *Nat. Commun.* **2019**, *10*, 2843.

Challenges in LHPs



Temperature-dependent crystal structures

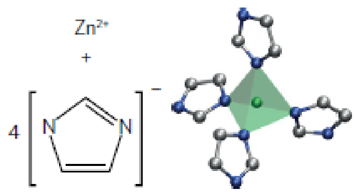
Degradation under exposure to light, heat, & water

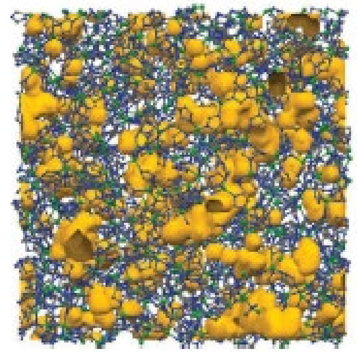
Phase separation/segregation (mixed ion designs)

Trap states from defects

MOF glasses: A 4th category

MOFs: Metal-organic frameworks



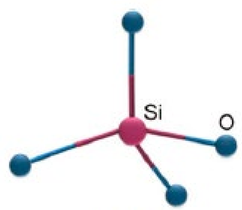


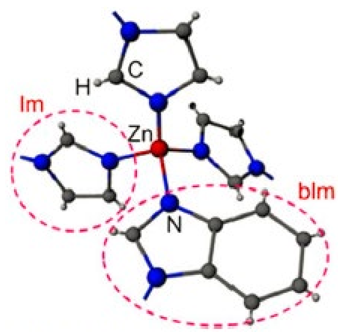
Pores highlighted in amber

ZIF-4

Heat treatment → **Melt-quenched glass**

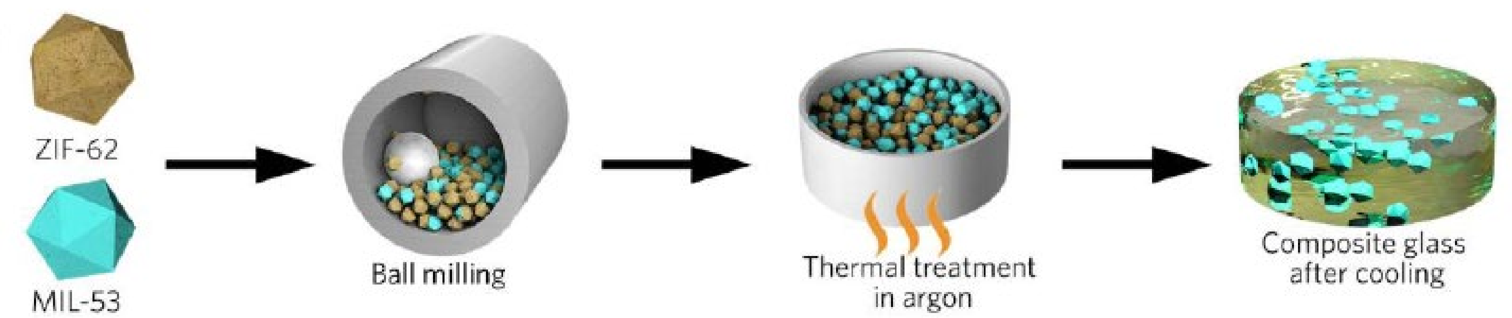
SiO_2

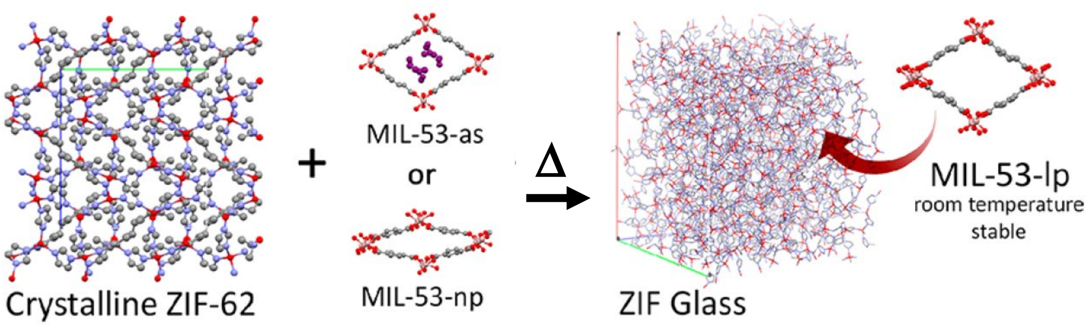


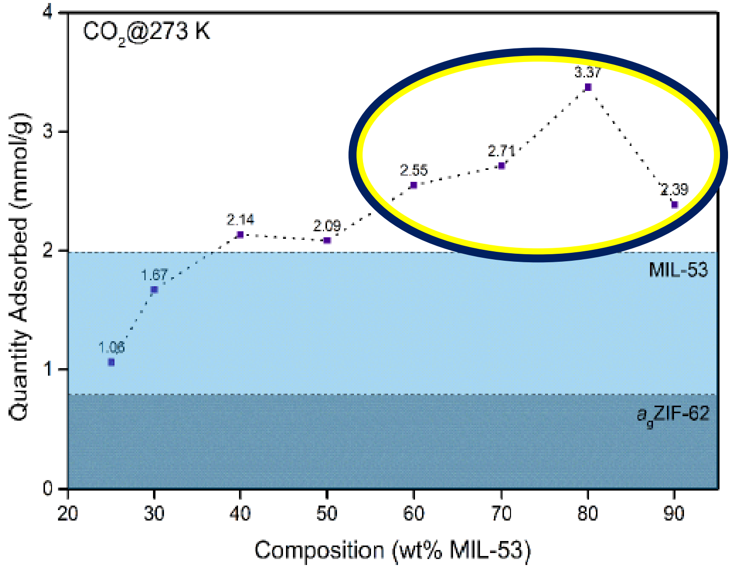


Qiao et al. *Sci. Adv.* **2018**, *4*, eaao6827.
Gaillac et al. *Nat. Mater*. **2017**, *16*, 1149-1154.

