



| The European Synchrotron

STRUCTURE OF MATERIALS GROUP



- ID03 Hard X-ray Microscope
- ID11 Materials science
- ID15A High-energy beamline for materials chemistry and engineering
- BM18 High throughput large field phase-contrast
- ID19 Microtomography beamline
- ID22 Powder diffraction
- ID31 High-energy beamline for buried interface structures and materials processing
- BM05 Instrumentation facility and proprietary tomo- and topography research

GREEN TRANSITION

- Clean energy, clean industry, security and sustainability of raw materials, etc.
- Research and innovation driven

- increasing complexity
- tackling multifaceted problems



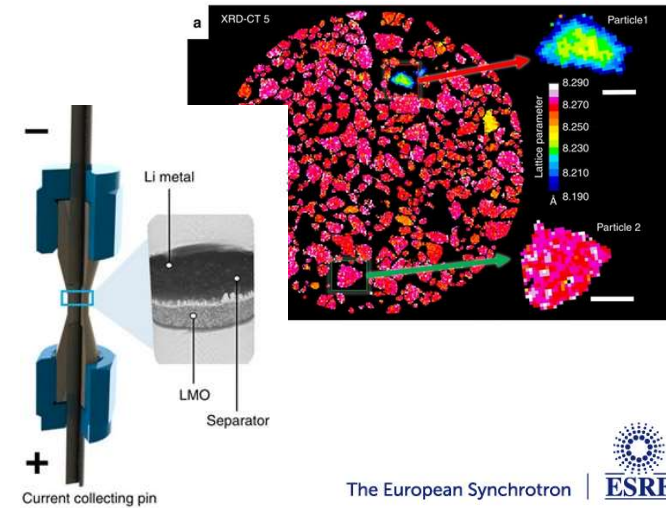
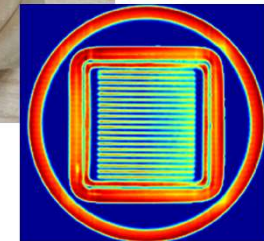
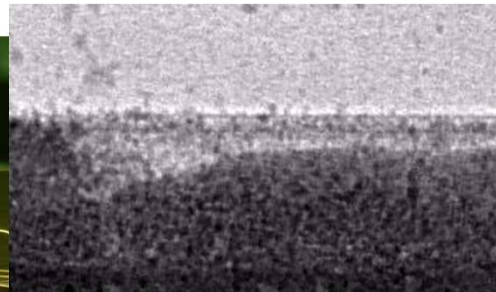
- requires the use of several techniques
- proceed from science to technology

- interdisciplinary groups
- centralized facilities



FILLING THE GAP

The GAP between Academia/Research and Industry



ADDITIVE MANUFACTURING

AM is

- versatile, flexible
- highly customizable
- enables rapid low-volume production highly complex components

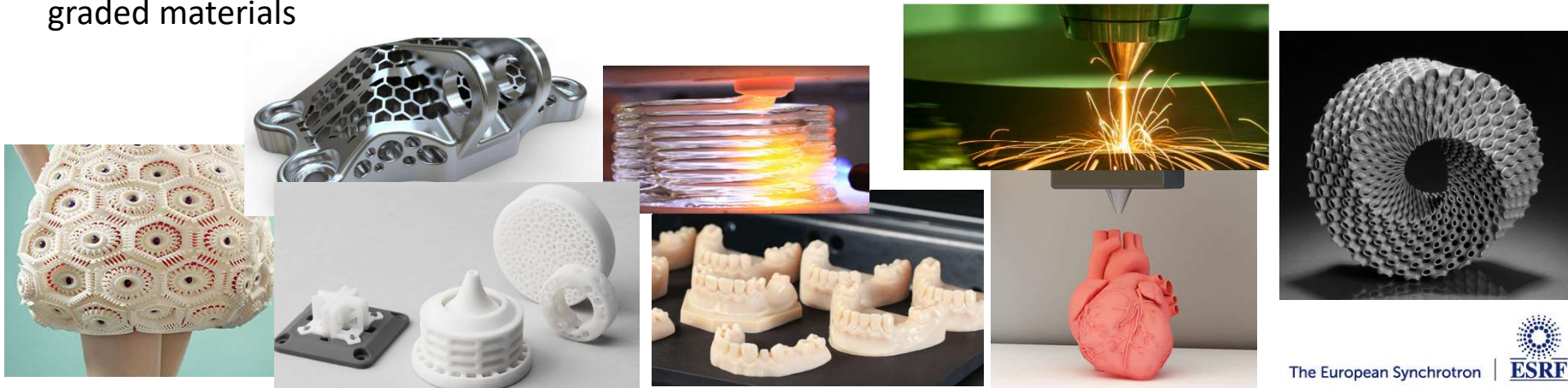
Widely varying materials

- metals, ceramics, glasses
- polymeric materials, tissues
- along with combinations in the form of composites, hybrid, or functionally graded materials

More art than science, the challenges remain

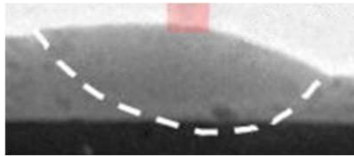
- great uncertainty in terms of the processes and controls
- obtaining objects that are functional
- 'materials' and 'metrology' to achieve this functionality in a predictive and reproductive ways
- control of material properties, geometry, and residual stresses

Tremendous discrepancy in materials and processes, with each having their own strengths and weaknesses

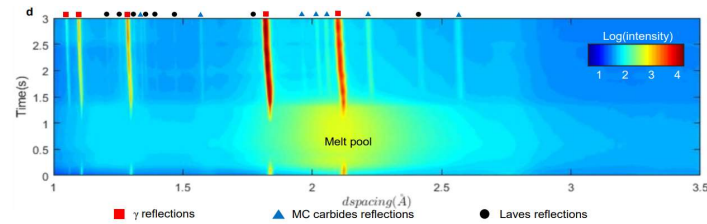


LASER ADDITIVE MANUFACTURING (LAM)

