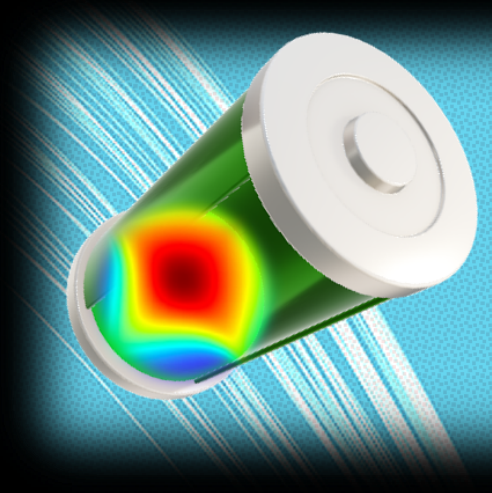

International Operando Battery Days

Grenoble, France, **12-14 June** 2024

Day One

12.06.2024

Welcome

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ESRF

ESRF

ESRF

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MAKALU

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Local team : V. Vanpeene, Y. Yang, G. Navallon, F. Cadiou, P. Perrenot, O. Korjus, J. Charbonnel, G. Paradol, N. Vostrov, Z. Zhang

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International Operando Battery Days
Grenoble, France, 12-14 June 2024
Program
Everything happens in the WTC
Best talk and best poster prizes for young scientists
Time | Wednesday, June 12th
8:00 | Registration
9:00 | Welcome speech
9:10 | H.G. Steinrück
9:40 | A. Black
10:00 | L. Guin
10:20 | Coffee Break
10:50 | A. Poletayev
11:10 | L. Shoji Aota
11:30 | F. Sayed
11:50 | K. Frenzel
12:10 | Lunch
14:00 | E. Ilott
14:30 | G. Jacquet
14:50 | R. Müller
15:10 | M. Jauregui
15:30 | Coffee break
16:00 | A. Demortière
16:30 | M. Wagemaker
16:50 | R. Raccichini, J. Drnec, V. Vanpeene, Partha Paul, P. A. Wieser, T. PAPAZOGLOU
18:15 – 21:00 | Poster session
Time | Thursday, June 13th
8:30 | E. Berg
9:00 | L. Simonelli, M. Johansen, J. Pfaff, A. Skurtvelt, J. Vijayakumar, S. Belin
10:20 | Coffee Break
10:50 | P. Perrenot
11:10 | M. Löw
11:30 | B. Hu
11:50 | O. Korjus
12:10 | Lunch
14:00 | D. Ravnsbæk
14:30 | S. Risse
14:50 | C. Renais
15:10 | J. Rizell
15:30 | Coffee Break
16:00 | M. Pasta
16:30 | E. Lübke
16:50 | I. Yousef, C. Lopez, E. Woillez, T. Nguyen
19:30 | Gala Dinner
Time | Friday, June 14th
8:30 | S. Franger
9:00 | B. Beckhoff
9:20 | G. S. Philips
9:40 | G. Oney
10:00 | M. R. Amara
10:20 | Coffee Break
10:50 | O. Fontaine
11:20 | A. Klein
11:40 | E. Kataev
12:00 | A. Morata
12:20 | Prizes and Ending Words
12:40 | Lunch Box
14:00 | Visit of ESRF and ILL

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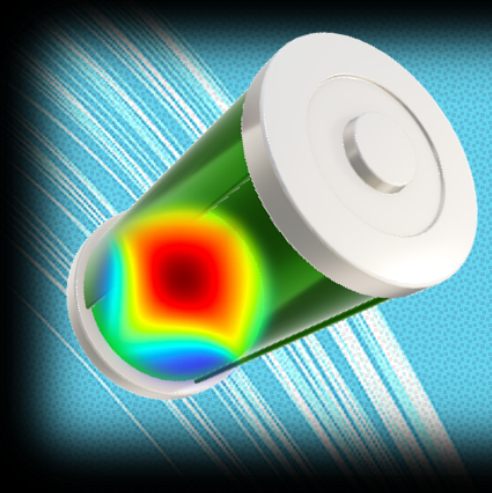

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12.06.2024

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UNDERSTANDING ENERGY STORAGE MATERIALS ACROSS SCALES BY BATHING MODEL SYSTEMS IN (COHERENT) X-RAYS
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RWTH AACHEN UNIVERSITY, INSTITUTE OF PHYSICAL CHEMISTRY
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Energy storage
How Lithium-ion Batteries Work
Complexity
Ionic resistance in bulk electrolyte
Ionic resistance in composite electrode
Charge transfer resistance
Diffusion in active materials
Carbon black
Surface film
Current collector
Electronic conduction
Sci Rep. 6:26382
Isolate individual components in model systems
Investigate atomic- to meso-scale in environments natural to functional devices → operando
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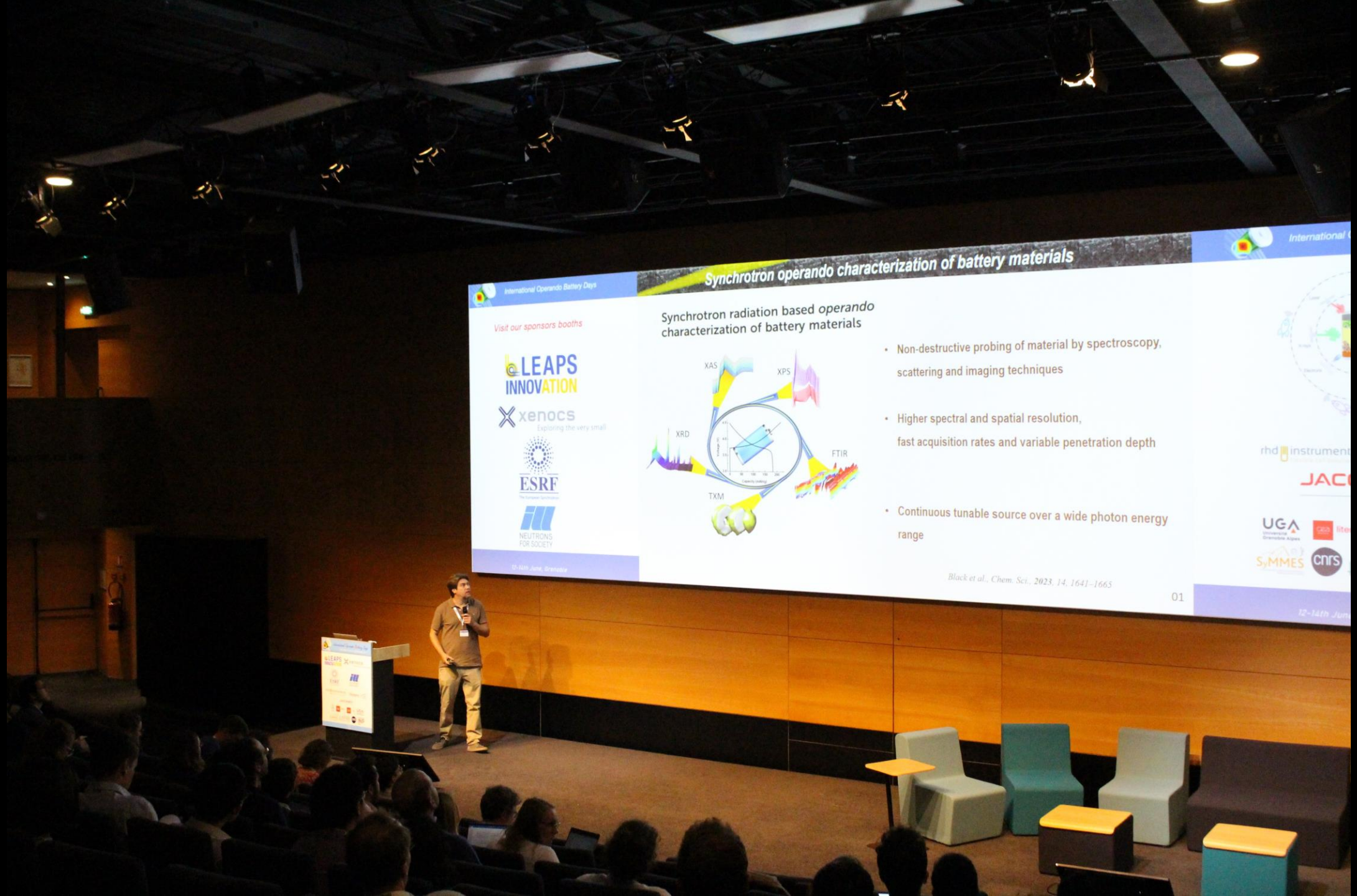
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Synchrotron operando characterization of battery materials
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Synchrotron radiation based operando characterization of battery materials
XAS
XPS
XRD
FTIR
TXM
Non-destructive probing of material by spectroscopy, scattering and imaging techniques
Higher spectral and spatial resolution, fast acquisition rates and variable penetration depth
Continuous tunable source over a wide photon energy range
Black et al., Chem. Sci., 2023, 14, 1641–1665
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XRD
FTIR
TXM
Voltage (V)
Capacity (mAh/g)
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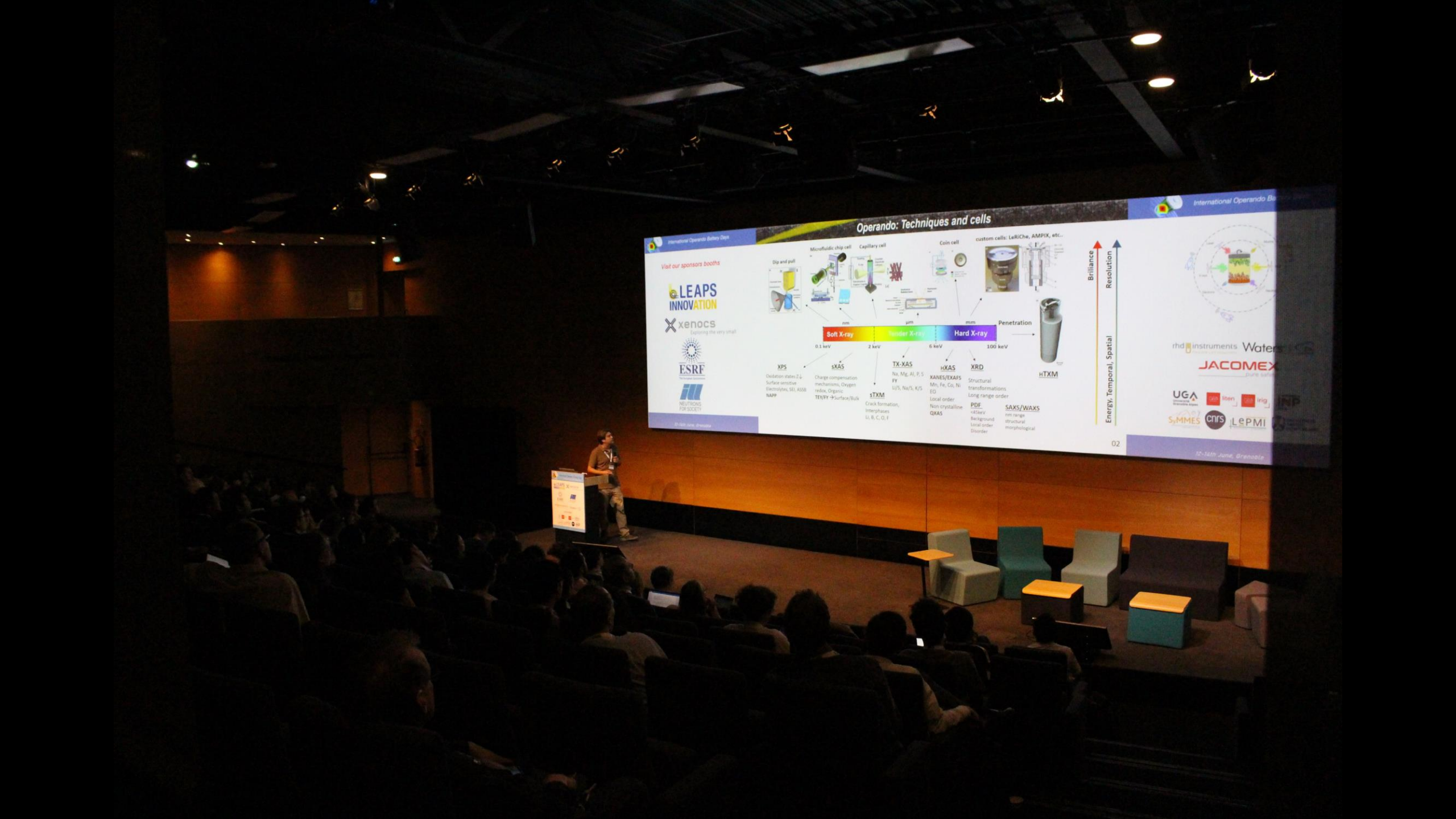
Operando: Techniques and cells
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Dip and pull
Microfluidic chip cell
Capillary cell
Coin cell
custom cells: LeRiChe, AMPIX, etc...
nm
µm
mm
Soft X-ray
Tender X-ray
Hard X-ray
0.1 keV
2 keV
6 keV
100 keV
Penetration
XPS
Oxidation states
Surface sensitive
Electrolytes, SEI, ASSB
NAPP
sXAS
Charge compensation mechanisms, Oxygen redox, Organic
TEY/FY → Surface/Bulk
sTXM
Crack formation, Interphases
Li, B, C, O, F
TX-XAS
Na, Mg, Al, P, S
FY
Li/S, Na/S, K/S
hXAS
XANES/EXAFS
Mn, Fe, Co, Ni
EO
Local order
Non crystalline
QXAS
XRD
Structural transformations
Long range order
PDF
>45keV
Background
Local order
Disorder
SAXS/WAXS
nm range
structural
morphological
hTXM
Brilliance
Resolution
Energy, Temporal, Spatial
02
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CSIC
Carlo Marini (Alba)
Laura Simonelli (Alba)
Jean-Noël Chotard (LRCS)

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ÉCOLE POLYTECHNIQUE
INSTITUT POLYTECHNIQUE DE PARIS
Effect of stress on the electrochemical response of silicon electrodes
Laurent Guin
Assistant Professor, Ecole polytechnique
With Anh Tuan Le, Xavier Bruant
and Michel Jabbour, François Ozanam, Michel Rosso
LMS
LABORATOIRE PMC
June 12th, 2024

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Silicon electrodes:
Swelling, chemistry-induced deformation due to up to 10x graph
50 nm
[Zhang et al., Prog. Mat. Sc. 2017]
→ Induced stress, in turn affects chemistry

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→ Induced stress, i
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Silicon electrodes: mechanics
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Notion of stress
Stress in a thin film
In the plane:
Along the normal to the plane:
LMS

Silicon electrodes: mechanics

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Capacity, mAh/g
low [Li]
high [Li]
Si
Li,Si