MOF crystal-glass composites

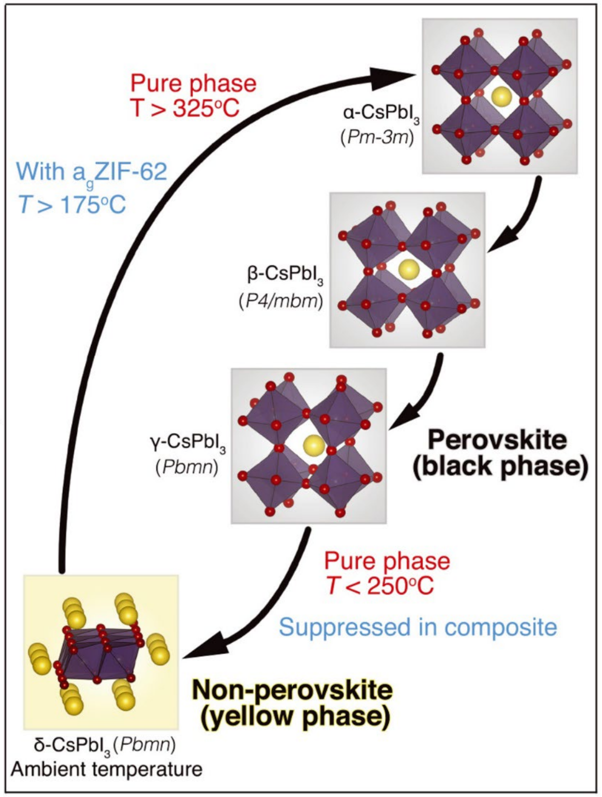


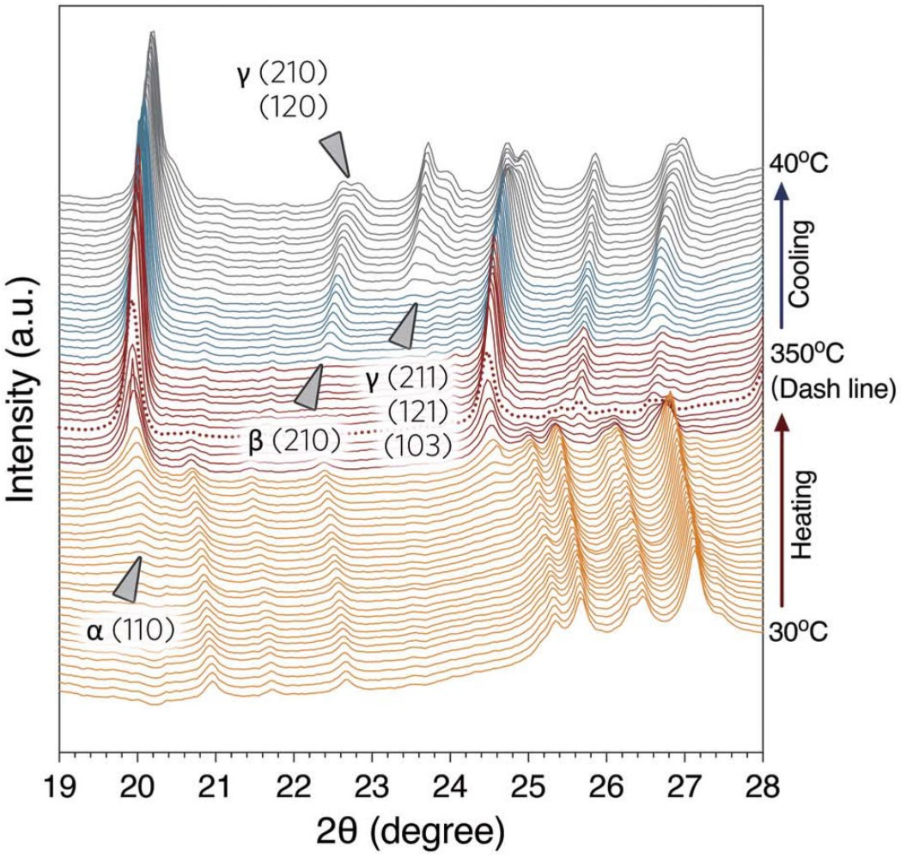




J. Hou et al. *Nat. Commun.* **2019**, *10*, 2580.
C.W. Ashling et al. *J. Am. Chem. Soc.* **2019**, *141*, 15641-15648.
S. Li et al. *Chem. Sci.* **2020**, *11*, 9910-9918.