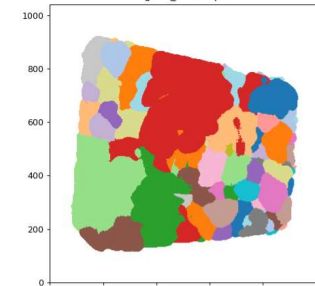
Correlative Synchrotron X-ray techniques



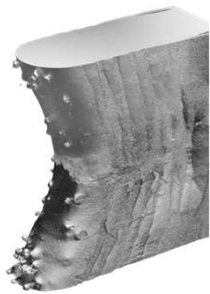
ID19: *In-situ* X-ray imaging of the laser-matter interaction



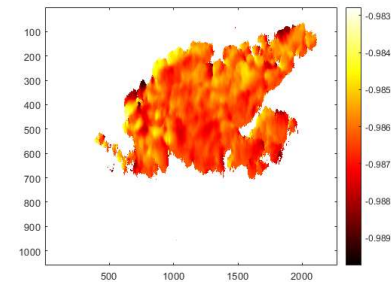
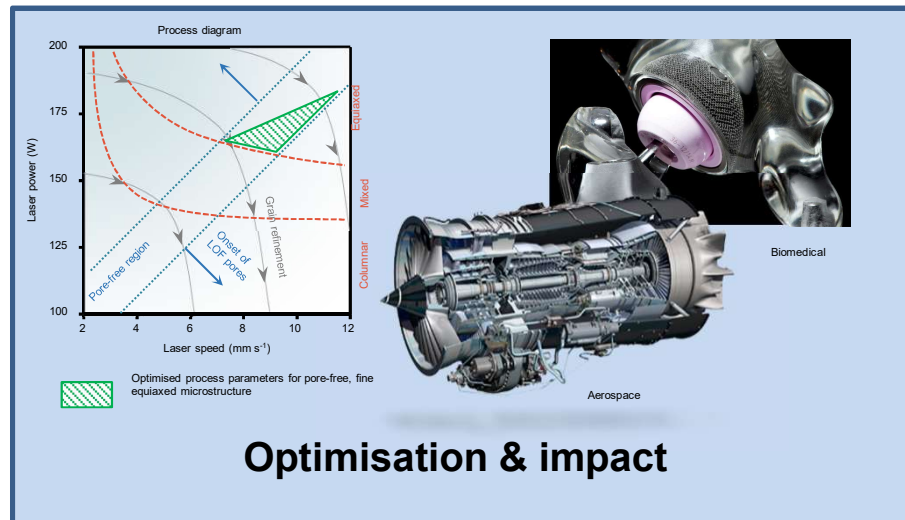
ID31: *In-situ* X-ray diffraction of the phase evolution



ID11: DCT and 3D-XRD LAM grain mapping



BM05, BM18 & ID19: Tomography of a LAM structure



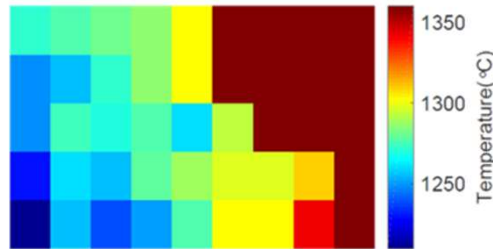
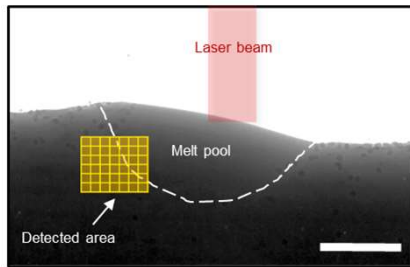
ID03: Dark Field microscopy reveal high angular resolution information of the LAM grain

Courtesy Y. Chen (RMIT)

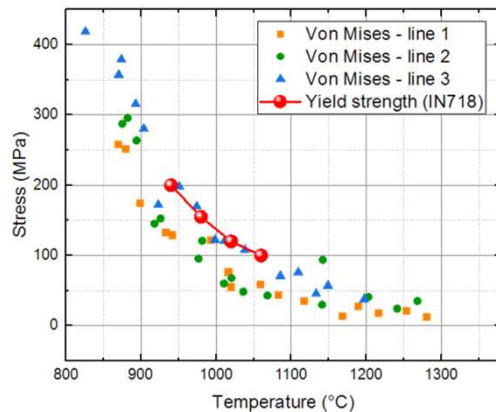
More modalities to come...

LASER ADDITIVE MANUFACTURING (LAM)

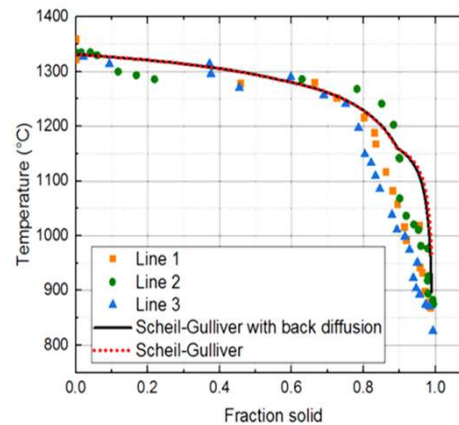
In situ X-ray diffraction of direct energy deposition LAM of IN718



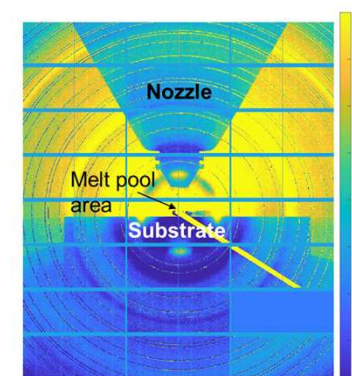
DED-LAM replicator at ID31



Solidification range of IN718



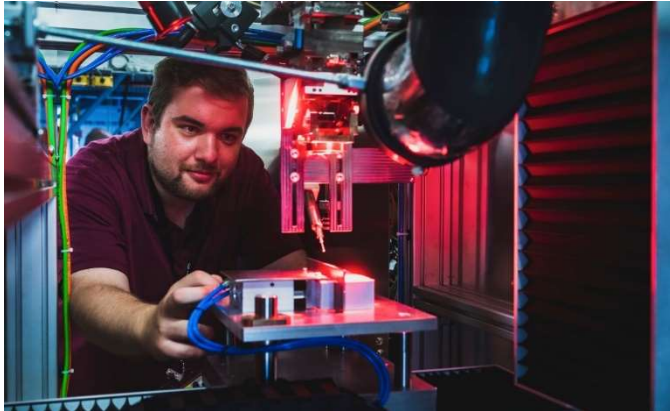
High temperature micromechanical behaviour of IN718