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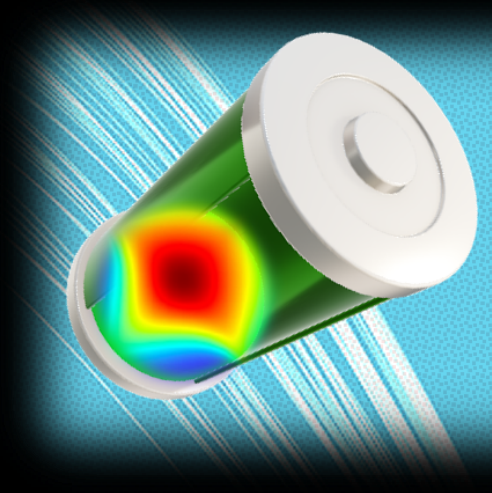

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Fingerprinting Ni L-edge Probes of Disproportionating Cathode $LiNiO_2$
Andrey Poletayev
THE FARADAY INSTITUTION

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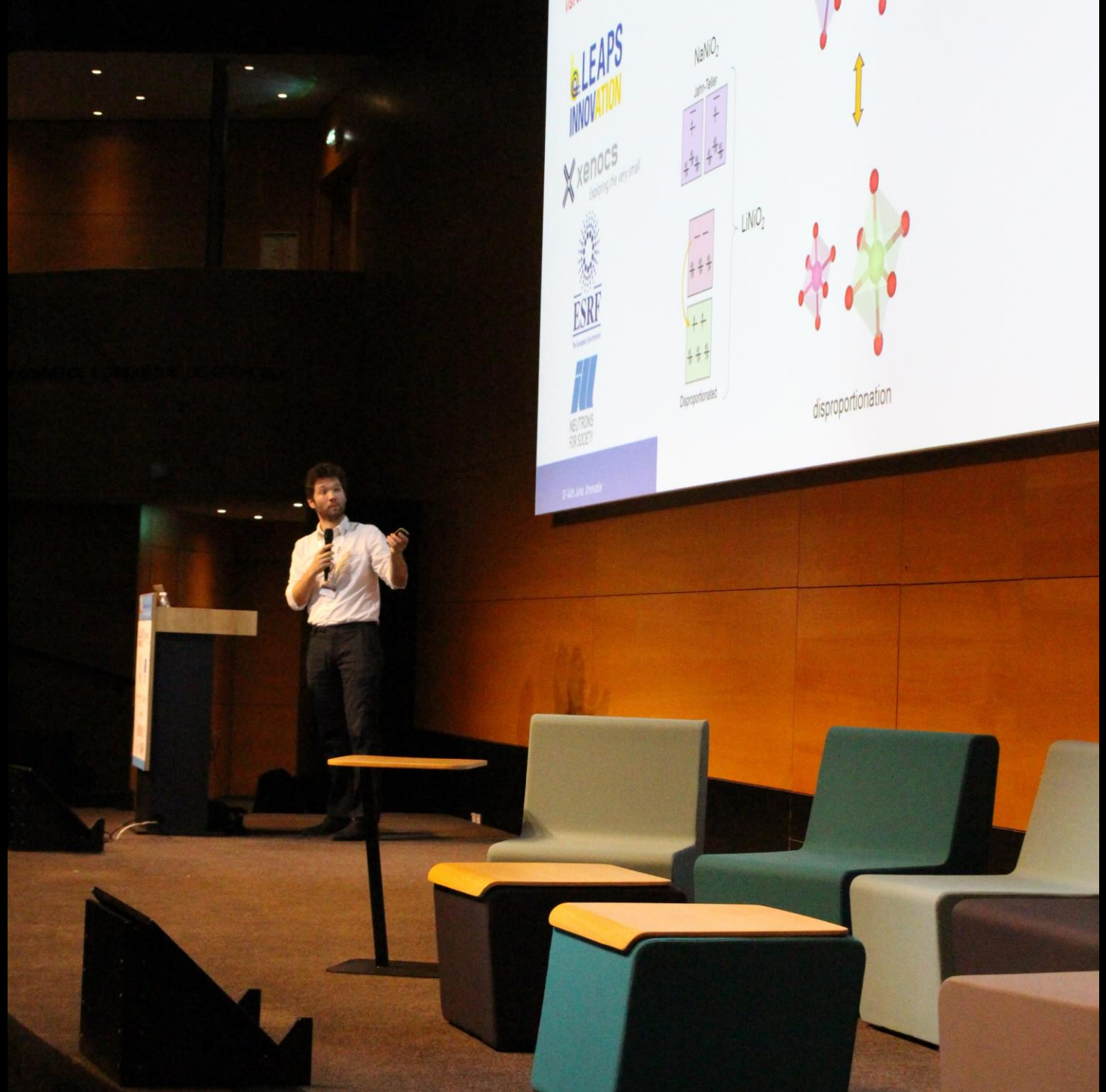
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NaNiO2
Jahn-Teller
LiNiO2
Disproportionated
disproportionation

Questions?
JACOME
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Grain boundary-controlled lithiation of Li-solid solution systems for lithium metal batteries
Leonardo Shoji Aota, Chanwon Jung, Siyuan Zhang, Ömer K. Büyükuslu, Poonam Yadav, Mahander Pratap Singh, Xinren Chen, Eric Woods, Christina Scheu, Se-Ho Kim, Dierk Raabe, Baptiste Gault
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Grain boundary-controlled li
solution systems for lithium
Leonardo Shoji Aota, Chanwon Jung
Büyükuslu, Poonam Yadav, Mahande
Eric Woods, Christina Scheu, Se-Ho K
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Introduction
SSE
SUS
Charge
Discharge
Cycling
Top
Middle
Bottom
Ag
C
Ag-Li alloy
Lee, YG et al. Nat Energy 5, 299–308 (2020)
Pt
Al
Mg
Zn
Ag
Au
0.05 V
0.00
0.02
0.04
Yan, K. et al. Nat Energy 1, 16010 (2016)
Max Planck Institute for Sustainable Materials | Leonardo Shoji Aota
2

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NEUTRONS FOR SOCIETY
Perpendicular to GB
GBPs
PFZ
Zhao, H. et al. Acta Materialia, v. 156, p. 318-329 (2018)
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NEUTRONS FOR SOCIETY
Within GB
Perpendicular to GB
GB
PFZ
Depletion
GBPs
PFZs
Zhao, H. et al. Acta Materialia, v. 156, p. 318-329 (2018)
Max Planck Institute for Sustainable Materials | Leonardo

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In situ structural investigation of dopant-modified Ni-rich cathode material for Li based batteries
Farheen N. Sayed
fs491@cam.ac.uk
Grey Group
University of Cambridge
12th June 2024
Operando Battery Conference, Grenoble
UNIVERSITY OF CAMBRIDGE
Department of Chemistry
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Yusuf Hamied
Department of Chemistry
Operando Battery C
12th Jun
University of
Grey

Lithium Nickel Oxide
Lithium Nickel Oxide, $LiNiO_2$ (LNO)
Layered structure with alternating Li and Ni layers
High theoretical capacity: 275 mAhg^{-1}
The diffusion of Li ions out of the layers leads to a series of phase transitions:
H1 -> M -> H2 -> H3
c = 14.4044(1) Å
c = 13.363(6) Å
H. Li et al. Chem Mater. 2021, 33, 9546–9559.
M. Bianchini et al. Angew. Chem. Int. Ed., 2019, 58, 10434–10458.
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Quantitative operando NEXAFS characterisation
of the solid electrolyte interphase formation
at the hard-carbon anode of Na-ion batteries
Katia Frenzel, Giorgia Greco[1], Lena Mathies, Adrian Jonas,
Philipp Hönicke[2], Yves Kayser[2], Philipp Adelhelm[4], Sergio Brutti[1], Burkhard Beckhoff
[1]University Sapienza, Rome, Italy
[2]Helmholtz-Zentrum Berlin, Germany
[3]MPG CEC, Mülheim, Germany
[4]Humboldt Universität zu Berlin, Germany
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Quantitative operando NEXAFS characterisation
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at the hard-carbon anode of Na-ion batteries
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Philipp Hönicke[2], Yves Kayser[3], Philipp Adelhelm[4], Sergio Brutti[1], Burkhard Beckhoff
[1] University Sapienza, Rome, Italy
[2] Helmholtz-Zentrum Berlin, Germany
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Chen X. et al. Carbon Energy. 2022;4:1133-1150. doi: 10.1002/cey2.196
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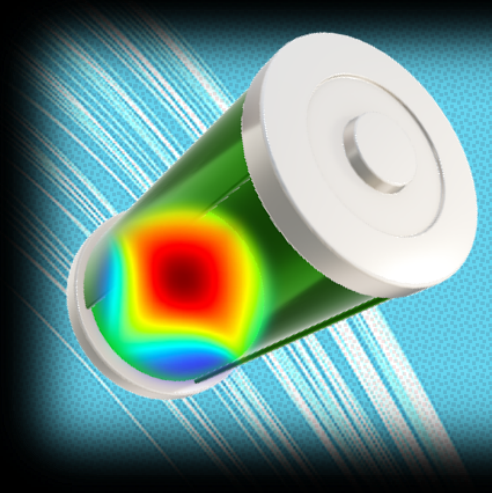

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Exploring beyond lithium-ion batteries using scattering techniques and advanced electrochemical analysis, from lab assessments to real-time monitoring.
Institute of Electrochemical Energy Storage
Helmholtz-Zentrum Berlin, Germany
Dr. Eneli Härk
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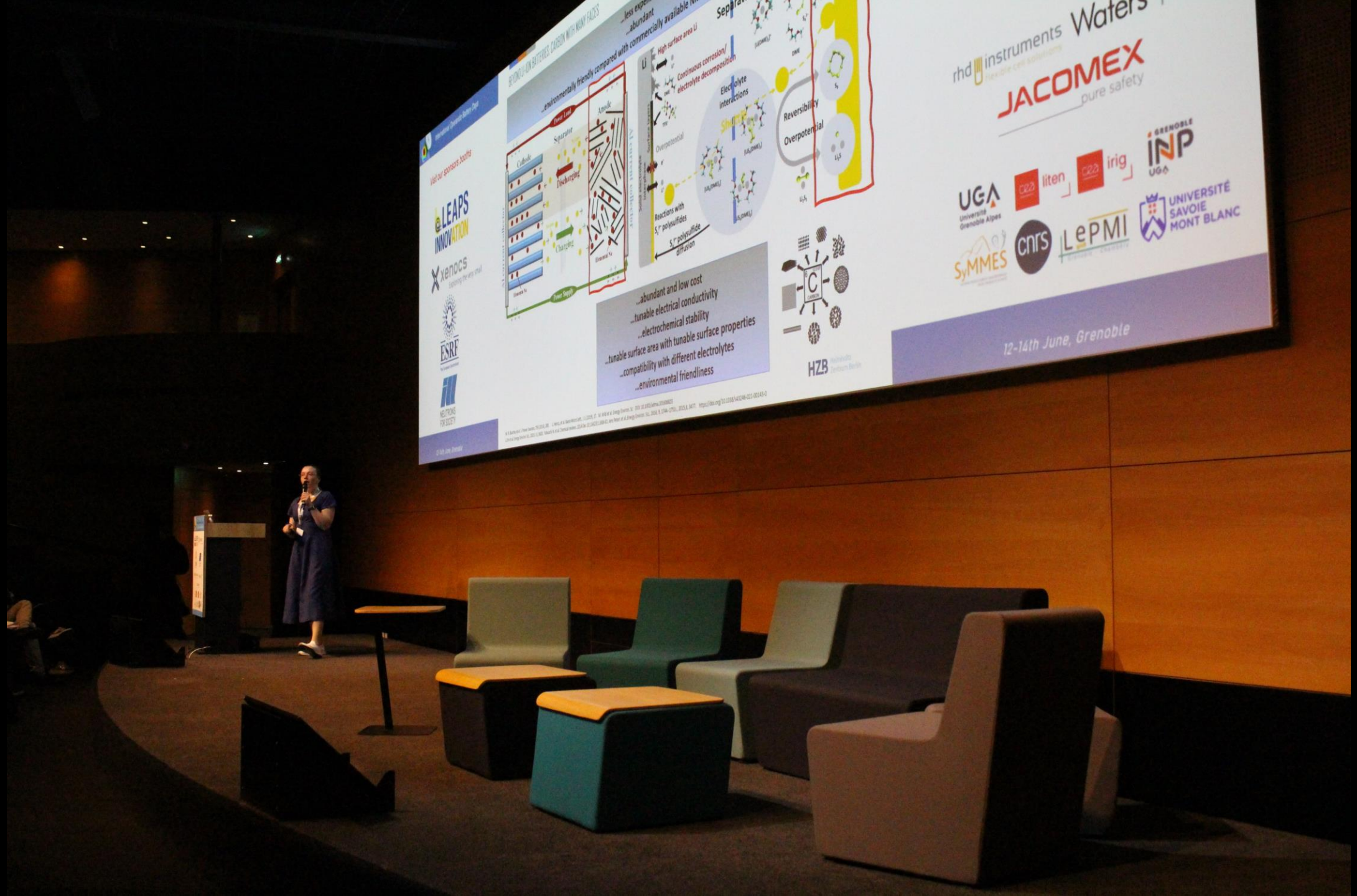
Scattering Technique
Structural Analysis
Electrochemical Analysis and Storage Mechanism
Remarks and Conclusion
neutrons
x-rays
by courtesy of Breno Luiz de Souza
$|q| = 4\pi \sin(\theta) / \lambda$
E/V
CAPACITY
S_8
Li_2S_x
Li_2S
HZB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...less expe
...abundant
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Anode
Discharging
Charging
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Power Supply
Li
High surface area Li
Continuous corrosion/ electrolyte decomposition
Overpotential
Electrolyte interactions
Reactions with S_x^{2-} polysulfides
S_x^{2-} polysulfide diffusion
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...electrochemical stability
...tunable surface area with tunable surface properties
...compatibility with different electrolytes
...environmental friendliness
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New insights into limiting processes of all electrochemically active anode less solid state batteries using micro XRD.
Quentin Jacquet[a], J. Cèle[b], L. Casiez[b], A. Medjahed[d], M. Burghammer[d], S. Tardif[c], S. Oukassi[b], S. Lyonnard[a]
[a]Univ. Grenoble, Alpes, CEA, CNRS, IRIG, SyMMES, F-38000 Grenoble,
[b]Univ. Grenoble Alpes, CEA, Leti Grenoble F-38000, France,
[c]Univ. Grenoble Alpes, CEA, IRIG, MEM, 38000 Grenoble, France
[d]European Synchrotron Radiation Facility (ESRF), CS 40220, 71 Avenue des Martyrs, 38043 Grenoble, France
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state batteries using mi
Quentin Jacquet[a], J. Cèle[b], L. Casie
Tardif[c], S. Oukassi[b], S. Lyonnard[a]
[a]Univ. Grenoble, Alpes, CEA, CNRS, IRIG, SyMMES, F-38000
[b]Univ. Grenoble Alpes, CEA, Leti Grenoble F-38000, France
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[d]European Synchrotron Radiation Facility (ESRF), CS 4022
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Operando µXRD on LiCoO2 micro
Beam stopper
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Potentiostat
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MULTIMODAL OPERANDO ANALYSES
of Li-S pouch cells
12.06.2024 Dr. Rafael Müller
HZB – CE-IEES – Risse group
inside batteries live