Phase transitions in $CsPbI_3$





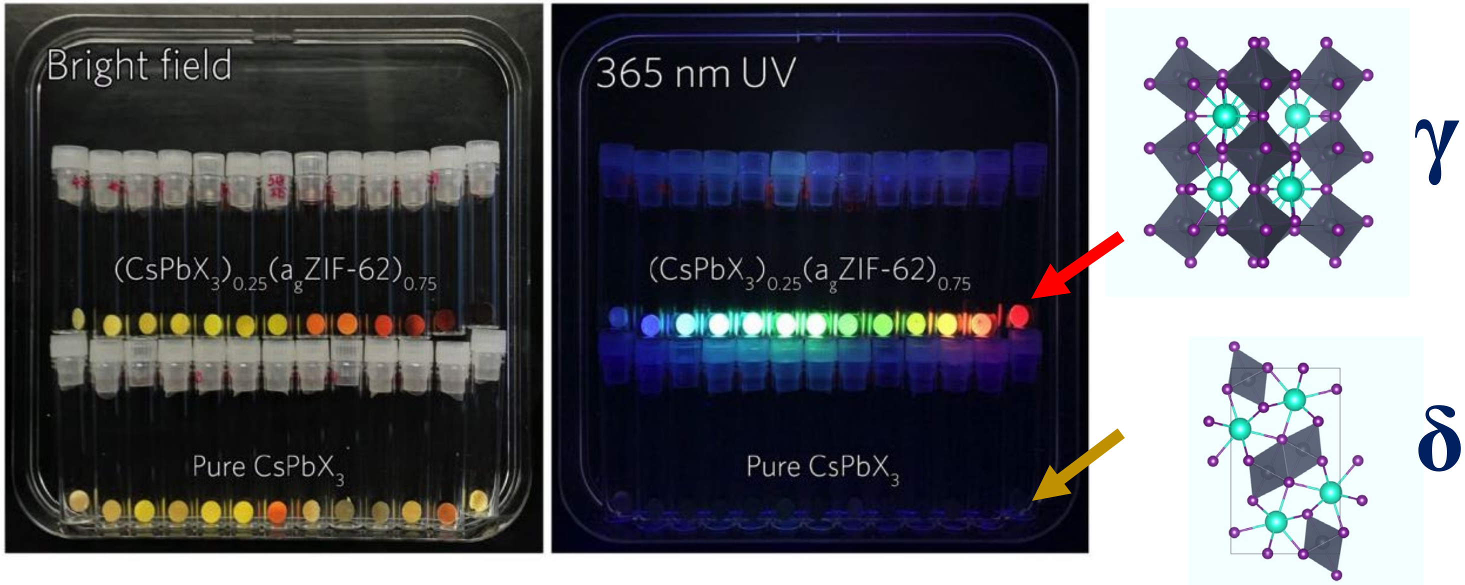
J. Hou et al. *Science*, **2021**, *374*, 621-625.

$CsPbX_3$ nanocrystals in $a_gZIF\text{-}62$

$CsPbI_3$

Glass

Physical mixture

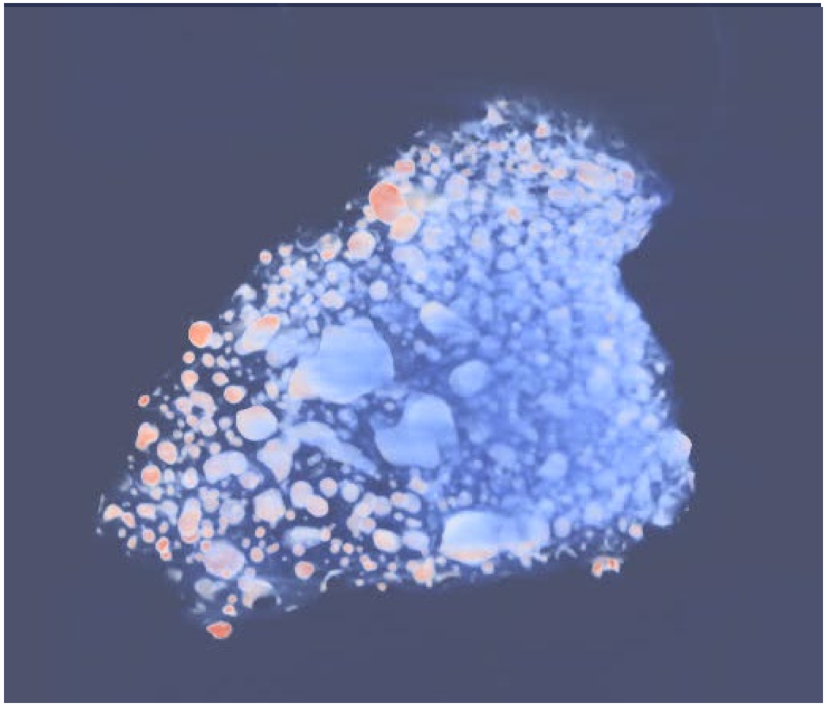


Composite formation favours bright luminescence across $CsPbX_3$ compositions

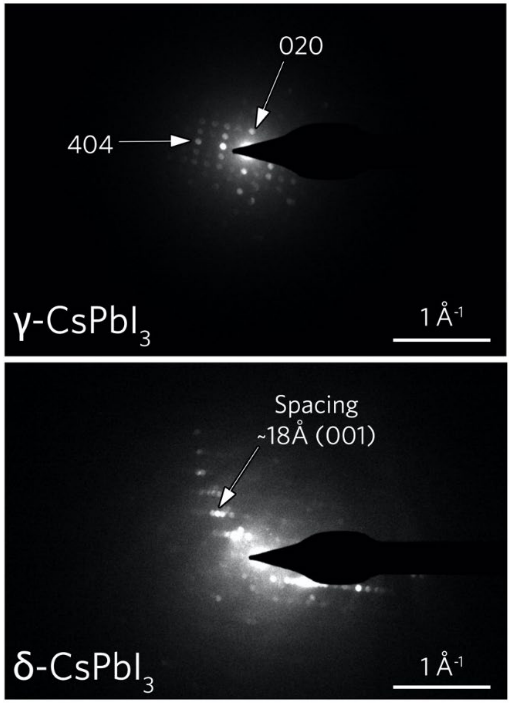
J. Hou et al. *Science*, **2021**, *374*, 621-625.