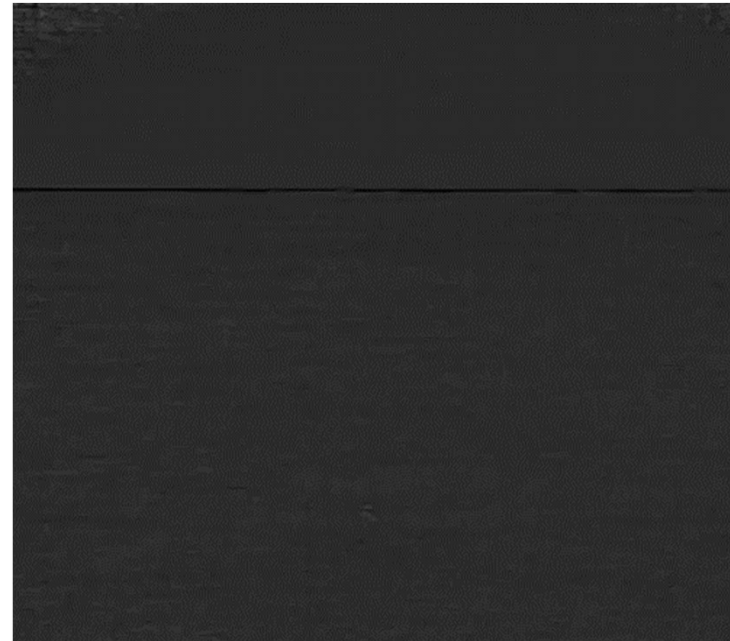
DED-LAM replicator shadows on the detector

Courtesy Y. Chen (RMIT)

LASER ADDITIVE MANUFACTURING (LAM)



studying the melt pool dynamics and the keyhole formation during laser speeds ≥ 8 m/min

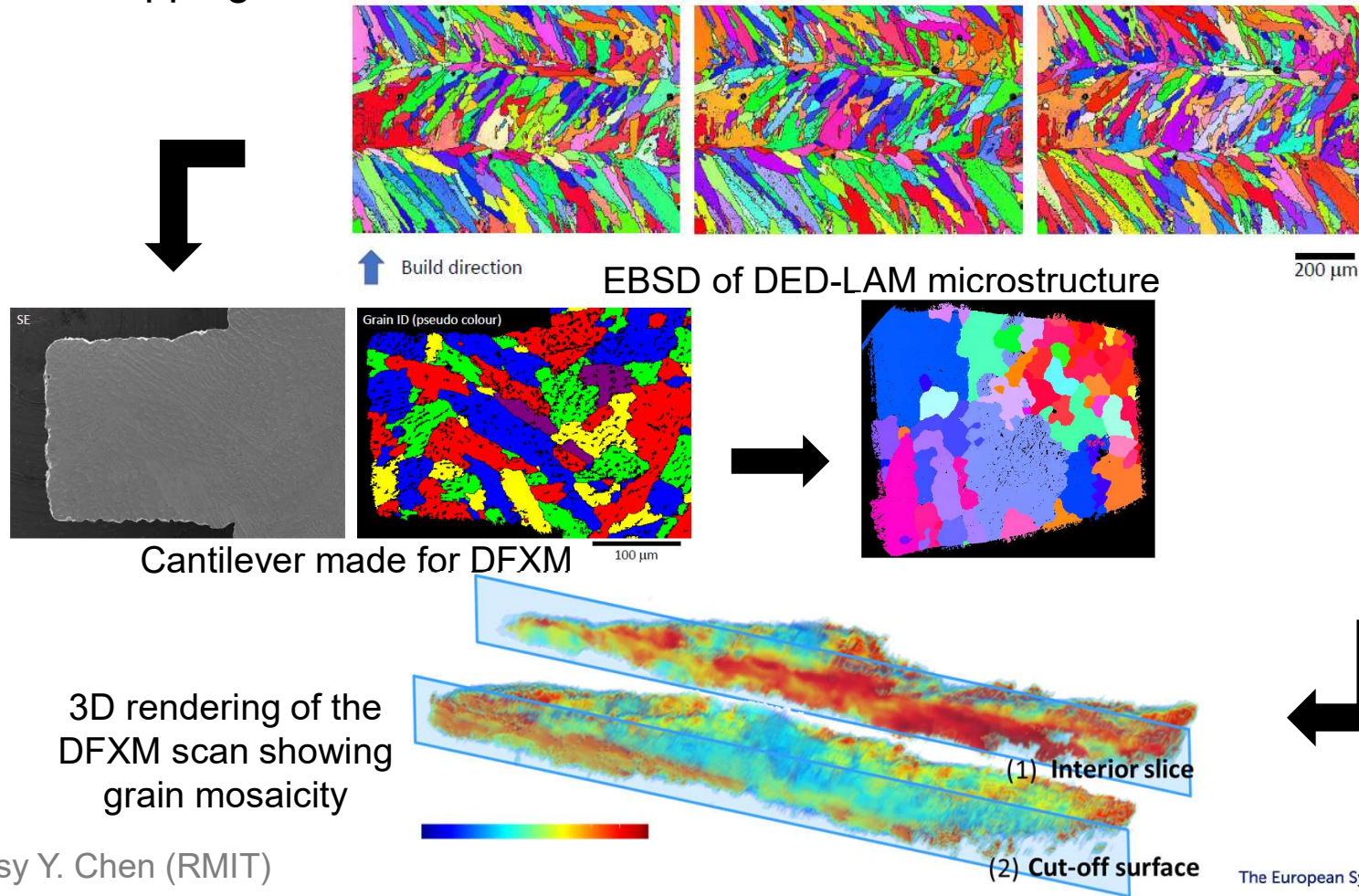


Beamline ID19 (SoM)
Technical University of Ilmenau (Germany)