OUTLINE
1. Concept
2. Pouch cell setup
3. Operando results
• Monolayer
• Electrolytes
• Multilayer
• Perforation
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Pouch cells and components
Wingcopter (KMU)
Multipurpose drones for delivery and mapping

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On the effect of the degree of graphitization on the Na+-diglyme co-intercalation mechanism of graphitic and non-graphitic Soft Carbon anodes by Operando PXRD.
Maria Jauregui
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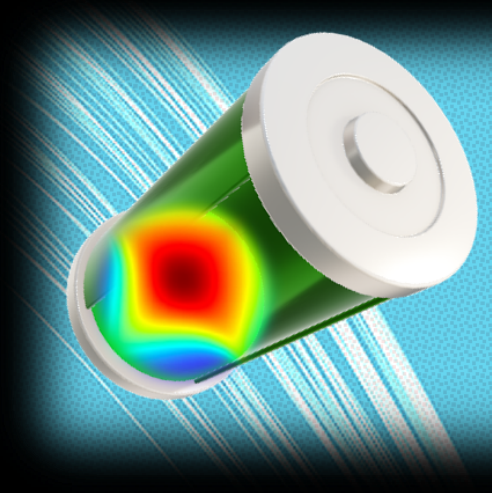

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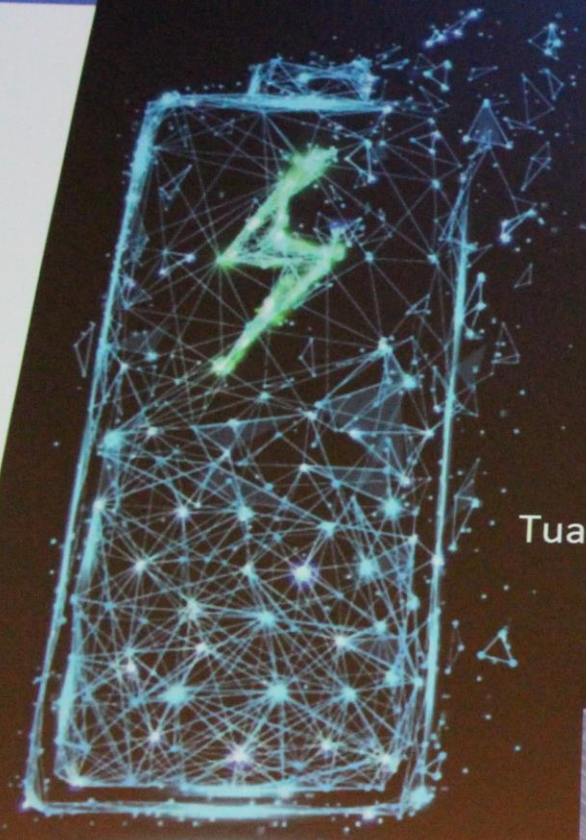
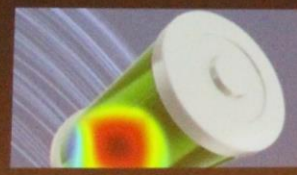


Combining X-ray Nano-CT and XANES Techniques for 3D Operando Monitoring of Lithiation Spatial Composition evolution in NMC Electrode

Arnaud Demortière

Tuan-Tu Nguyen, Zeliang Su, Jiahui Xu, Antoine BottenMuller, Kevyn Gallegos Moncayo, Florent Magaud, Fayçal Adrar, Petr Dokladal, Julie Villanova Mario Scheel, Timm Weitkamp, Viktor Nikitin, Vincent De Andrade



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Impact of formation cycling in Li-metal batteries

Swapna Ganapathy

Delft University of Technology
Department of Radiation Science and Technology

TU Delft

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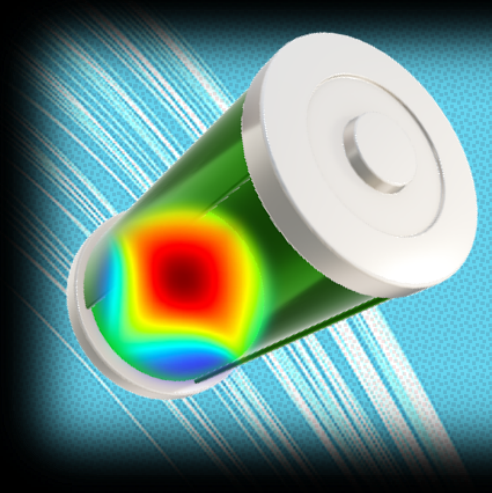

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Fast Track 1

Understanding the editorial process at Nature journals

Rinaldo Raccichini, PhD

Senior Editor, *Nature Communications*

rinaldo.raccichini@nature.com

in rinaldoraccichini

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How can LEAPS facilities collaborate in Operando battery research?

https://www.leaps-innov.eu/

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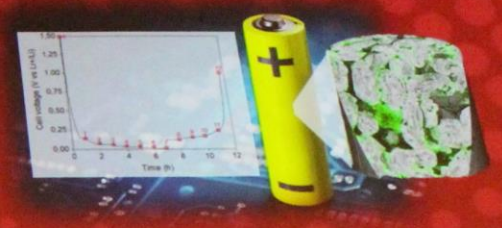

Going toward operando X-ray nano-tomography: the challenges?

V. Vanpeene[a,b], O. Stamati[b], C. Guilloud[b], R. Tucoulou[b], B. Holliger[b], M. Chandesris[a], S. Lyonnard[c] & J. Villanova[b]

[a] Univ. Grenoble Alpes, CEA, Liten, DEHT, 38000 Grenoble, France
[b] ESRF-The European Synchrotron, F-38000 Grenoble, France.
[c] Univ. Grenoble Alpes, CEA, CNRS, Grenoble INP, IRIG, SyMMES, 38000 Grenoble, France

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Quantifying Heterogeneous Degradation Pathways and Deformation Fields in All Solid-State Batteries

Partha P. Paul (ppaul@esrf.fr)
Visiting Scientist, ESRF
Senior Research Fellow, The University of Manchester

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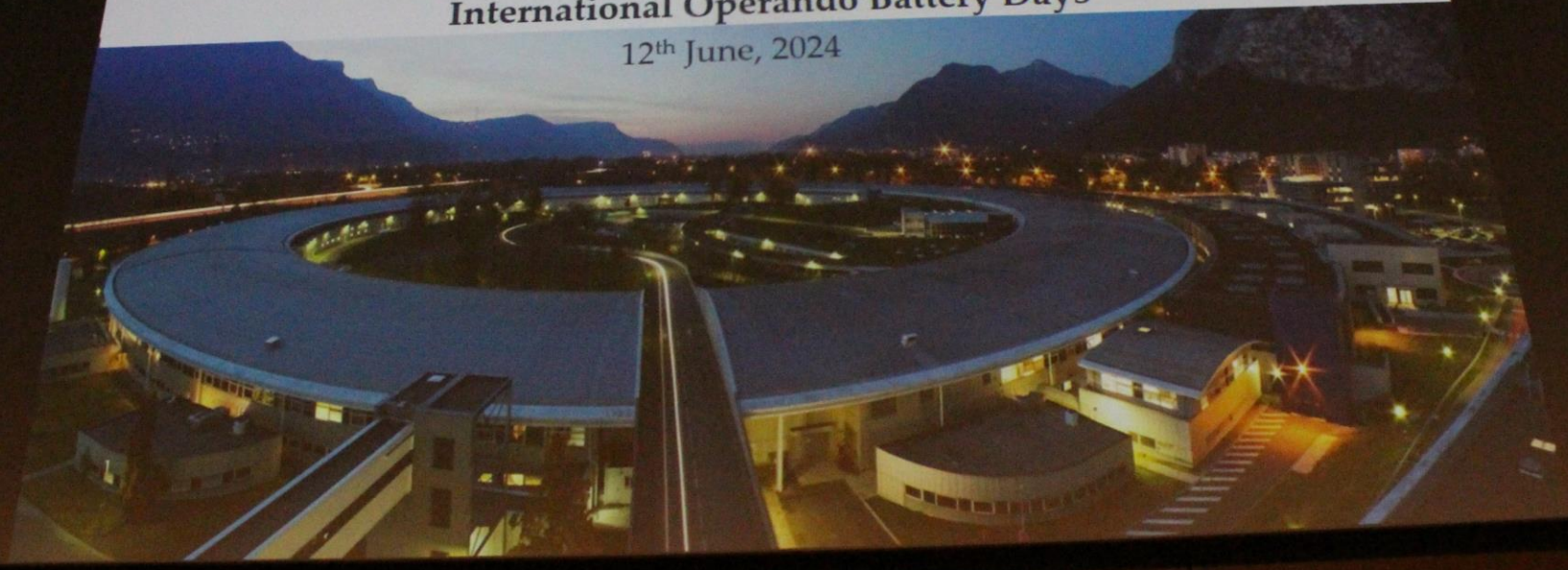

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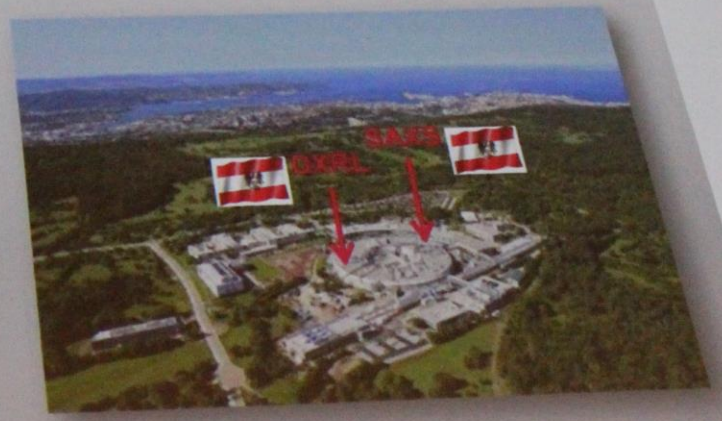
Operando GISAXS to study materials for energy storage

Philipp Aldo Wieser, Simone Dal Zilio, David Moser, Bernhard Gollas, Heinz Amenitsch

Elettra Sincrotrone Trieste



Institute of Inorganic Chemistry, Graz University of Technology, Graz, Austria & Austrian SAXS Beamline, Elettra-Sincrotrone Trieste, Italy

philipp.wieser@tugraz.at

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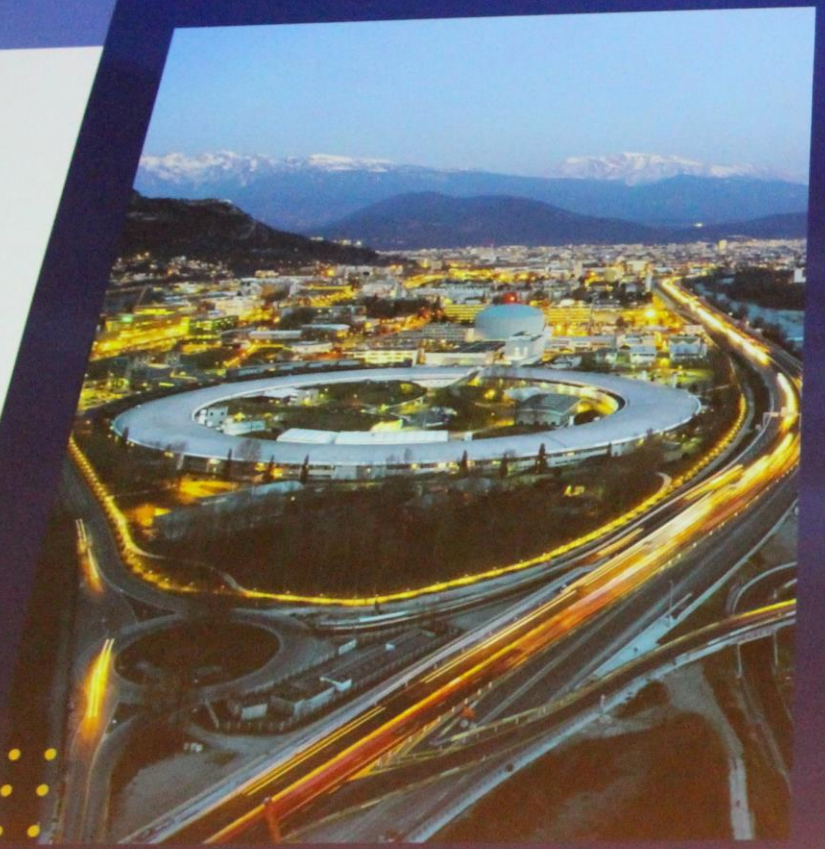

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ESRF: Pushing the boundaries of material characterisation.