$(CsPbI_3)(a_gZIF\text{-}62)$

ADF tomography



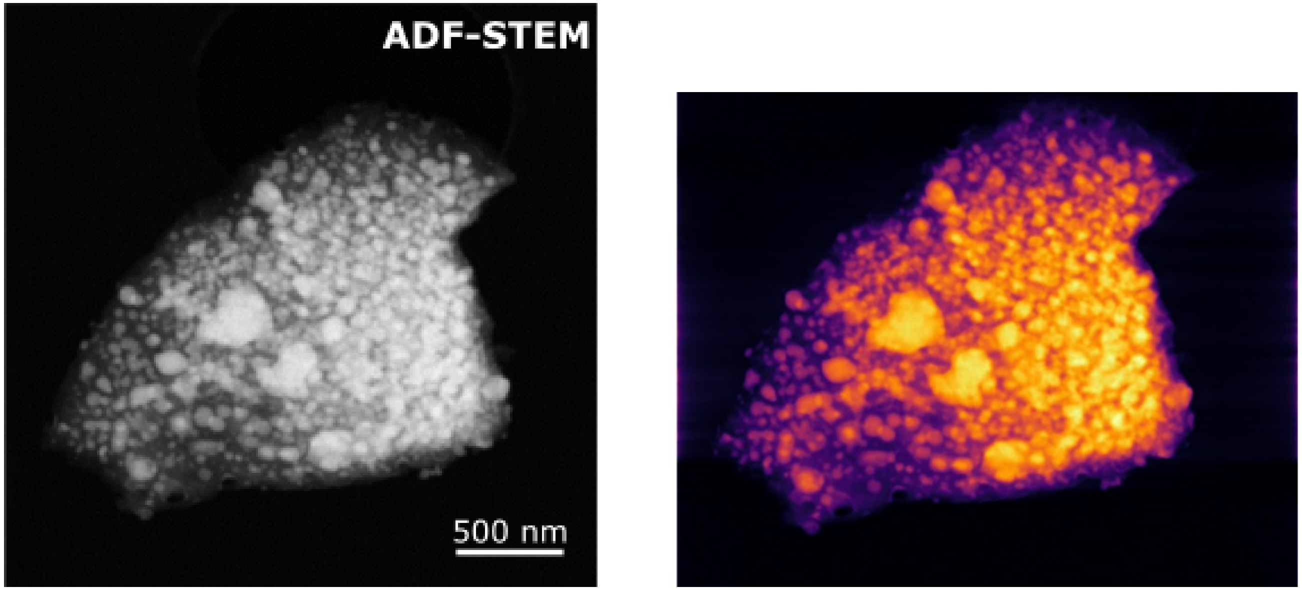
Nanobeam diffraction



'Pencil' beam: (1) Nanobeam point diffraction from individual grains
(2) Increased depth of focus for tomography of micron-wide glass shard

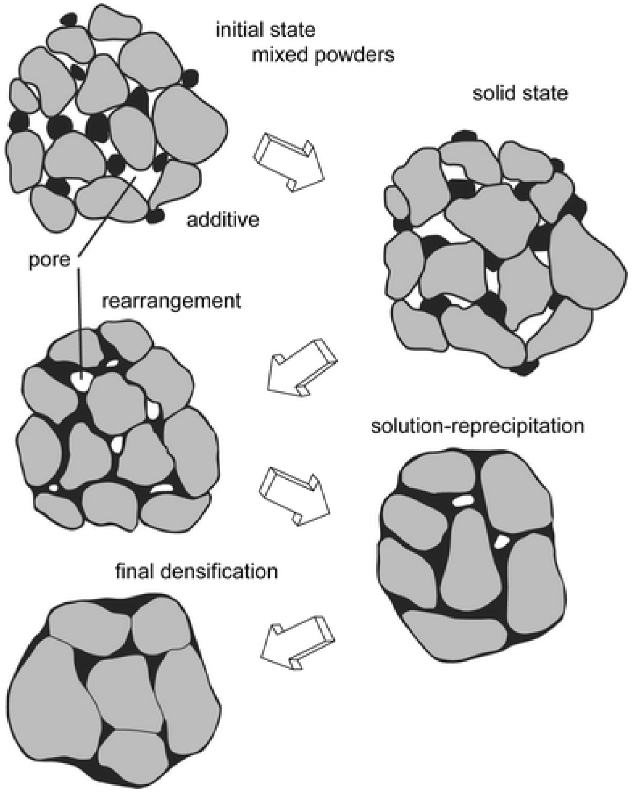
J. Hou et al. *Science*, **2021**, *374*, 621-625.