Courtesy Y. Chen (RMIT)

LASER ADDITIVE MANUFACTURING (LAM)

Grain mapping of LAM microstructure



Courtesy Y. Chen (RMIT)

DARK FIELD X-RAY MICROSCOPY (DFXM)

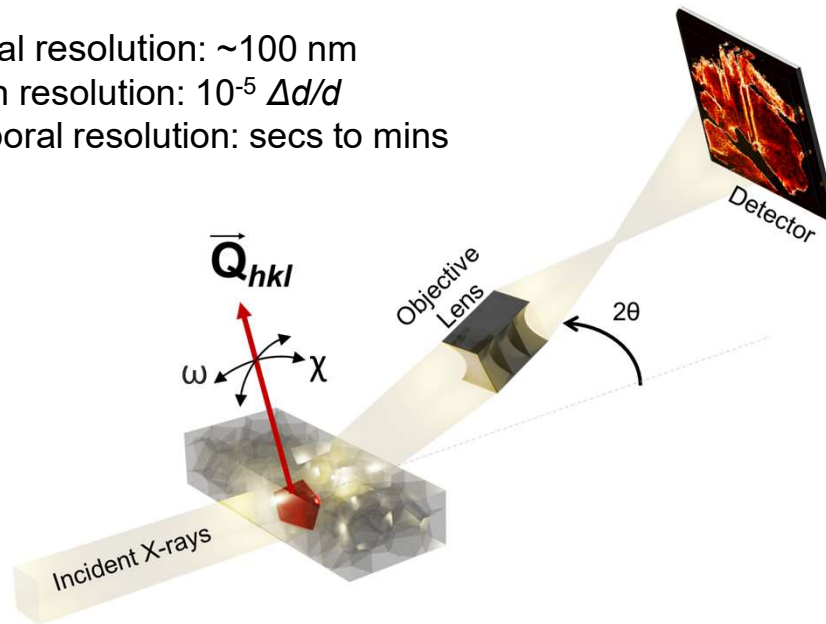
New flagship beamline EBSL02-ID03

A unique microscope to image embedded structures

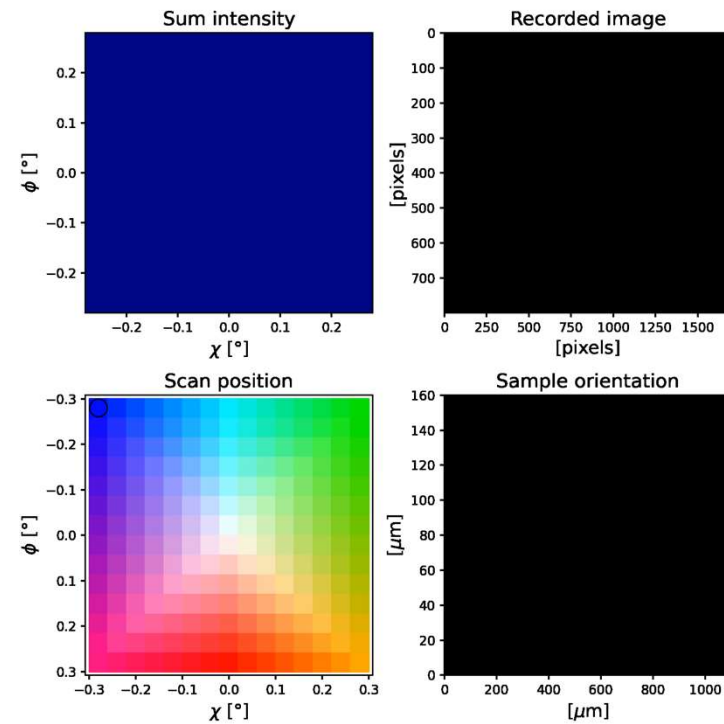
Spatial resolution: ~ 100 nm

Strain resolution: $10^{-5} \Delta d/d$

Temporal resolution: secs to mins



H. Simons *et al*, *Nat. Comm.* 2015



Courtesy C. Yildirim (ESRF)

DARK FIELD X-RAY MICROSCOPY (DFXM)

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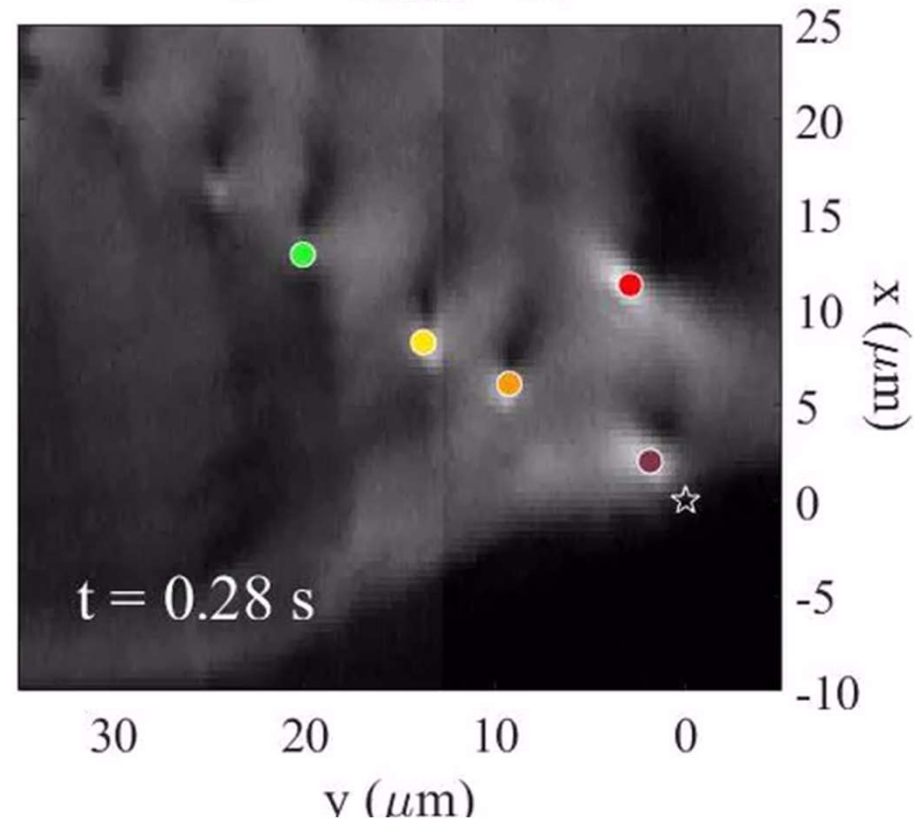
Strain resolution: $10^{-5} \Delta d/d$