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Business Development Officer, ESRF

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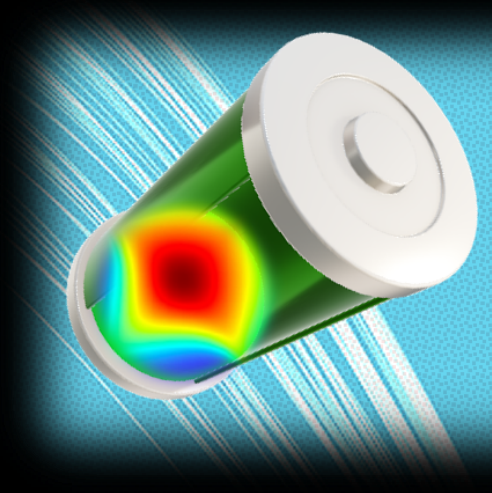

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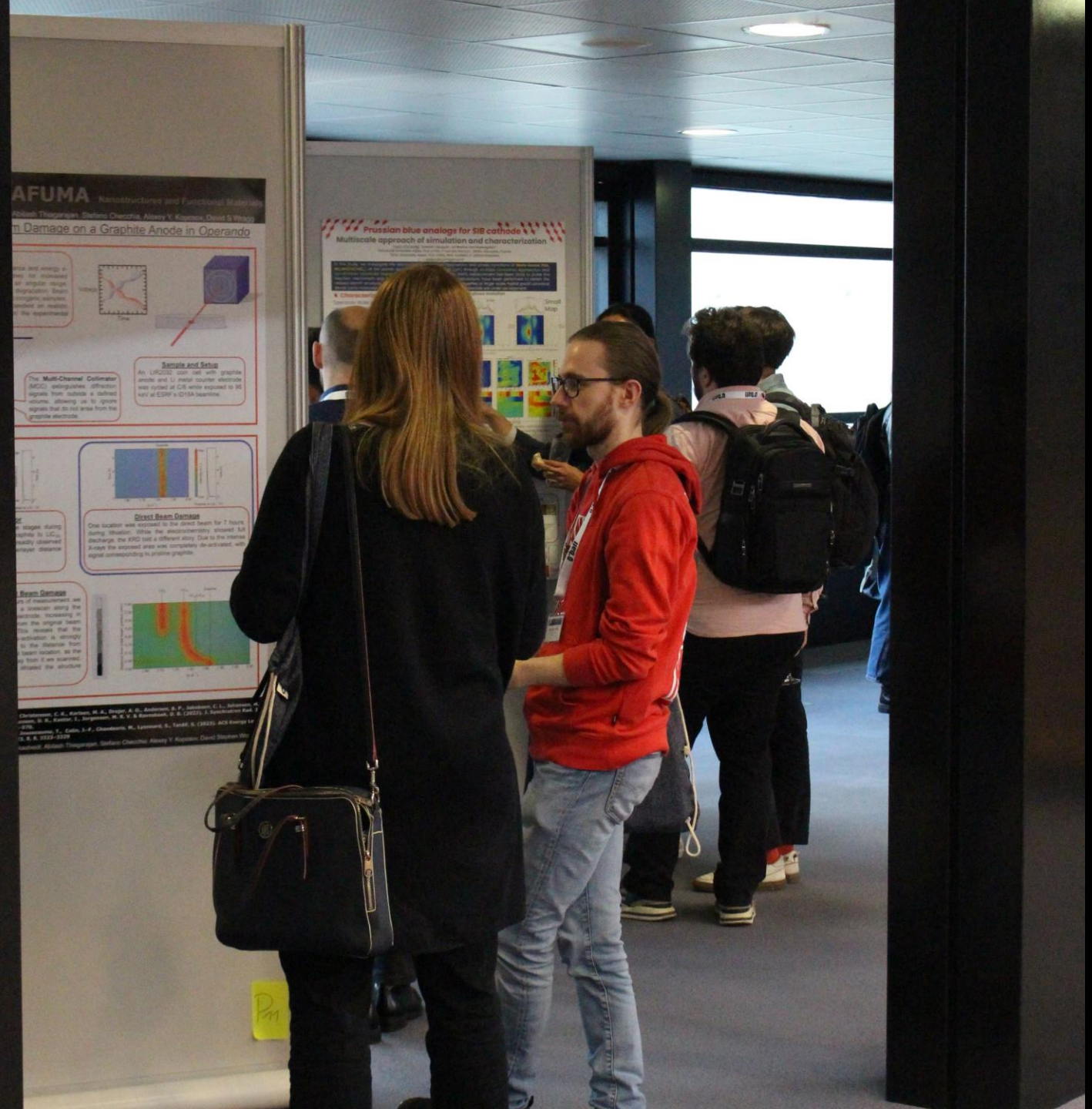
Direct Beam Damage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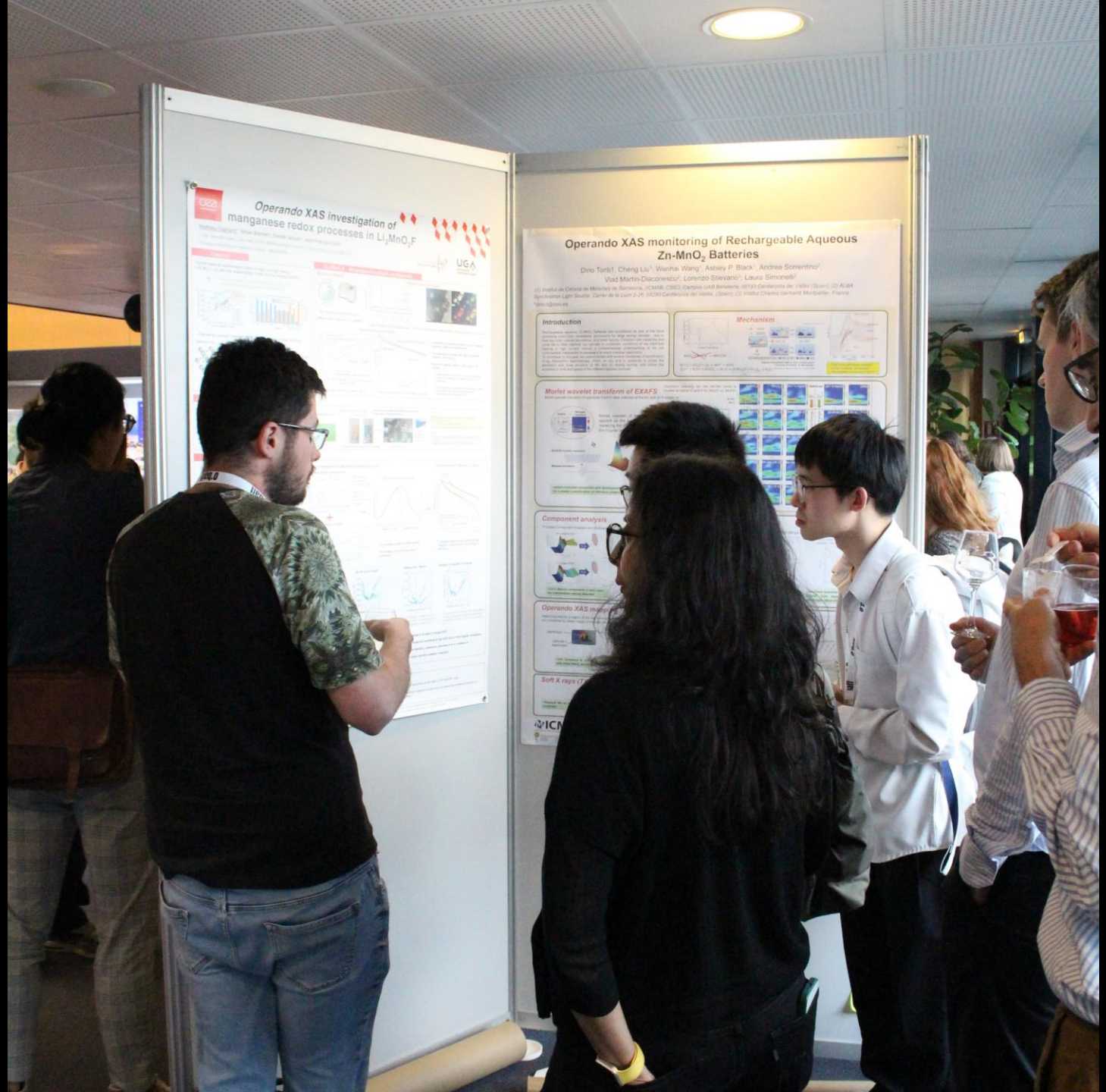
Damage on a Graphite Anode in Operando
Sample and Setup
Direct Beam Damage
Prussian blue analogs for SIB cathode
Multiscale approach of simulation and characterization


Operando XAS investigation of manganese redox processes in Li_2MnO_2F
Operando XAS monitoring of Rechargeable Aqueous Zn-MnO_2 Batteries
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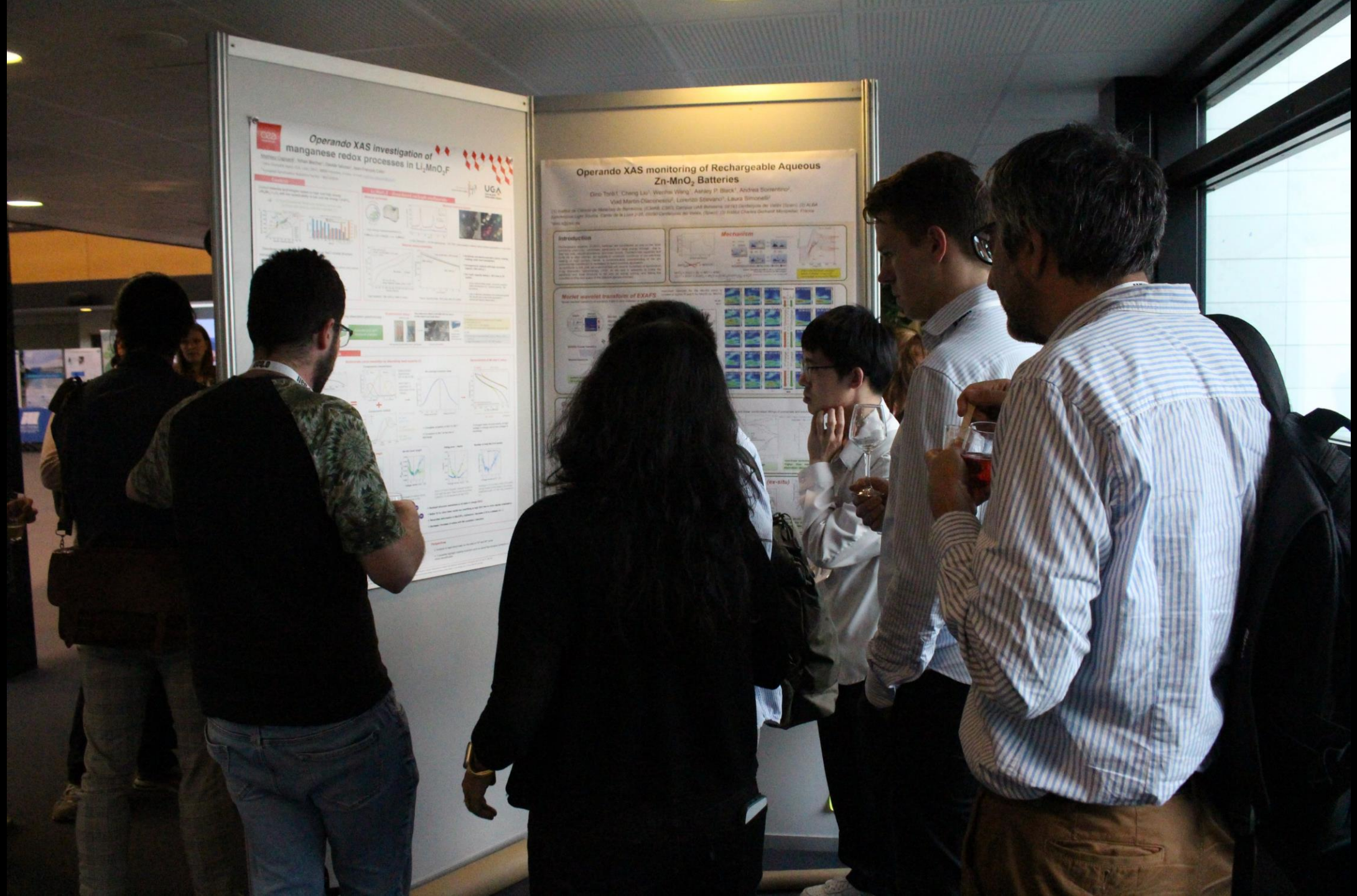
Mechanism
Morlet wavelet transform of EXAFS
Component analysis