Liquid-phase sintering

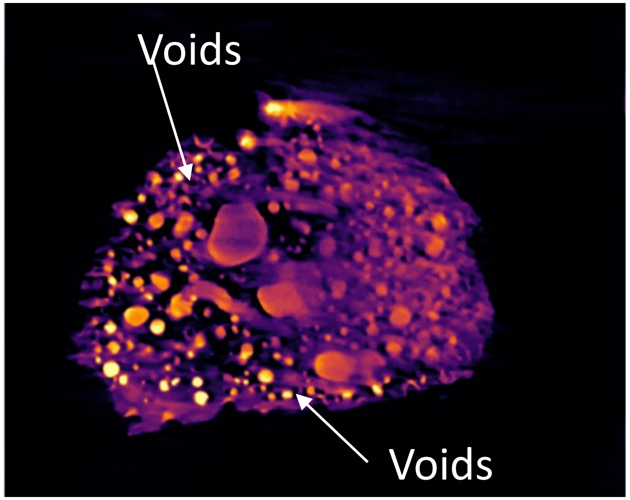


J. Hou et al. *Science*, **2021**, *374*, 621-625.

Liquid-phase sintering

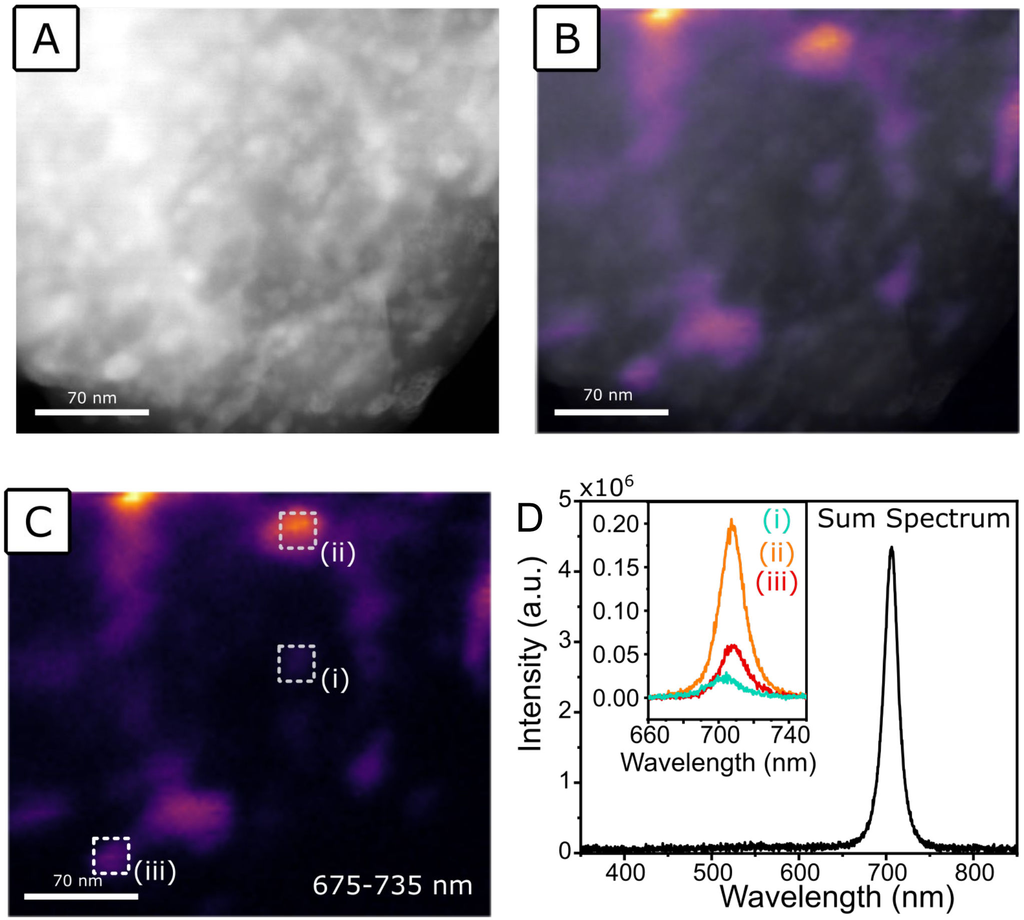


R.M. German, P. Suri, S. J. Park *J. Mater. Sci.* **2009**, *44*, 1-39.



J. Hou et al. *Science,* **2021**, *374*, 621-625.

CL from perovskite nanocrystals



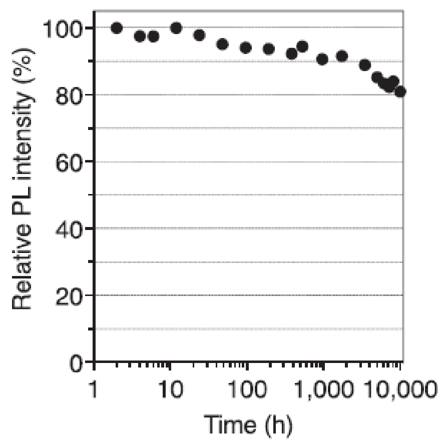
Narrow-band emission arises from embedded nanocrystals

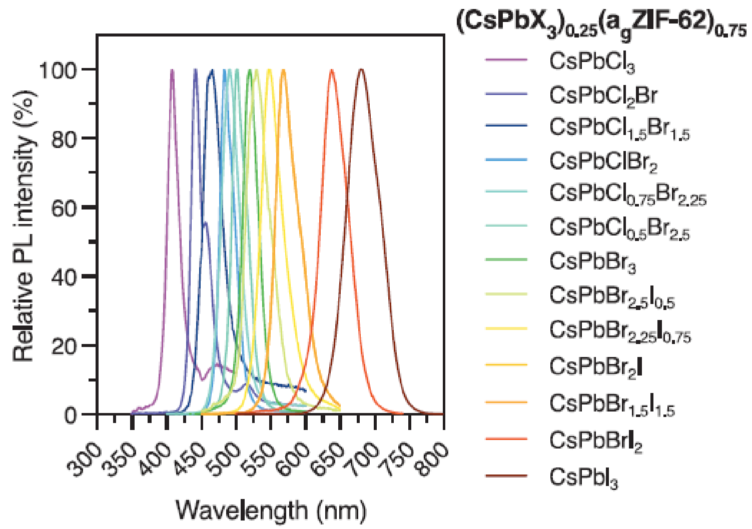
Characteristic emission for **γ-$CsPbI_3$**