Temporal resolution: secs to mins

motion of dislocation
boundaries in Al

Dresselhaus Marais *et al*, *Sci Rep.* 2023

$T = 638$ °C

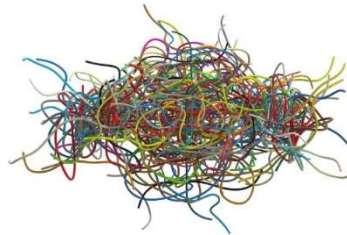


Courtesy C. Yildirim (ESRF)

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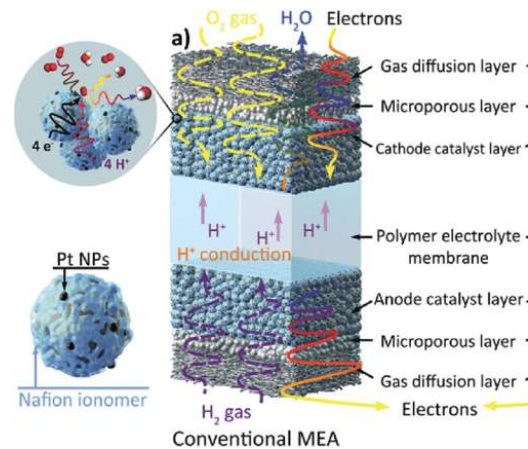
- requires the use of several techniques
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HYDROGEN AND CO₂ CONVERSION

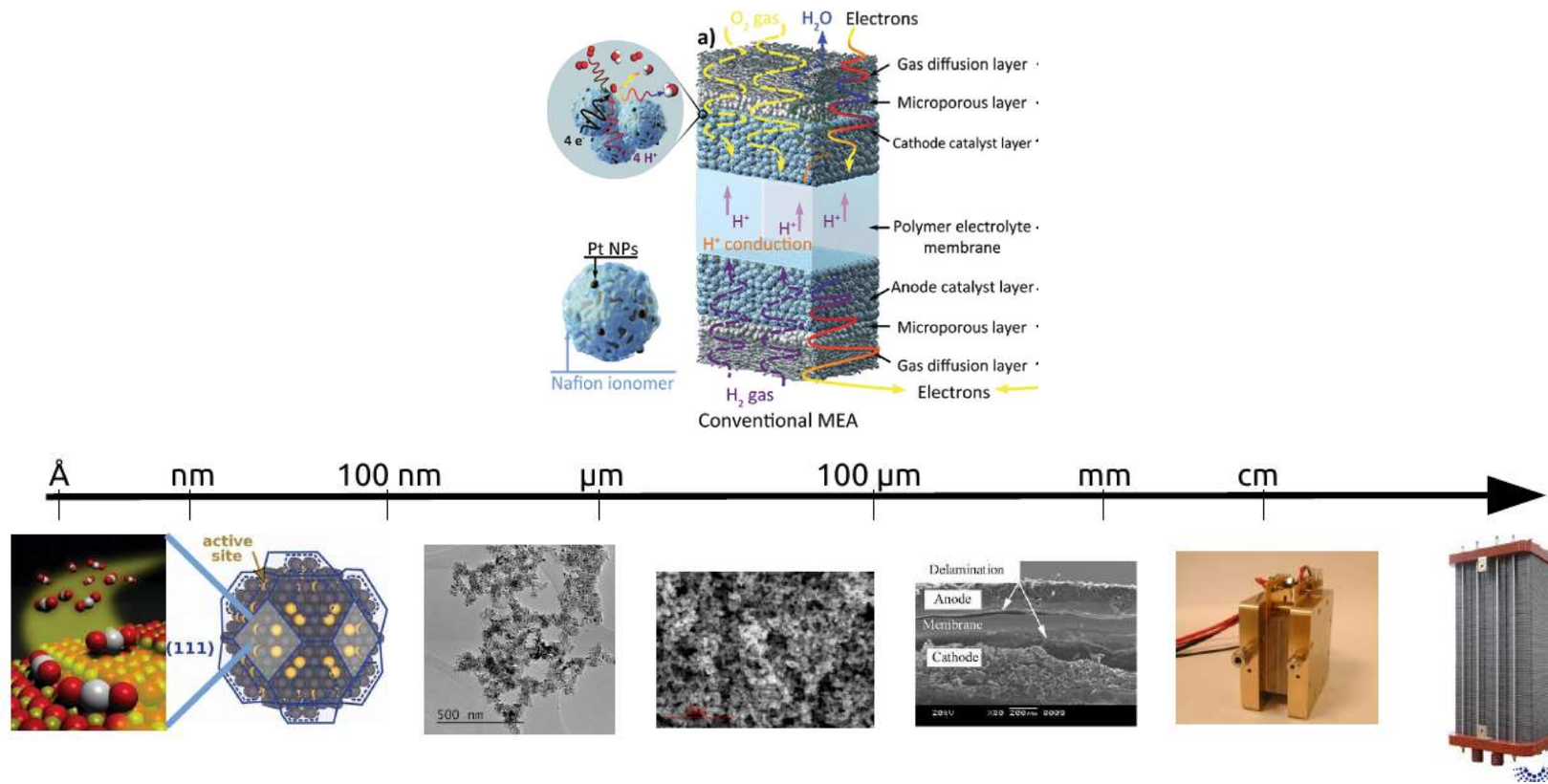
- Electrolyzers and fuel cells are **complicated heterogeneous electrochemical systems** with critical phenomena happening at many different scales