Operando XAS investigation of manganese redox processes in Li_2MnO_3F
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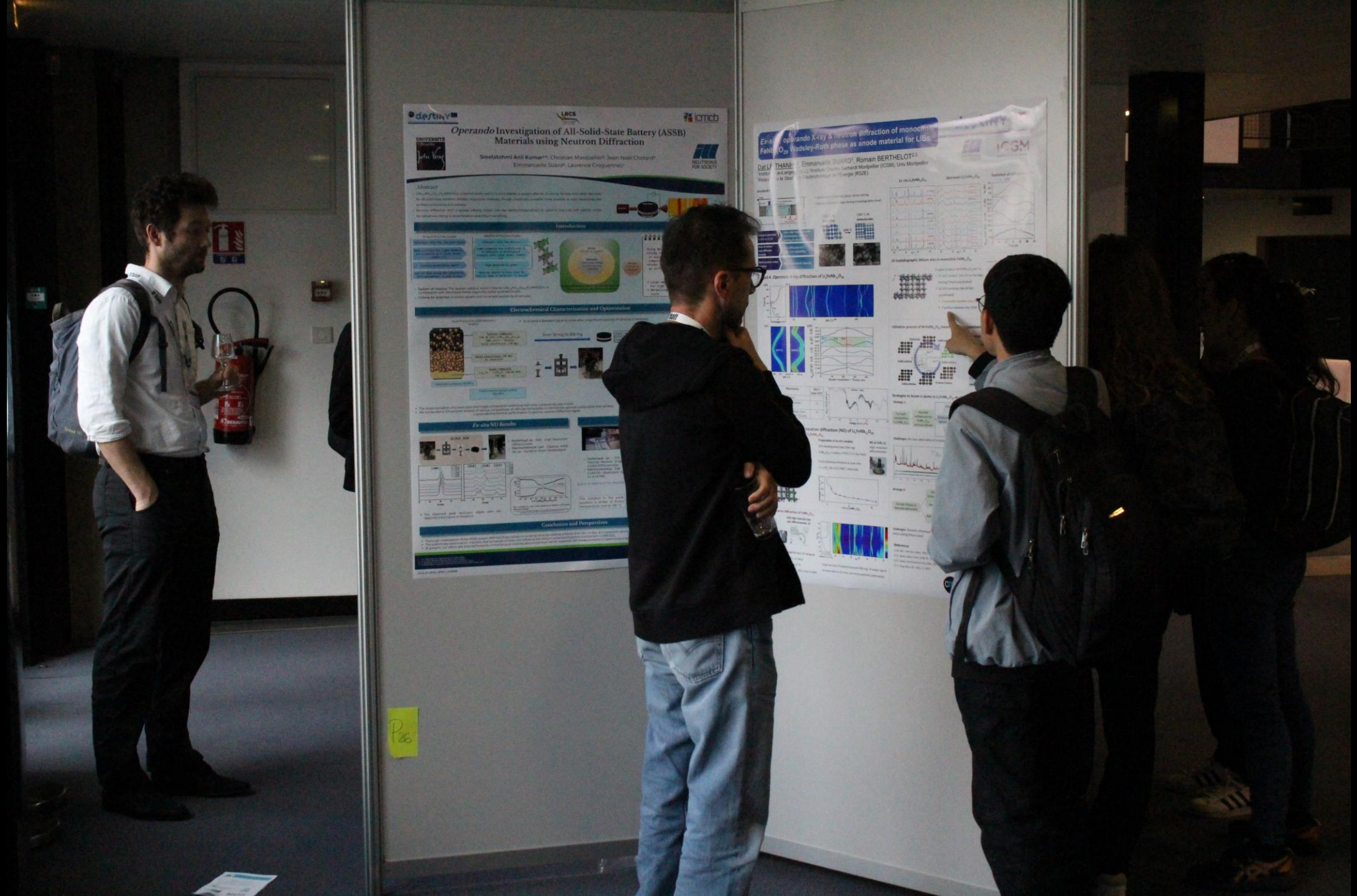
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Operando Investigation of All-Solid-State Battery (ASSB) Materials using Neutron Diffraction
Sreelakshmi Anil Kumar, Christian Masquelier, Jean-Noël Chotard, Emmanuelle Suard, Laurence Croguennec
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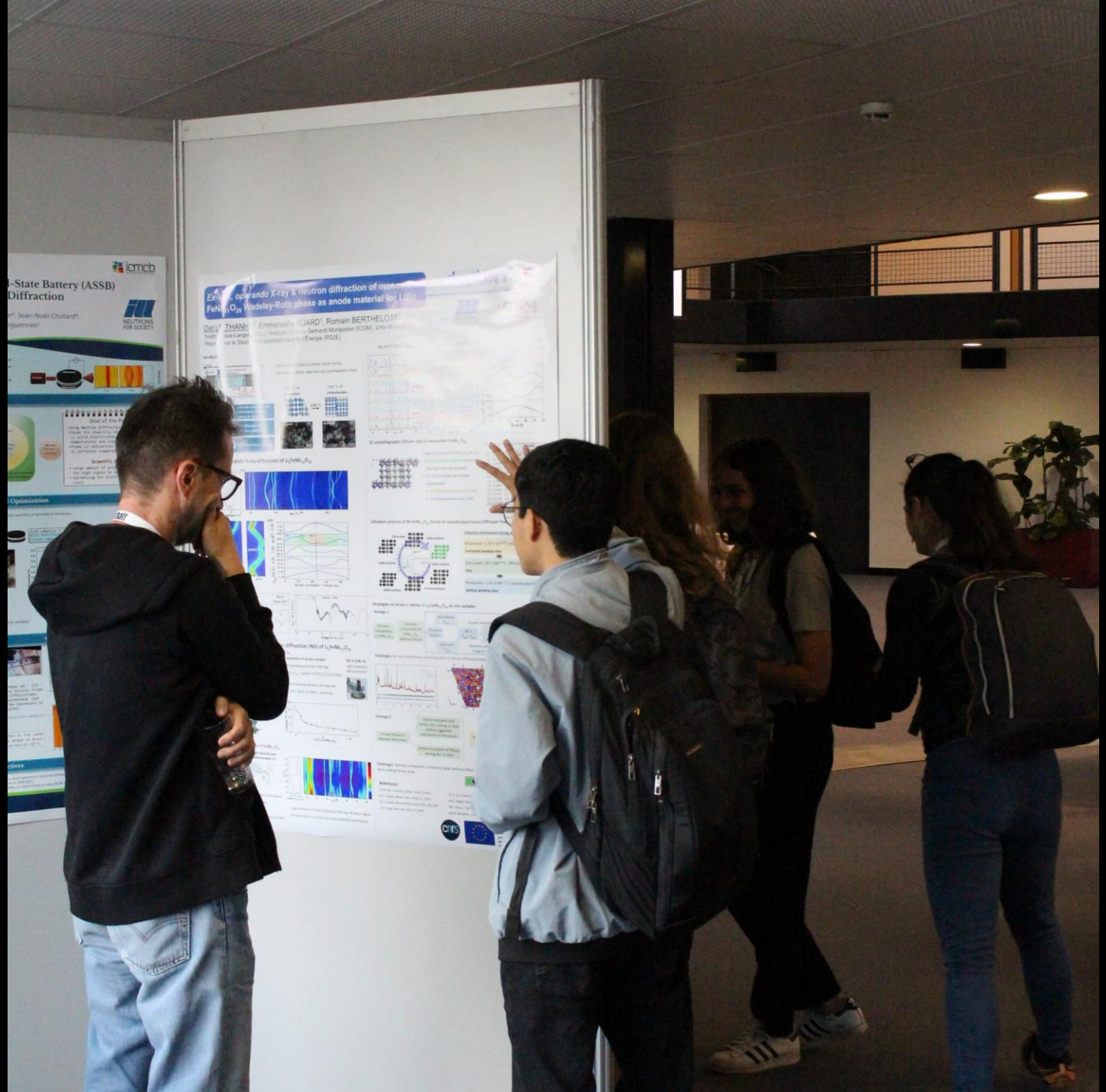
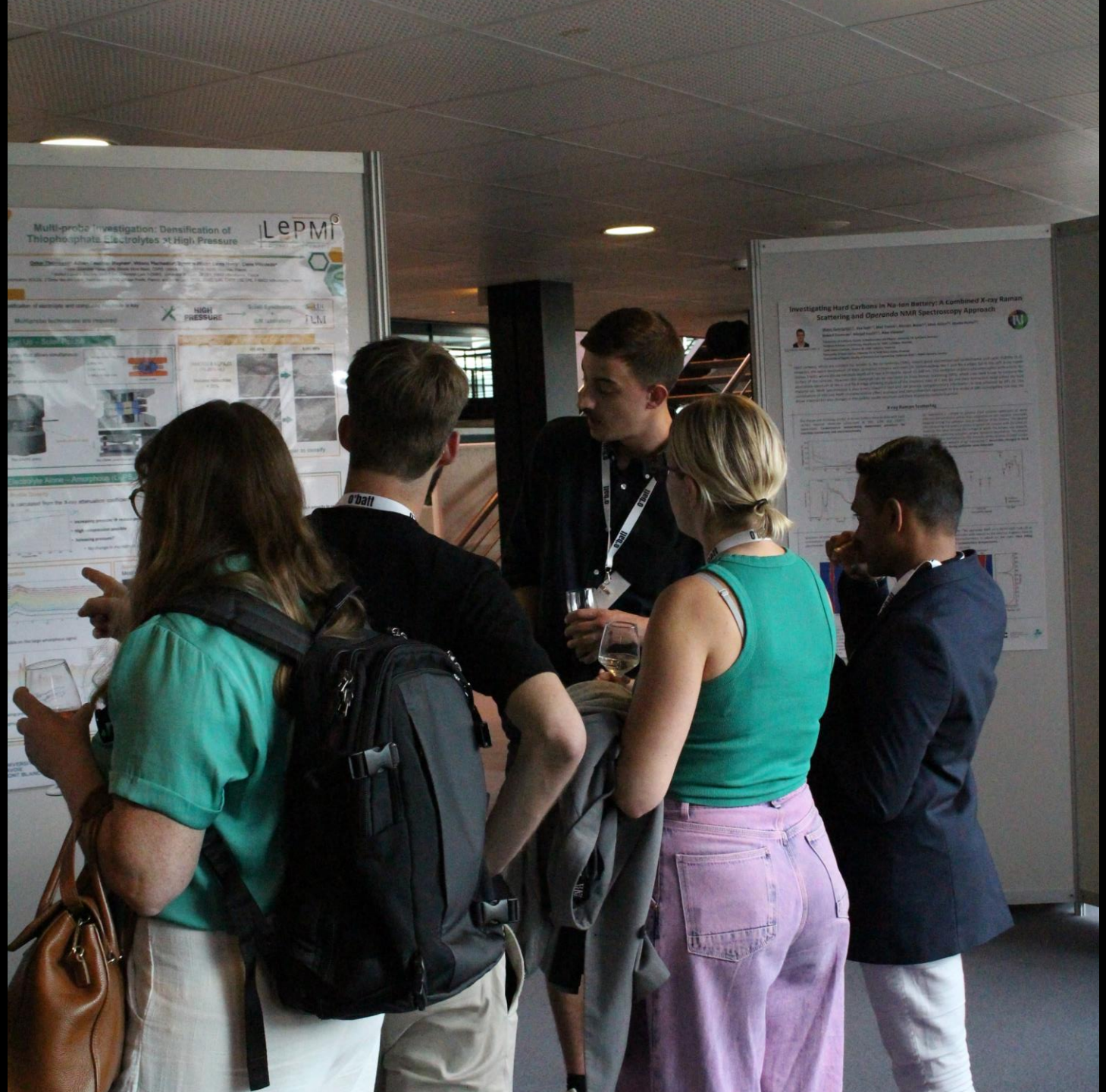
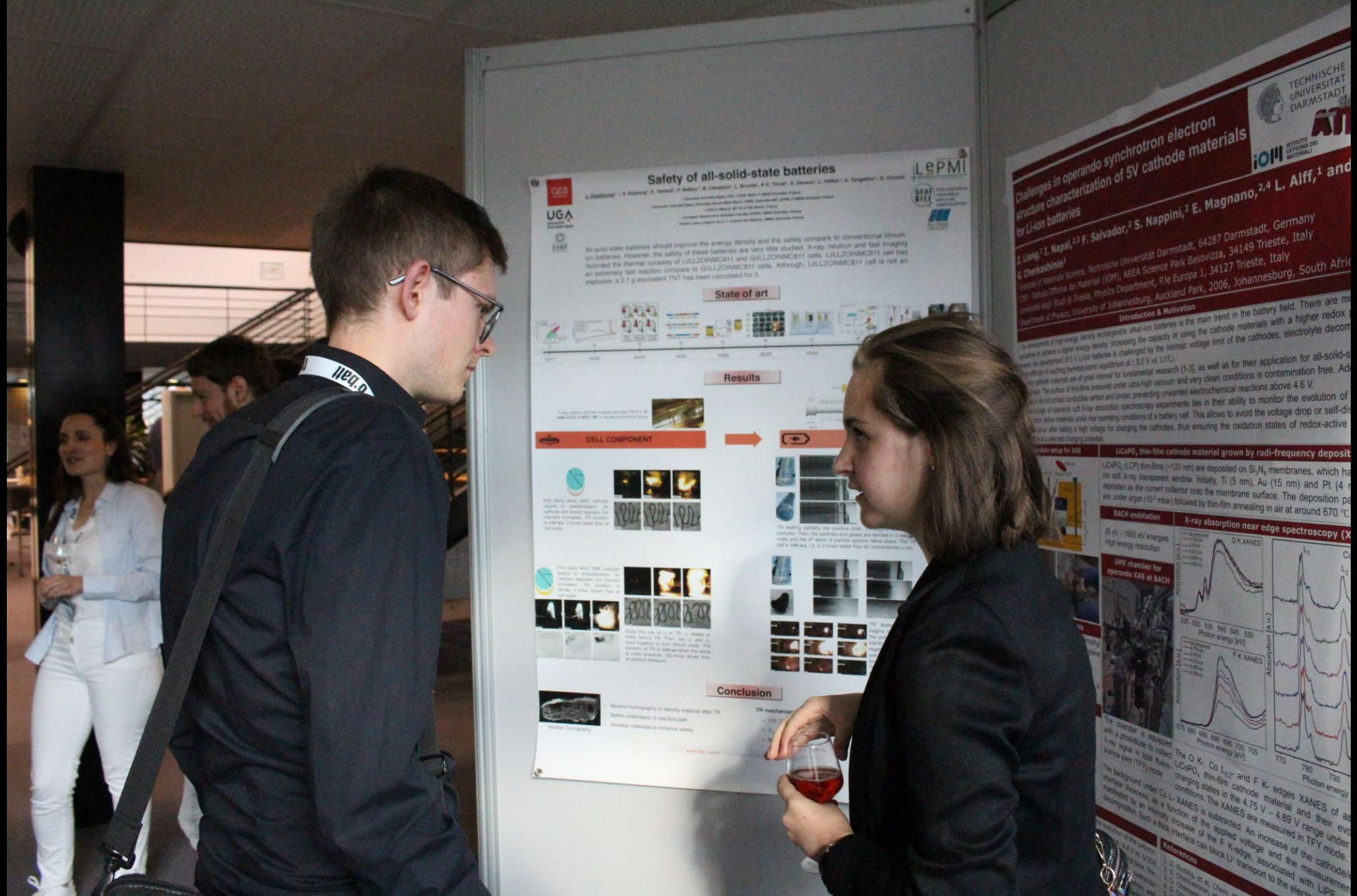
Conclusion and Perspectives
Emmanuelle SUARD, Romain BERTHELOT
ICGM
P26

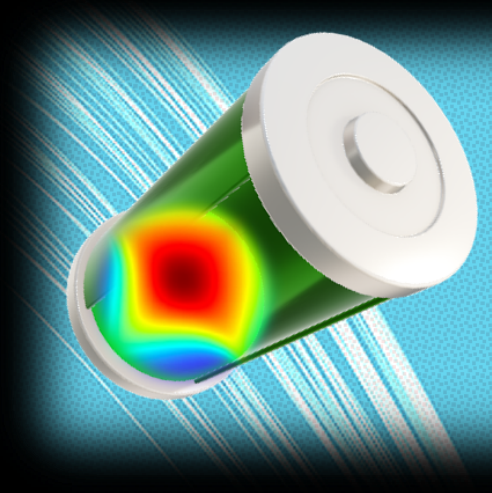

icmcb
-State Battery (ASSB)
Diffraction
NEUTRONS FOR SOCIETY
Optimization
Emmanuelle SUARD, Romain BERTHELOT

LEPMI
UGA
INP


LEPMI
Multi-probe Investigation: Densification of Thiophosphate Electrolytes at High Pressure
HIGH PRESSURE
Investigating Hard Carbons in Na-ion Battery: A Combined X-ray Raman Scattering and Operando NMR Spectroscopy Approach
X-ray Raman Scattering

Challenges in operando synchrotron electron structure characterization of 5V cathode materials for Li-ion batteries


Safety of all-solid-state batteries
LEPMI
State of art
Results
CELL COMPONENT
Conclusion
Challenges in operando synchrotron electron structure characterization of 5V cathode materials for Li-ion batteries
TECHNISCHE UNIVERSITÄT DARMSTADT
Introduction & Motivation
BACH endstation
UHV chamber for operando XAS at BACH
O K XANES
F K XANES
References