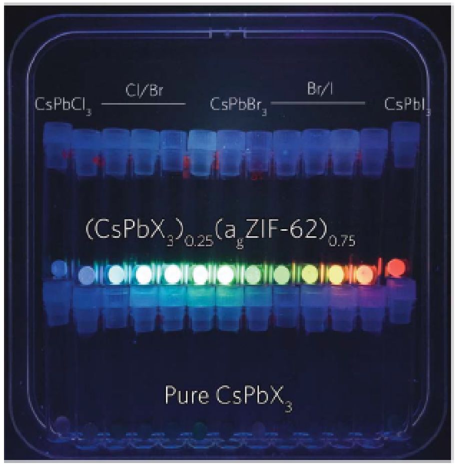
STEM@LPS

J. Hou et al. *Science*, **2021**, *374*, 621-625.

Durable narrow-band emission





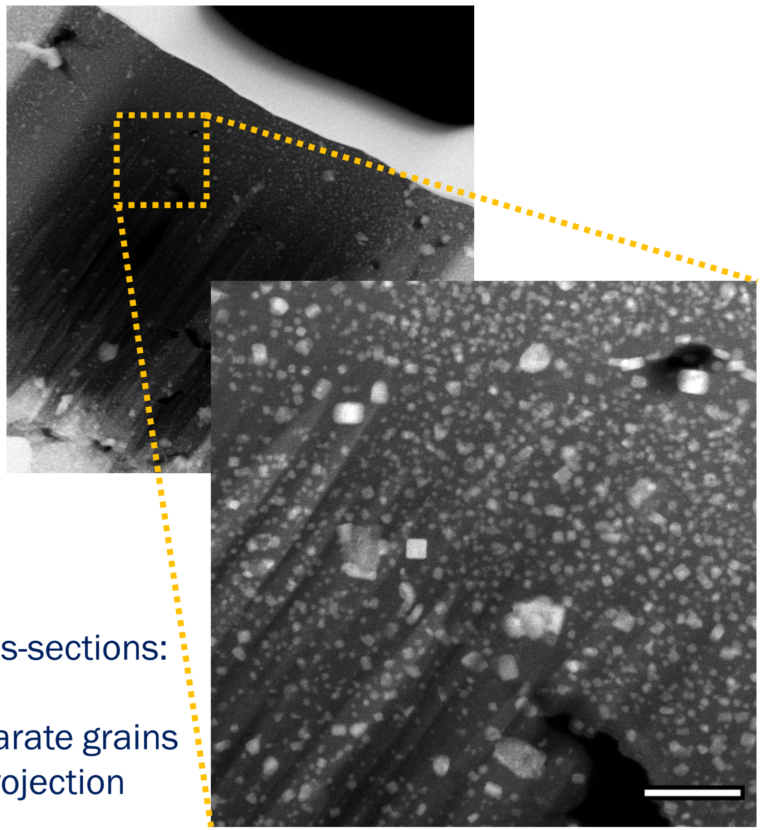


J. Hou et al. *Science*, **2021**, *374*, 621-625.

Correlative CL-SED, CL-EDS

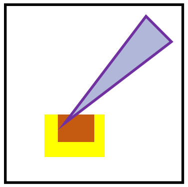
Cross-sections:

Separate grains in projection



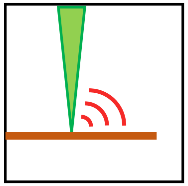
Scale bar: 250 nm

Sample fabrication FIB milling



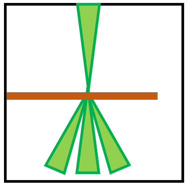
The University of Queensland Australia

CL spectrum image data



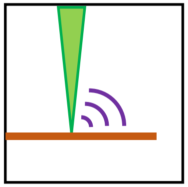
université PARIS-SACLAY

SED from identical regions



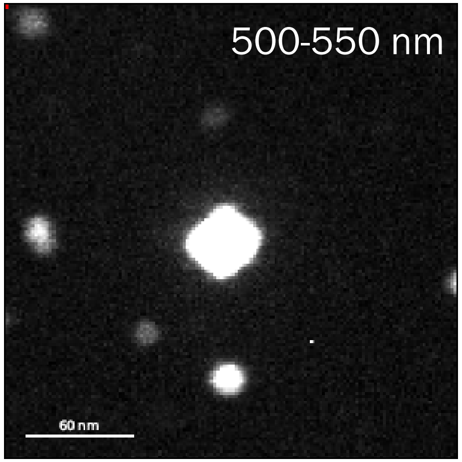
diamond

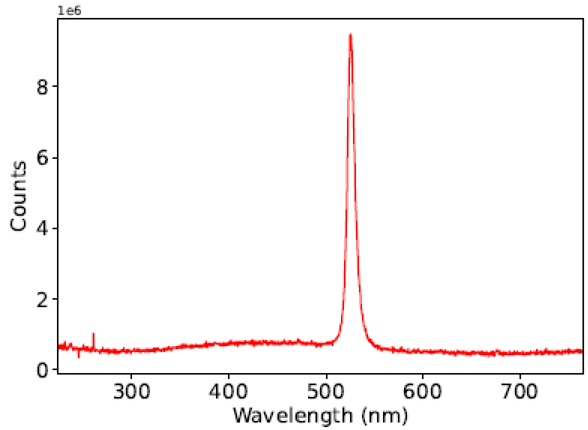
EDS from identical regions



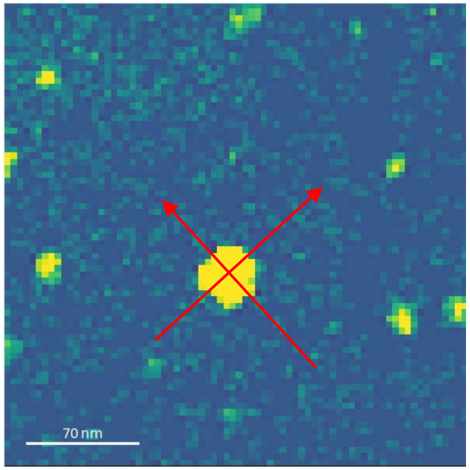
UNIVERSITY OF LEEDS

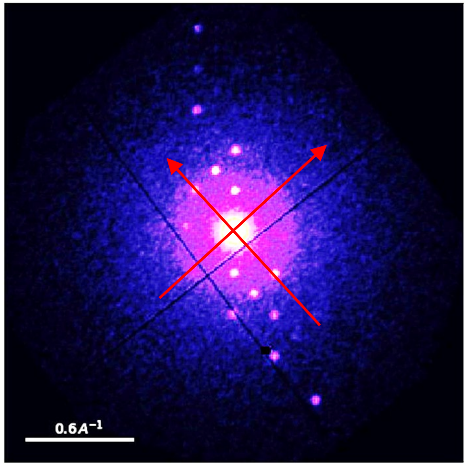
Correlative CL-diffraction





Dr Sang Pham





Bright, single peak for $CsPbBr_3$

SED:
phase identification and orientation

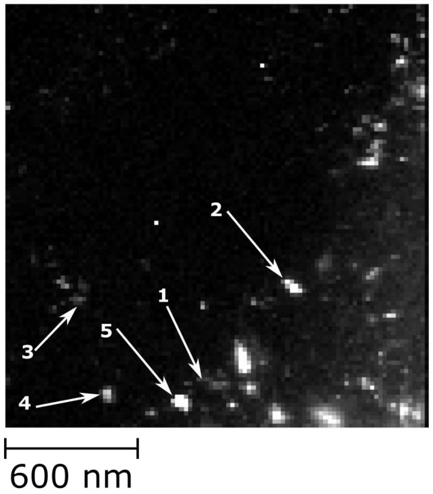
STEM@LPS

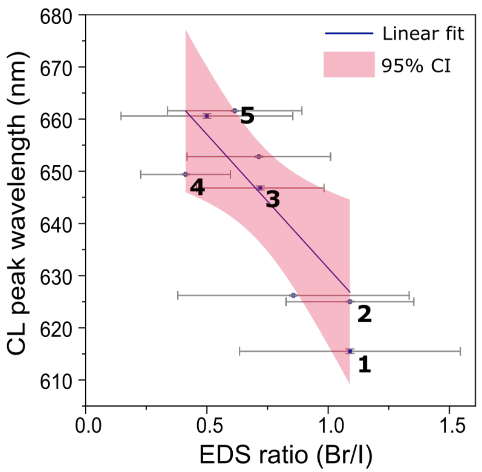
diamond

$(CsPbBr_3)(a_gZIF\text{-}62)$

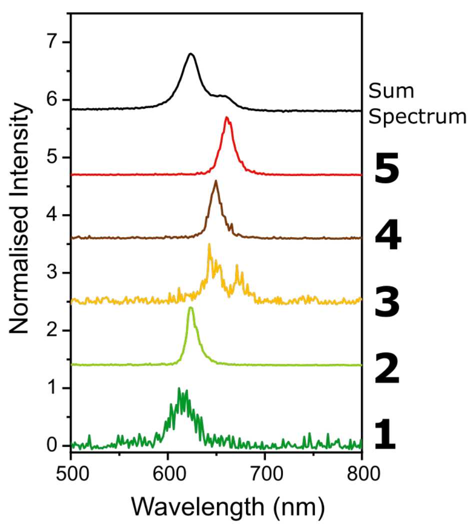
Correlative CL-EDS

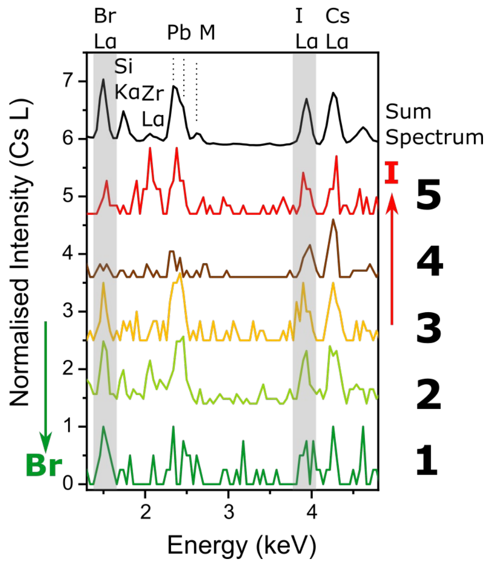
CL





$CsPbBr_xI_{3-x}$





EDS

STEM@LPS

LEMAS

Acknowledgements

Materials

Tom Bennett (*U. Cambridge)*

Xuemei Li, Jingwei Hou (*U. Queensland, Australia)*

Electron microscopy and data processing

Rana Doasa, Mark S'Ari, Sang Pham (*U. Leeds)*

Duncan Johnstone *(U. Cambridge)*

Luiz Tizei *(LPS, U. Paris-Saclay, CNRS, France)*

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diamond

cnrs

UKRI Engineering and Physical Sciences Research Council

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université PARIS-SACLAY

UNIVERSITY OF LEEDS

Bragg Centre for Materials Research
School of Chemical and Process Engineering & School of Chemistry

UNIVERSITY OF CAMBRIDGE

THE UNIVERSITY OF QUEENSLAND AUSTRALIA

esteem3

TNA user feedback

Dr Sophia Houari
Paris, France

Tooth enamel pathologies of environmental origin from macro- to nano-scale

Presentation available only during the online event

Assistance Publique Hôpitaux de Paris · Université Paris Cité · Inserm

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

COFFEE BREAK

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

TNA user feedback

Electron microscopy characterization of graphite-microparticle interface in spheroidal graphite iron alloys

Presentation available only during the online event

PhD., Leander Michels
Elkem Silicon Products
ISGI project - collaboration with UCAM

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

ROUTES OF SUSTAINABILITY: EDREAM AND ARIE

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

Routes of sustainability

- First steps towards sustainability: Creation of a "European Distributed Research infrastructure for Advanced electron Microscopy" (*e^-* DREAM) as a non-profit initiative (https://e-dream-eu.org/).

e^- DREAM

EUROPEAN DISTRIBUTED RESEARCH
INFRASTRUCTURE FOR ADVANCED
ELECTRON MICROSCOPY

Routes of sustainability

- e-DREAM is a member of the Analytical Research Infrastructures in Europe (https://arie-eu.org/).