- **Materials and engineering issues** are at the origin of performance limitations
- Materials perform characteristically within their **ideal sample environment**
- The correlative characterization (*in-situ* and *operando*) during development of the cell has the highest importance as it is the only route to solve the material problems

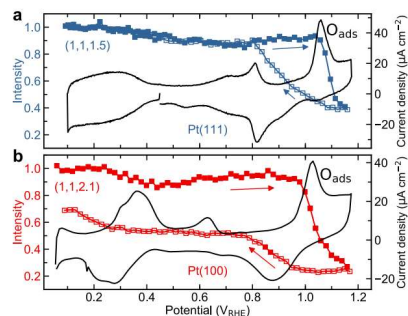
HYDROGEN AND CO₂ CONVERSION

- Electrolyzers and fuel cells are **complicated heterogeneous electrochemical systems** with critical phenomena happening at many different scales



HYDROGEN AND CO₂ CONVERSION

Model systems



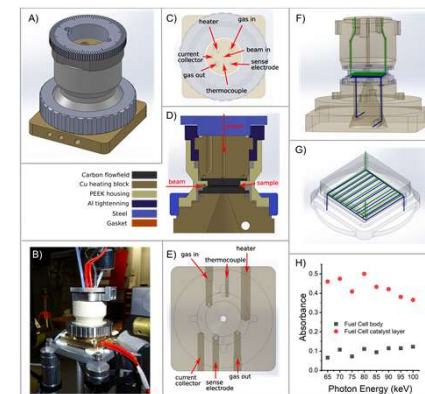
Wiegmann *et al.*, *ACS Catal.*, 2022
 Fuchs *et al.*, *Nat. Catal.*, 2020
 Dmec *et al.*, *Echim. Acta.*, 2017
 Ruge *et al.*, *JACS*, 2017
 ...

Applied systems

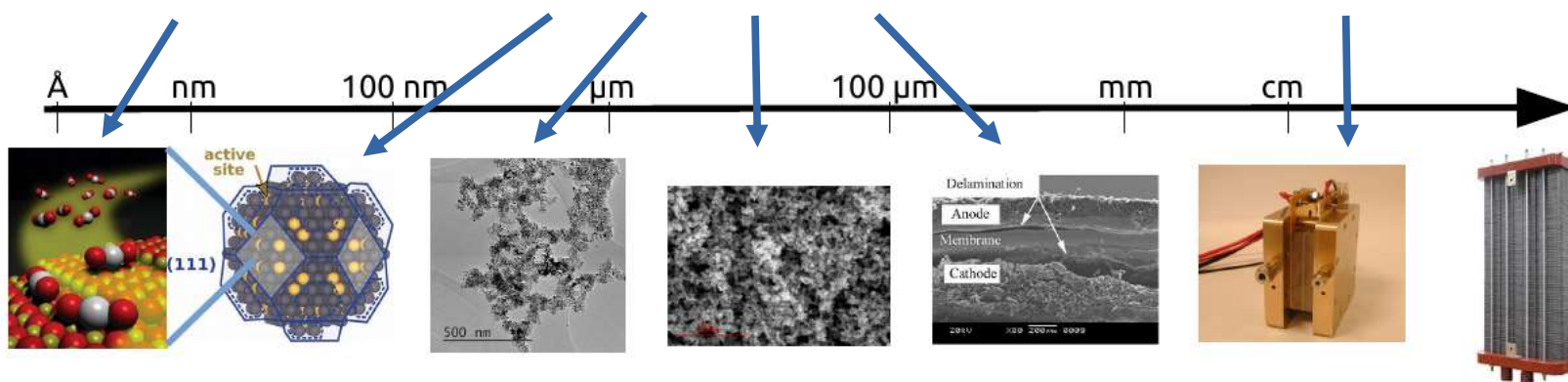


Chattot *et al.*, *JACS*, 2021
 Chattot *et al.*, *ACS Energy Lett.*, 2020
 Chattot *et al.*, *ACS Catal.*, 2020
 Martens *et al.*, *ACS Appl. Energy Matter.* 2019
 Chattot *et al.*, *Nat. Matter.*, 2018
 ...

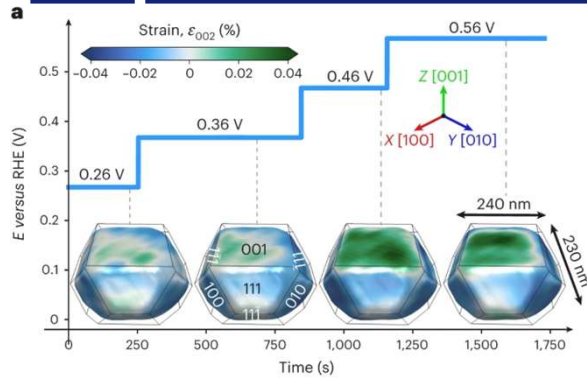
Devices



Martens *et al.*, *J. Power Sources*, 2022
 Martens *et al.*, *ACS Energy Lett.*, 2021
 Martens *et al.*, *J. Power Sources*, 2020



POWER TO X (CONVERSION OF HYDROGEN AND CO₂)



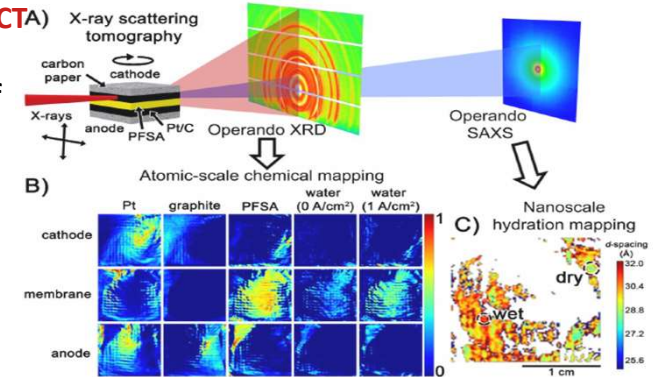
Atlan, J. *et al*, Nature Materials (2023) - ID01