International Operando Battery Days

Grenoble, France, **12-14 June** 2024

Day One

12.06.2024

On the sides

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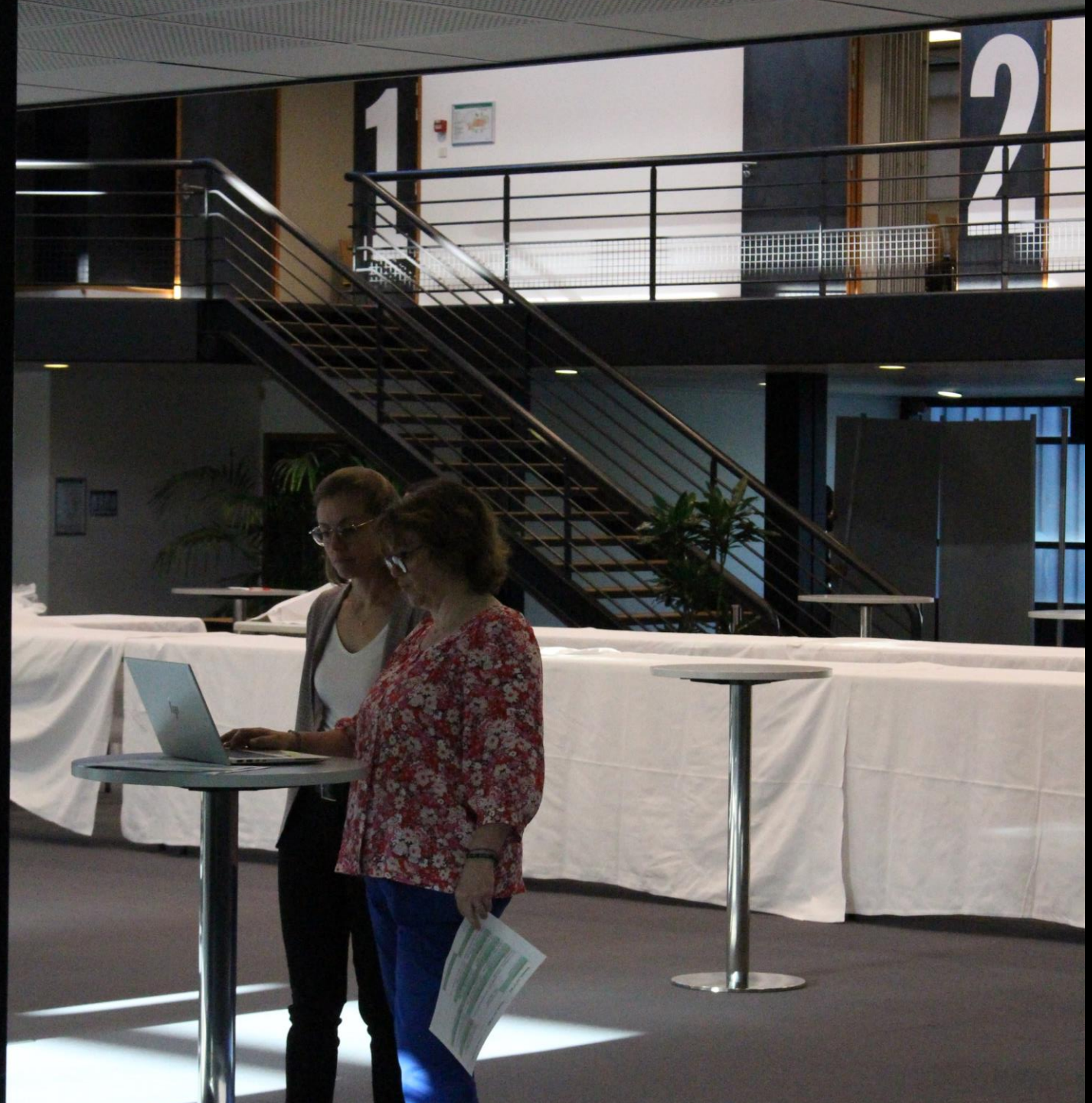
DURABLE



CONSIGNES DE SECURITE
PLAN D'EVACUATION
WORLD TRADE CENTER

Nos vies
changent

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A to D

A to D

MONT-BLANC
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Organization
- OBstt 2024 -

Tapez ici pour effectuer une recherche

4

MAKALU

MAKALU

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High-throughput analytical services
ESRF
The European Synchrotron
A hub for materials research
Access Routes
User Support
Find us here!
NEUTRONS FOR SOCIETY
NEUTRONS FOR SOCIETY

Grenoble

Time	Wednesday, June 12th
8:00	Registration
9:00	Welcome speech
9:10	H-G. Steinruck
9:40	A. Black
10:00	L. Guin
10:20	Coffee Break
10:50	A. Poletayev
11:10	L. Shoji Aota
11:30	F. Sayed
11:50	K. Frenzel
12:10	Lunch
14:00	E. Härk
14:30	Q. Jacquet
14:50	R. Müller
15:10	M. Jauregui
15:30	Coffee break
16:00	A. Demortière
16:30	M. Wagemaker
16:50	R. Raccichini J. Drnec V. Vanpeene ~~J. Hu~~ Partha Paul P. A. Wieser T. PAPAZOGLOU
18:15 21:00	Poster session

Time	Thursday, June 13th
8:30	E. Berg
9:00	L. Simonelli M. Johansen J. Pfaff A. Skurtvelt J. Vijayakumar S. Belin
10:20	Coffee Break
10:50	P. Perrenot
11:10	M. Löw
11:30	B. Hu
11:50	O. Korjus
12:10	Lunch
14:00	D. Ravnsbaek
14:30	S. Risse
14:50	C. Renais
15:10	J. Rizell
15:30	Coffee Break
16:00	M. Pasta
16:30	E. Lübke
16:50	I. Yousef C. Lopez E. Woillez T. Nguyen
19:30	Gala Dinner

Time	
8:30	
9:00	
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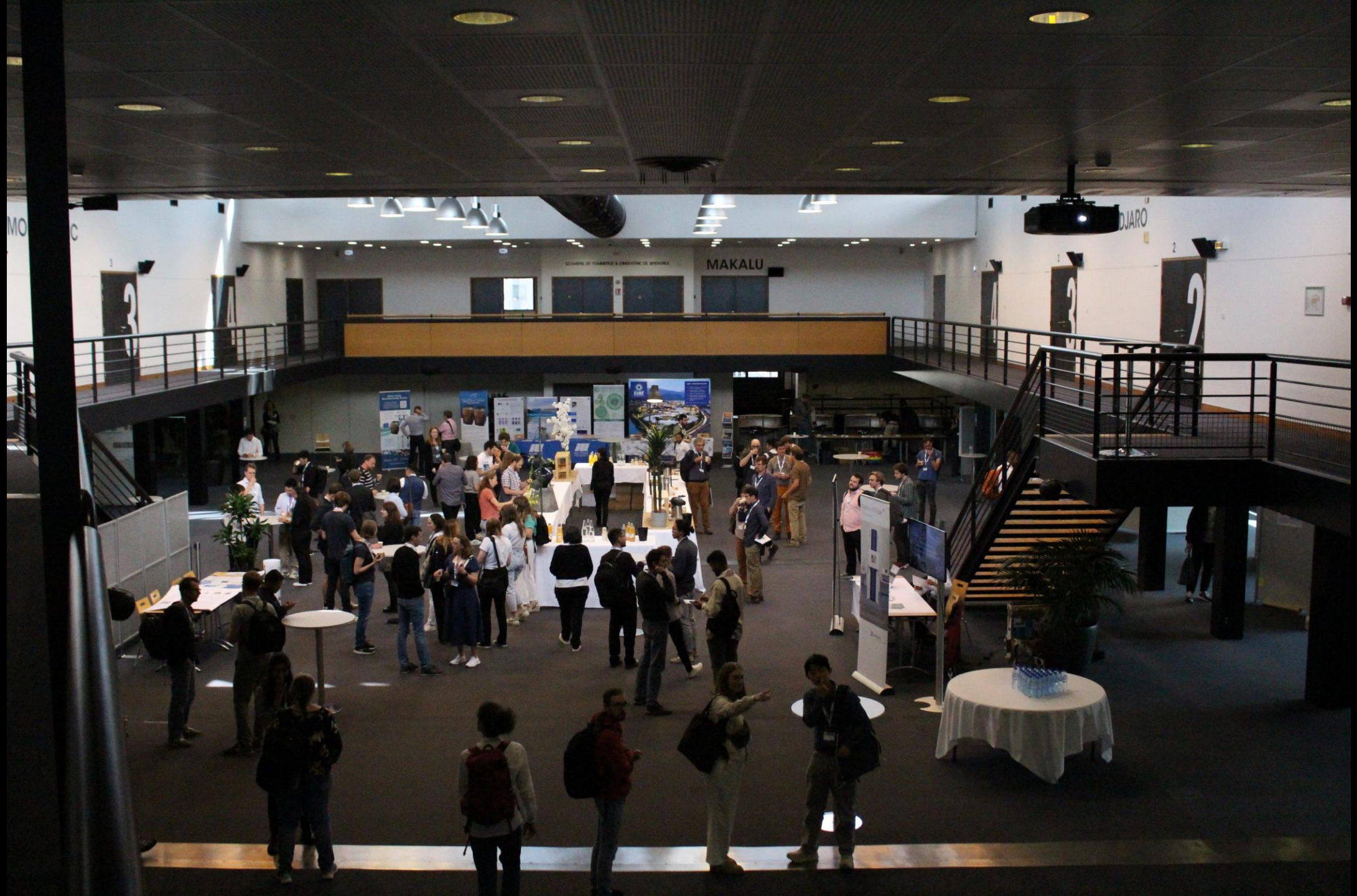
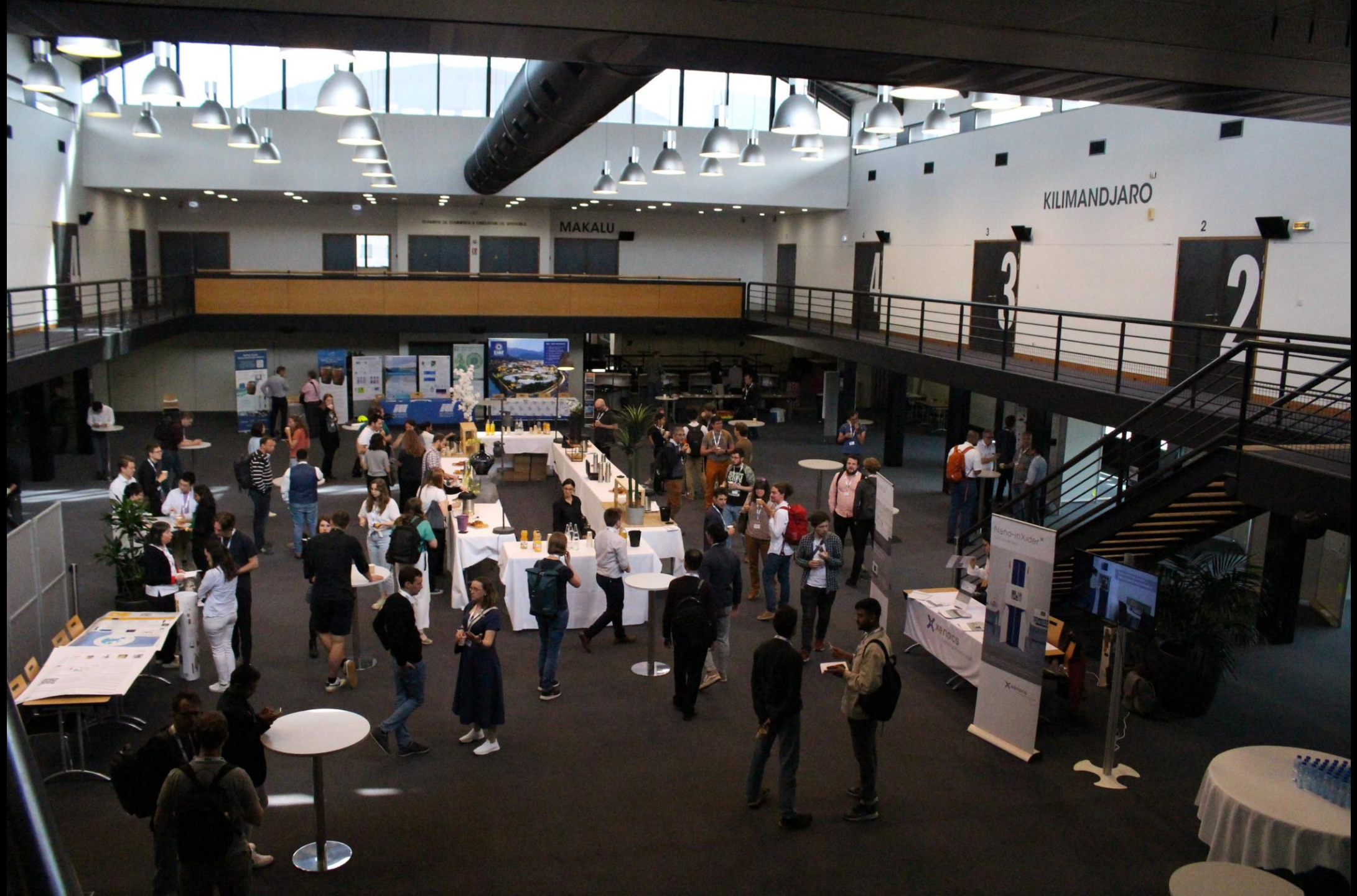
12:20	Prizes
12:40	
14:00	Visit


MAKALU
DJARO


MAKALU
KILIMANDJARO
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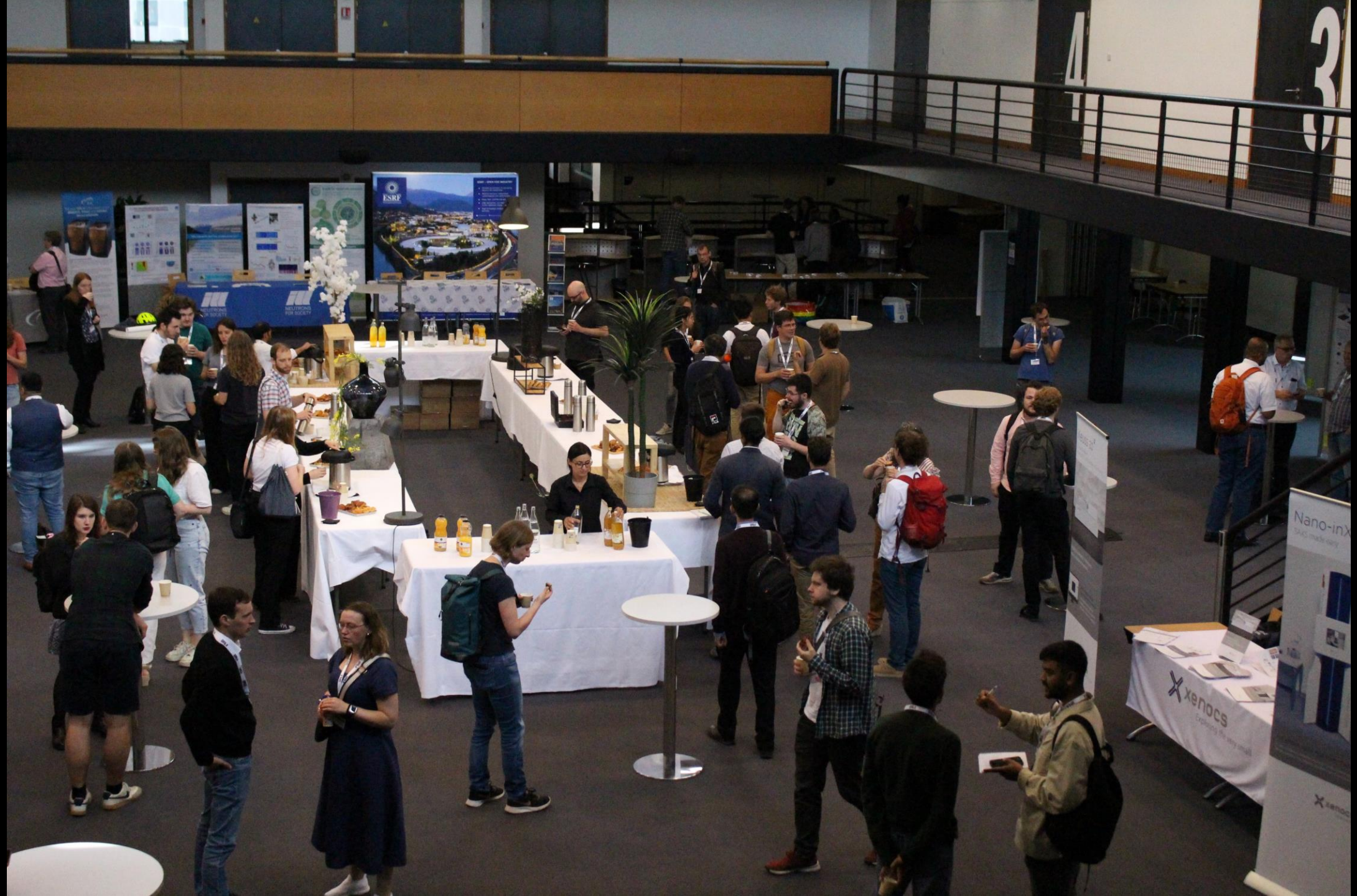
Xenocs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Xenocs
Nano-inX