Support for the European EM community:

- Long-term strategic initiatives
- Funding for access and joint research
- Contact point to policy makers and politicians
- Contact point to EM organisations on other continents
- Community-driven actions to support researchers
- Synergy with other communities to take advantage of complementary expertise
- Sustainable situation beyond current Grand Challenges

e⁻ DREAM: Members

Forschungszentrum Jülich	Rafal Dunin- Borkowski
CEMES-CNRS	Etienne Snoeck
LPS-CNRS	Mathieu Kociak
NTNU (Trondheim)	Randi Holmestad
Universiteit Antwerpen	Johan Verbeeck
University of Oxford	Angus Kirkland
CNR-IOM	Regina Ciancio
Graz Univ. of Technology	Gerald Kothleitner
ICN2 (Barcelona)	Jordi Arbiol

Open to further members soon

e^- DREAM: Working groups

1. European strategy

- This working group will address short-term and long-term perspectives and sustainability of electron microscopy in the European landscape of Research Infrastructures.
- It will define schemes to maximize the impact of electron microscopy.
- It will develop a user service evolution strategy at international, national and local levels.

e^- DREAM: Working groups

2. Data policy

- This working group will address data management, storage, access and file formats, with a focus on open science.
- It will compile information about existing solutions, work on data policy and approaches for best practice and contribute to developments for the benefit of the community.

e^- DREAM: Working groups

3. Software

- This working group will address software for data acquisition, analysis, simulation, instrument control and remote access.
- It will compile information about existing solutions and contribute to developments for the benefit of the community.

e^- DREAM: Working groups

4. Hardware

- This working group will address hardware standardisation of instruments to improve interoperability of multi-vendor and self-made equipment to enable an open ecosystem that fosters innovation.
- It will compile information about existing and emerging technologies and collaborate with related fields to decrease the gap between what is technically possible and what is available for electron microscopy.

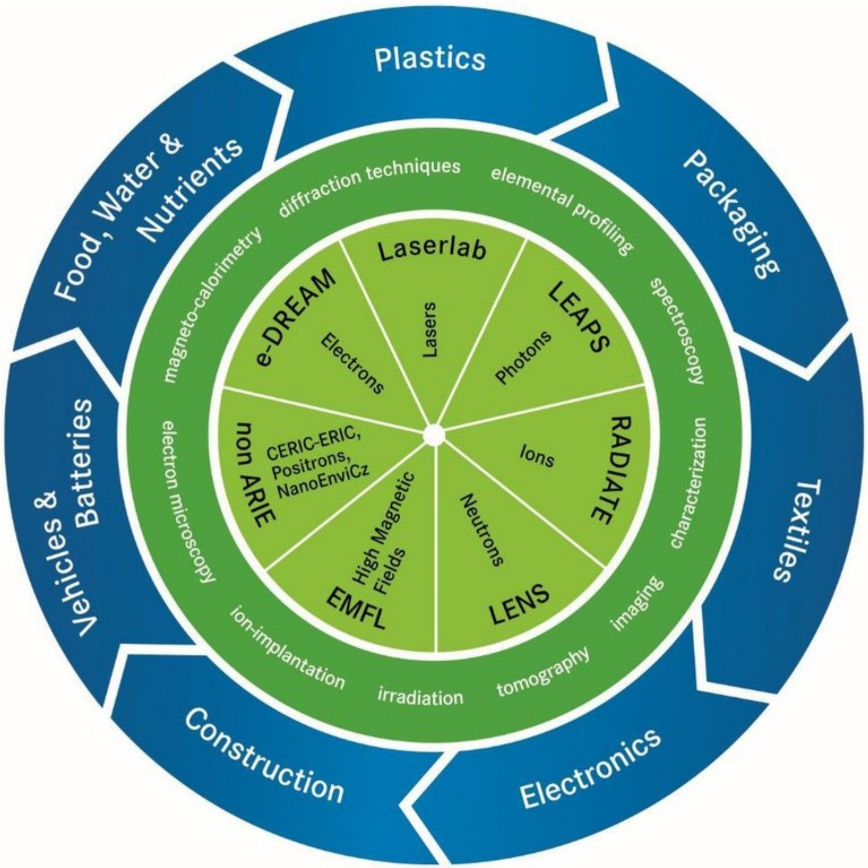
e^- DREAM: Current actions

Horizon Europe proposals together with ARIE:

- European Green Deal
- Circular economy
- Cancer
- Remote access
- Digital twins
- Transition towards a SusTainable Environment
- Nanoscience & Nanotechnology

Horizon Europe projects

- HORIZON-INFRA-2021-SERV-01-04 “Recyclable materials development at analytical research infrastructures” (**ReMade@ARI**) co-ordinated by HZDR (2023-2027).
- Provide scientists with analytical tools to study recyclable materials.



Horizon Europe projects

- HORIZON-INFRA-2022-TECH-01-01 "Interoperable electron Microscopy Platform for Advanced RESearch and Services" (**IMPRESS**) co-ordinated by CNR (2023-2027).
- Instrumentation development for transfer of experiments between characterization tools at TRL 8 based on open standards through pre-commercial procurement.

e⁻ DREAM: Current actions

Formation of legal entity (AISBL)

- First draft of statues prepared
- Law firm commissioned

Discussions with policy makers and ministers about support for the formation of a sustainable distributed research infrastructure:

- **ESFRI**
- **ERIC**

CONCLUSION

THIS PROJECT HAS RECEIVED FUNDING FROM THE EUROPEAN UNION'S HORIZON 2020 RESEARCH AND INNOVATION PROGRAMME UNDER GRANT AGREEMENT NO **823717**

Enabling **S**cience and **T**echnology through **E**uropean **E**lectron **M**icroscopy

Thank you for your attention!

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