Bragg Coherent Diffraction Imaging

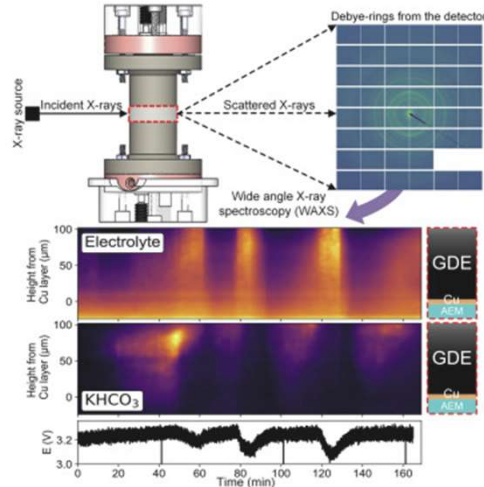
Strain evolution of Pt catalyst under applied potential

Operando XRD/SAXS-CTA

Correlative imaging of high performance hydrogen fuel cell showing water distribution in the cell and its relation to the ageing phenomena



Martens *et al*, ACS Energy Letters (2021) - ID31



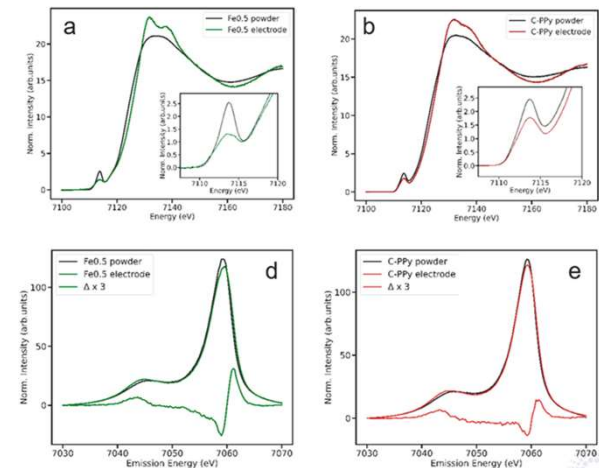
Moss *et al.*, Joule (2023) - ID31

Operando XRD

Emergence of complex oscillatory behavior in CO₂ electrolyzer

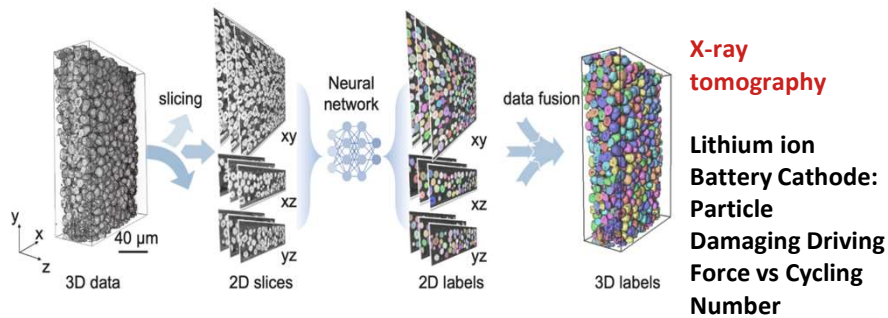
XAS on non-PGM catalysts

Evolution of electronic state of the active catalyst during ink catalyst layer preparation



Savaleva *et al.*, ACS Appl. Energy Matter (2023) - ID26

BATTERIES



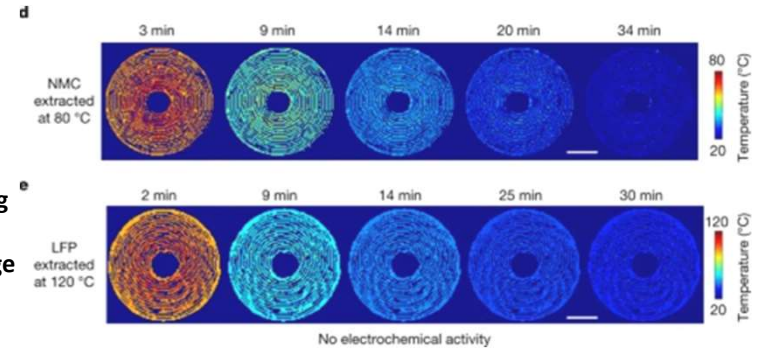
J. Li *et al.*, Science (2022) - ID16A

X-ray tomography

Lithium ion Battery Cathode: Particle Damaging Driving Force vs Cycling Number

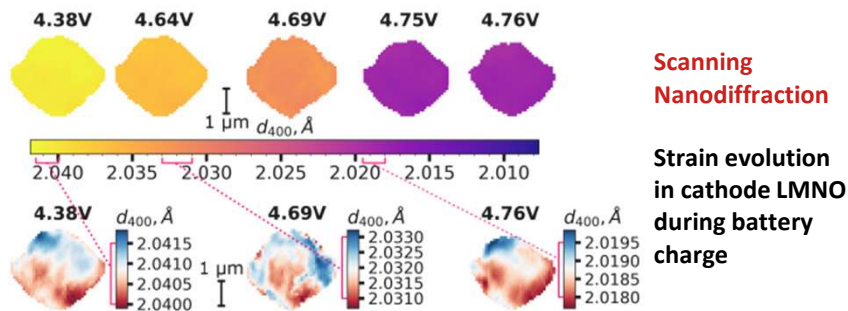
XRD-CT

Battery temperature evolution during fast charge/discharge



Heenan *et al.*, Nature (2023) - ID15A

- ESRF capabilities span from fundamental research of battery materials to characterization of commercial cells
- Multiscale and multimodal
- Atomic scale structure → nanoscale and mesoscale morphology → coin cells → cylindrical commercial cells



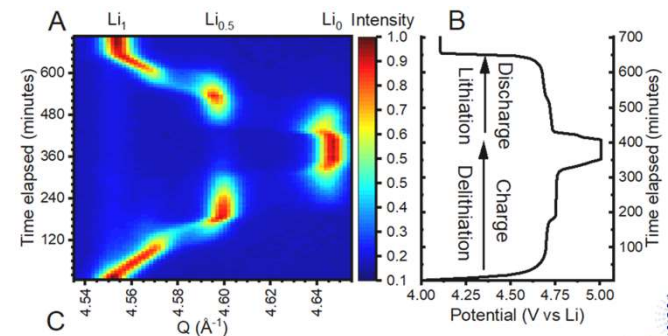
Martens *et al.*, 10.26434/chemrxiv-2022-lkzbj(2023) - ID01