KILIMANDJARO
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xenocs

Battery Cycler
Microcalorimeter Solution
Waters
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tainstruments.com
The new TAM IV is the world's most
SENSITIVE, STABLE and FLEXIBLE
Microcalorimeter
INSTITUT LAUE LANGEVIN
Grenoble
Why Operando BATTery studies at the ILL?
A hub for materials research
Access Routes
User Support
ESRF
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NEUTRONS
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MONT-BLANC
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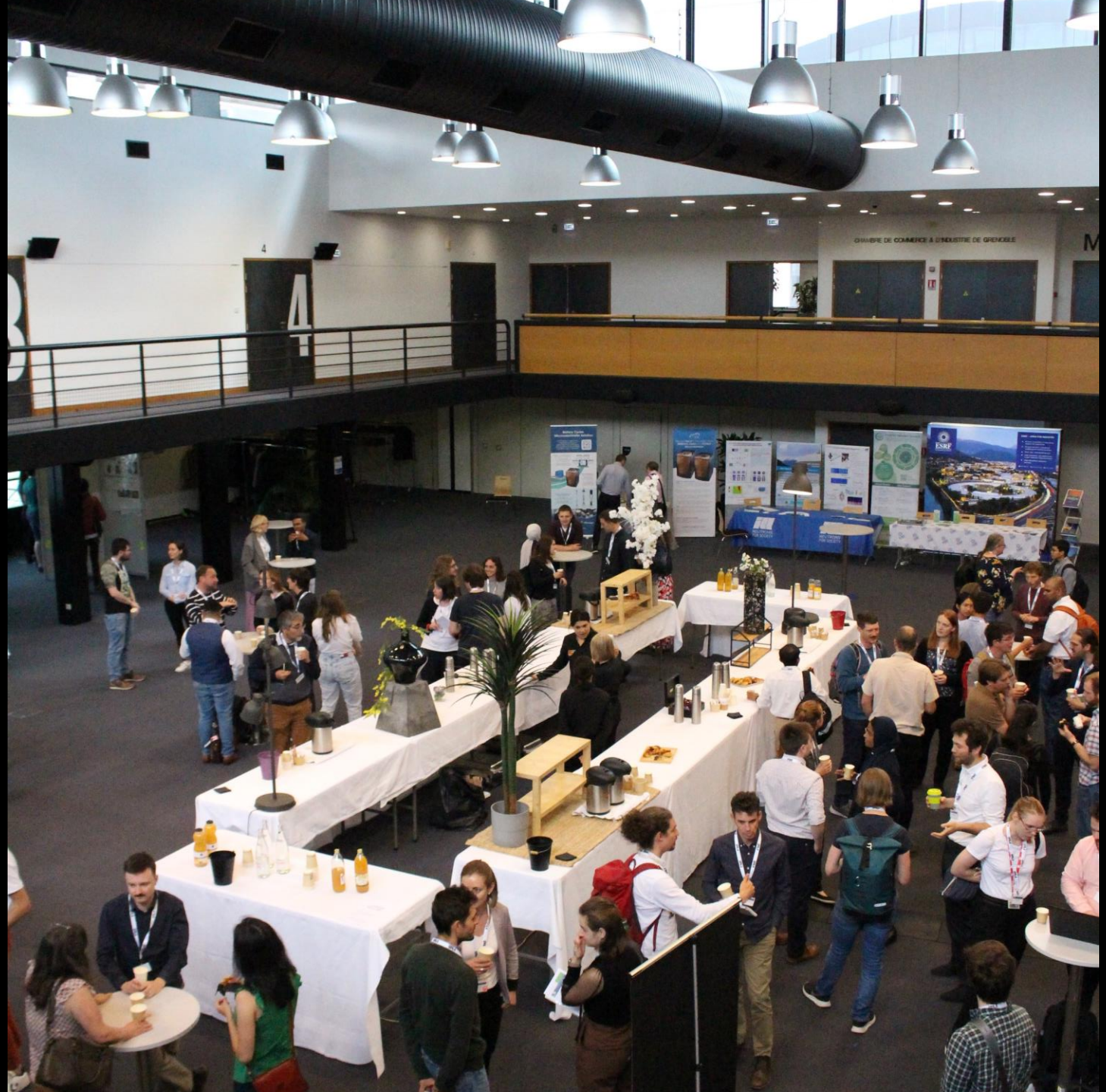
ESRF


CHAMBRE DE COMMERCE & D'INDUSTRIE DE GRENOBLE
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NEUTRONS FOR SOCIETY
NEUTRONS FOR SOCIETY

LEAPS
INNOVATION
xenocs
ESRF
Fingerprintin
Cathode LiN
Andrey Poletayev

LEAPS INNOVATION
xenocs
ESRF
Fingerprinting Ni L-edge Probes of Disproportionating Cathode $LiNiO_2$
Andrey Poletayev
THE FARADAY INSTITUTION

KILIMANDJARO
MAKALU
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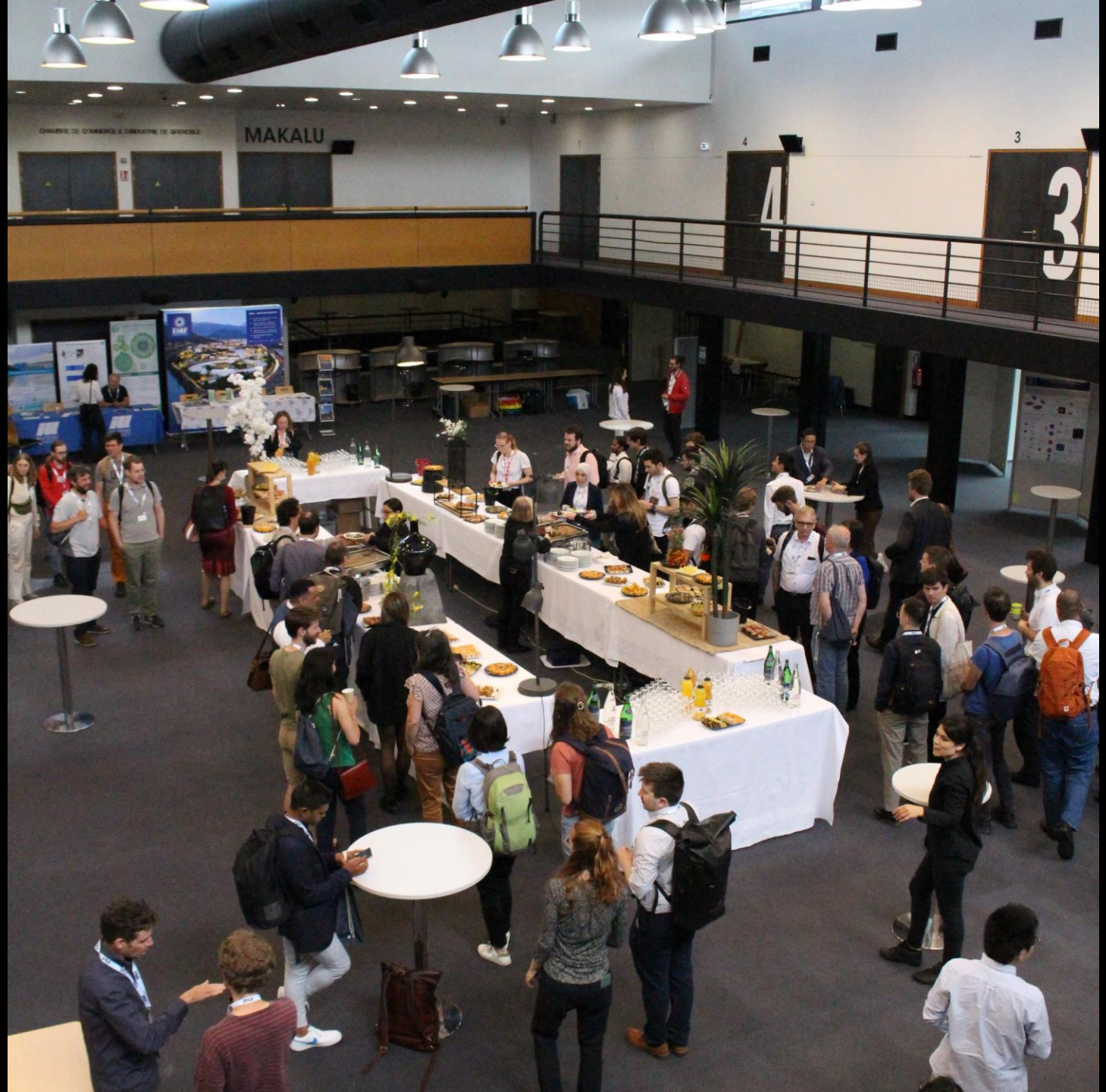
MAKALU
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KILIMANDJARO
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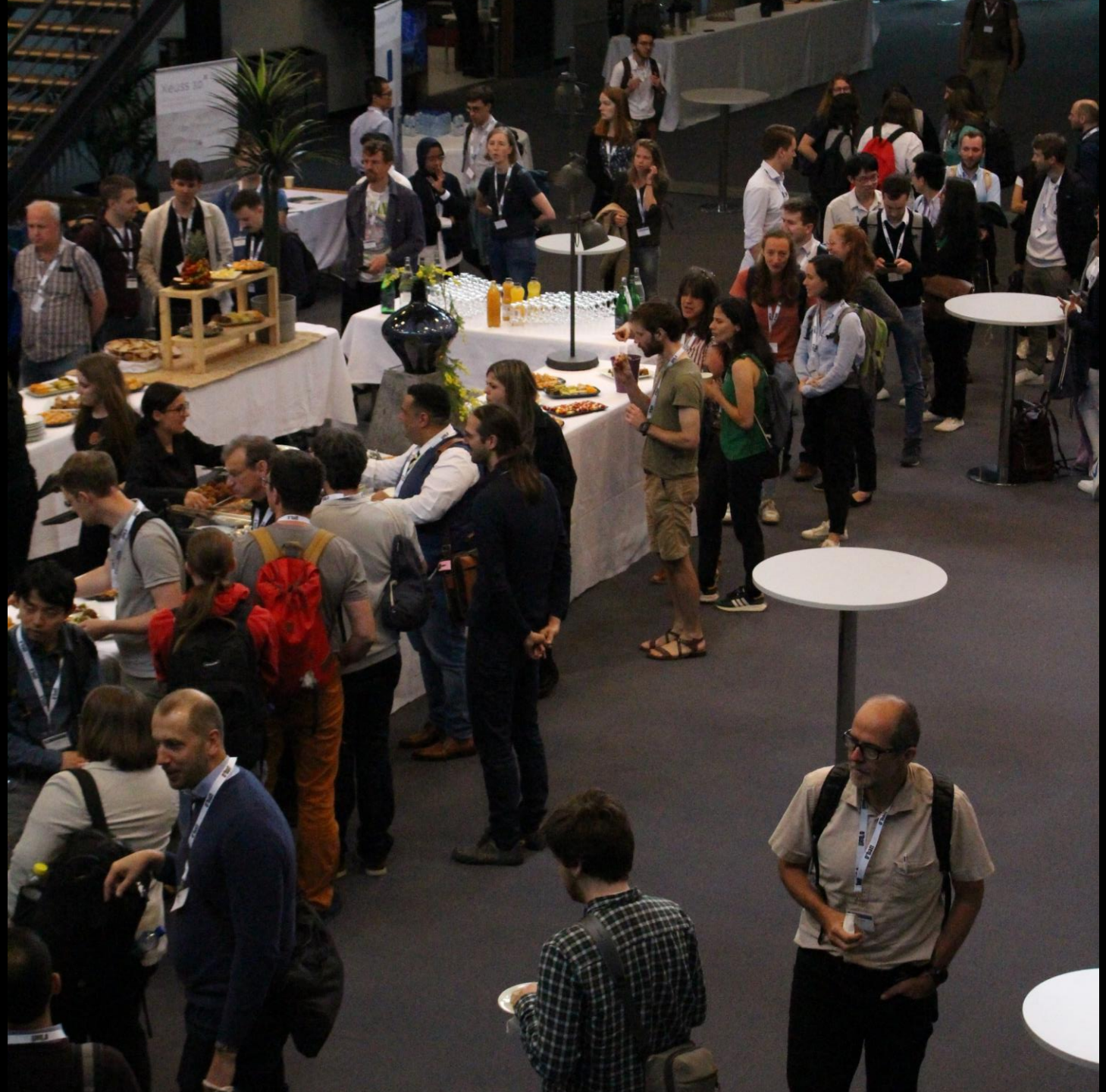
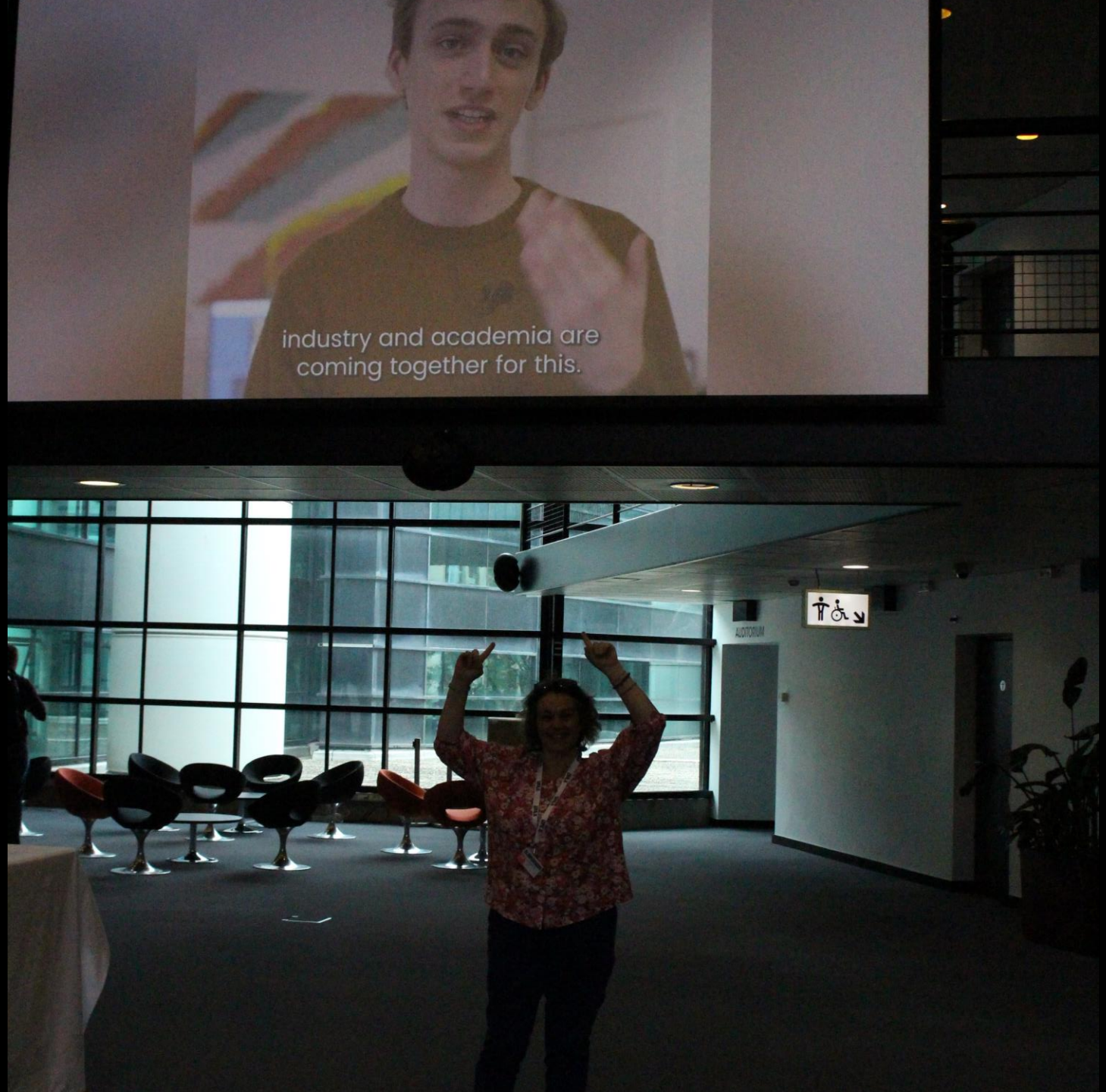