Scanning Nanodiffraction

Strain evolution in cathode LMNO during battery charge

Operando XRD

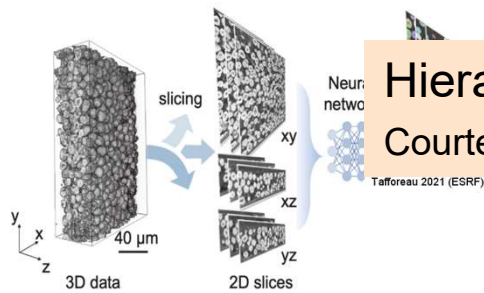
Phase transitions in cathode LMNO cathode material during charge and discharge



Martens *et al.*, ACS Materials Letter (2022) - ID31

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BATTERIES



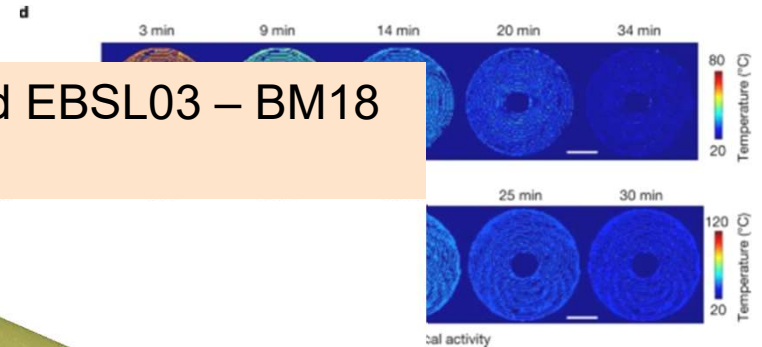
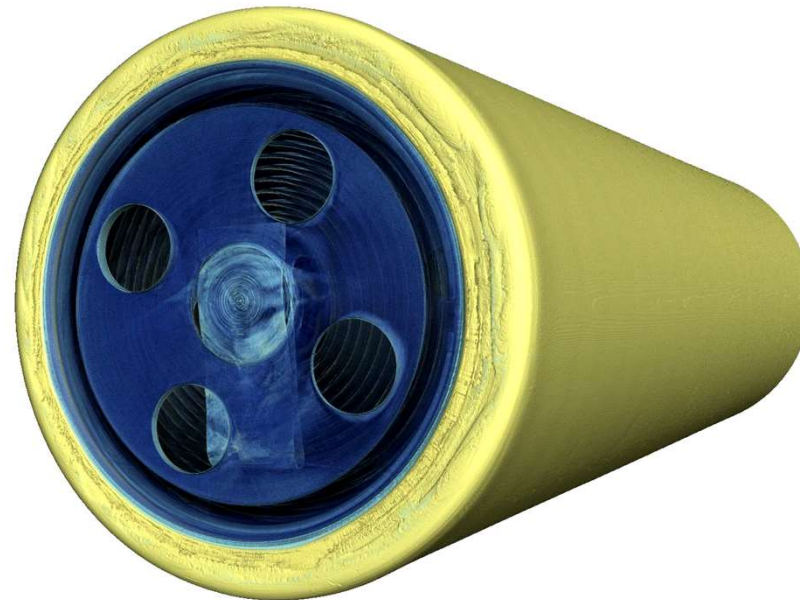
Hierarchical imaging at BM05 and EBSL03 – BM18

Courtesy P. Tafforeau

Tafforeau 2021 (ESRF)

J. Li *et al.*, Science (2022) - ID15A

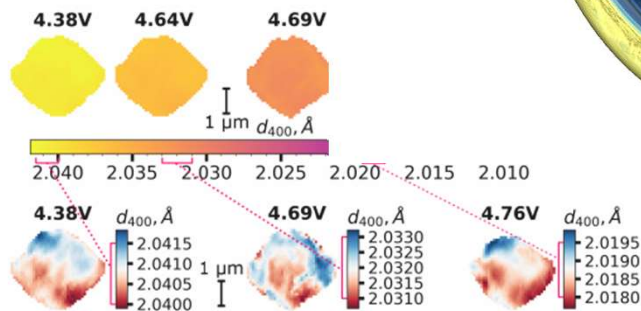
- ESRF capabilities for battery cells
- Multiscale and multi-modal imaging of battery cells
- Atomic scale structural analysis of battery cells



Thermal activity (2023) - ID15A

Evolution of commercial

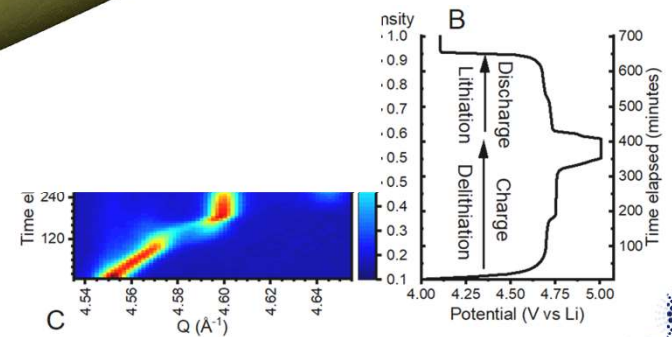
of commercial



Martens *et al.*, 10.26434/chemrxiv-2022-lkzbj(2023) - ID01

Structural evolution in cathode LMNO during battery charge

Structural evolution of cathode material during charge and discharge



Martens *et al.*, ACS Materials Letter (2022) - ID31

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THANK YOU FOR YOUR ATTENTION
CREATING TOGETHER VALUE FOR ALL
SCIENCE FOR A SUSTAINABLE SOCIETY

PIONEERING SYNCHROTRON SCIENCE



STREAMLINE

Streamline project has received funding from the European Union's Horizon 2020 research and innovation programme under the INFRADEV grant agreement No 870313



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