What is really cool about this
project is that both


industry and academia are
coming together for this.

that are sometimes also rather
creative

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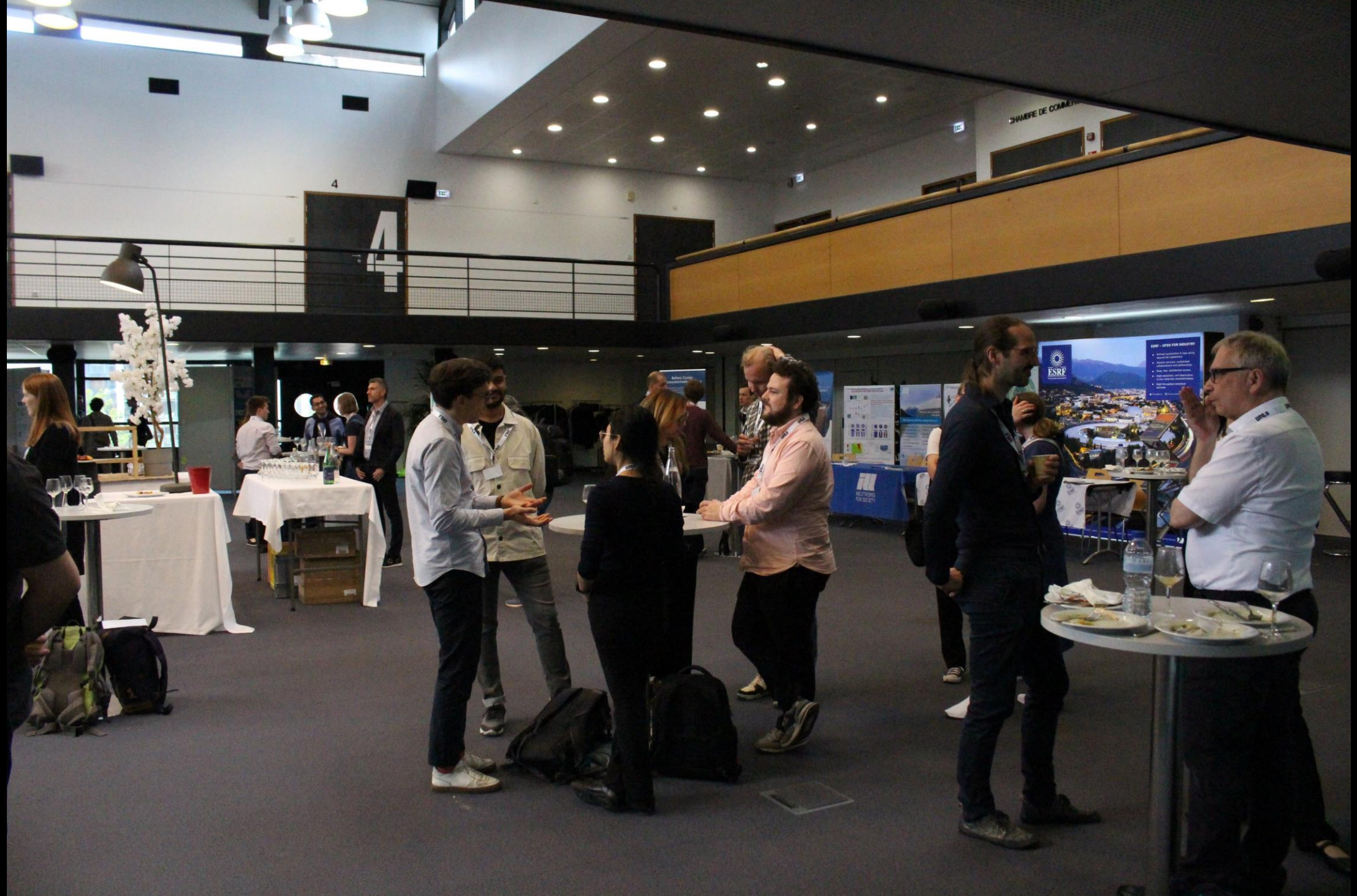
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Grenoble

World Trade Center
Séminaires
Expositions

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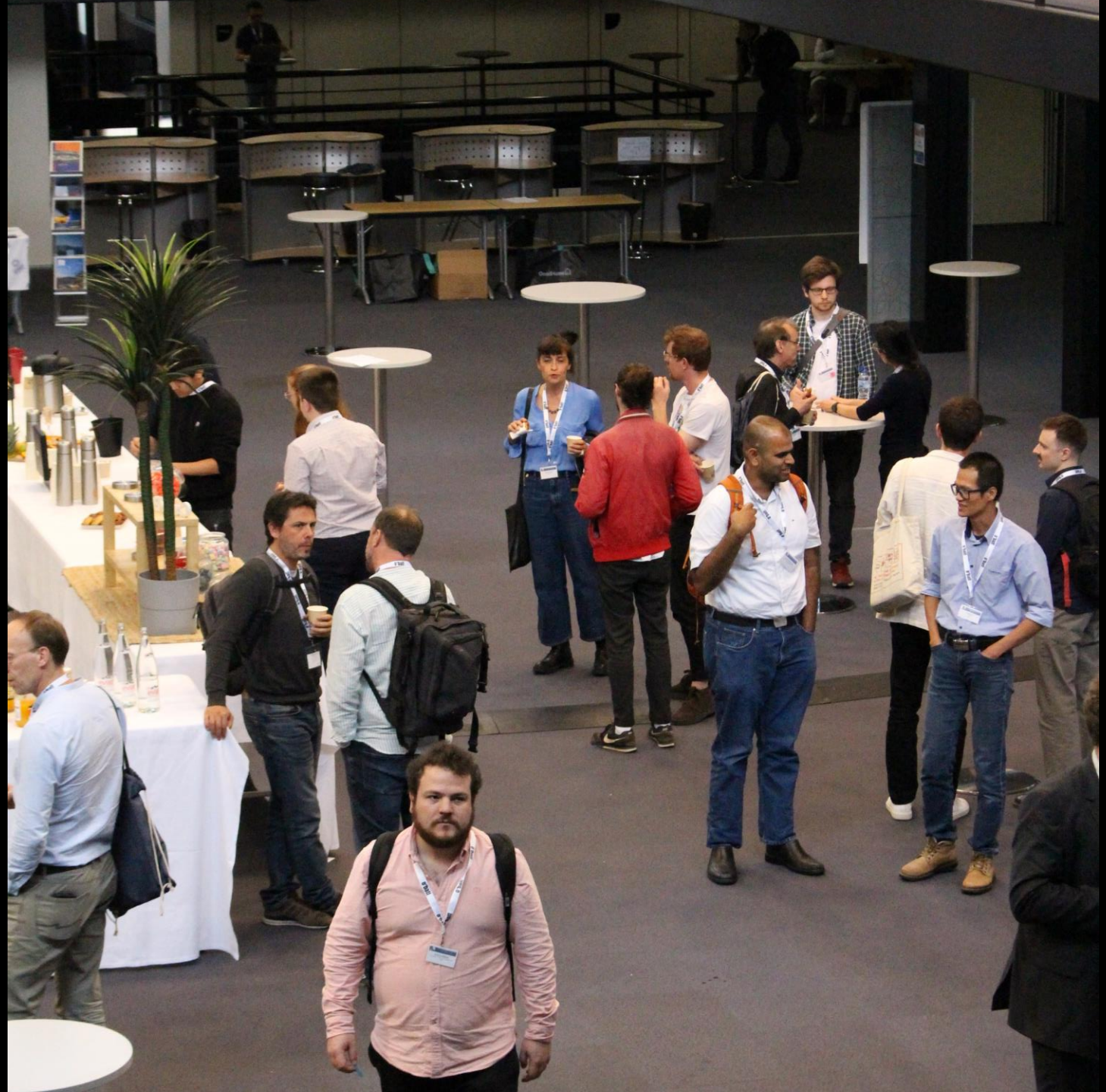
KILIMANDJARO

Xeuss 3.0
xenocs
Exploring the very small



KILIMANDJARO
MAKALU
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Xeuss 3.0*
Nano-inXider*
xenocs
exploring the very small

Xeuss 3.0

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SORTIE
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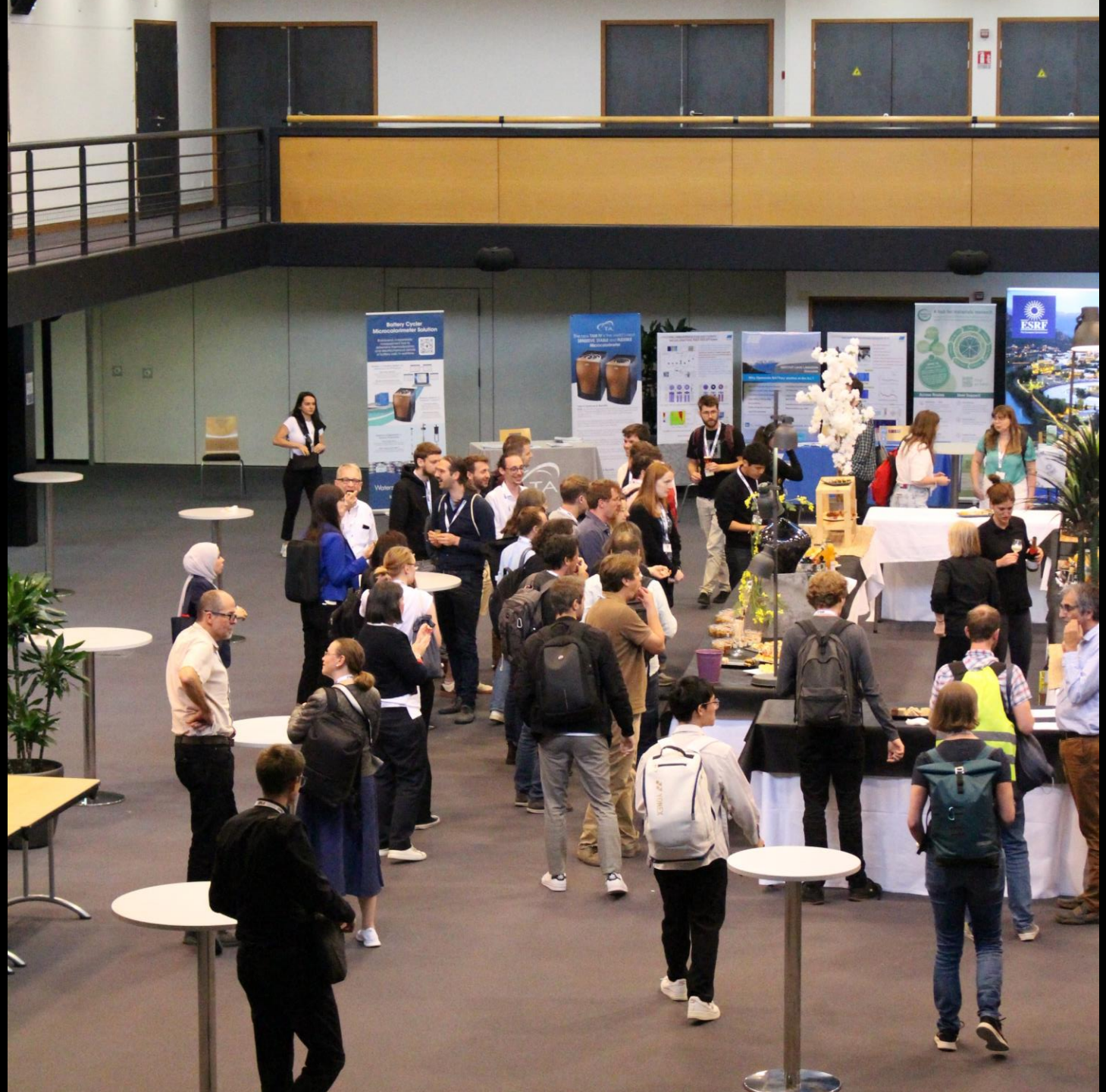
PARADOX

MAKALU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Microcalorimeter Solution
ESRF

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The Next Generation
IGISAXS/WAXS/USAXS beamline for the laboratory
XSACT analysis software
X-ray Scattering Analysis and Calculation Tool
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XSACT analysis software
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