

# *International Operando Battery Days*

Grenoble, France, 12-14 June 2024

Day Three

14.06.2024

Ninth Session

université  
PARIS-SACLAY

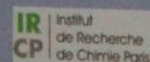
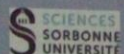


Institute for Materials  
and Molecular Chemistry



# Operando dielectric measurement of battery materials

*Sylvain Franger, J. Akinribido, L. Assaud, O. Dubrunfaut, J-C. Badot*



rhd in

UGA  
Université  
Grenoble Alpes

SyMMES

FOR SO

12-14th Ju



Operando Bottany De



12-14th June,



Battery Days  
12-14 June 2024

**nocs**  
Exploring the very small

12-14th June, G

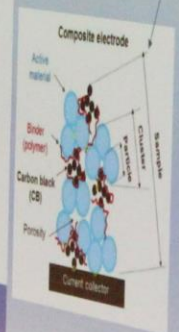


Battery Days  
12-14 June 2024

Visit our sponsors booths



12-14th June, Grenoble



# Objectives...

Understanding the electrical properties/limitations of electrodes : interfaces are the key



Existence of space charges at interfaces :

- Ionic double layer (electrolyte side)
- Depletion or accumulation layer (semiconductor side)

The electronic properties of active materials are rarely understood especially when filled with electrolytes



What kind of measures to achieve ?

- Slow kinetics → ion transfers → Low-frequencies
- Rapid kinetics → electron transfers → High-frequencies



Broadband Dielectric Spectroscopy (BDS)  
100 Hz – 10 GHz



12-14th June



























Physikalisch-Technische Bundesanstalt  
Braunschweig und Berlin

# Quantitative operando analysis of the polysulfide driven degradation in LiS batteries

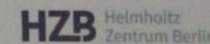
Katja Frenzel<sup>1</sup>, Adrian Jonas<sup>1</sup>, Lena Mathies<sup>1</sup>, Tamara Ruellas<sup>2</sup>, Marco Agostini<sup>2</sup>,

Sergio Brutti<sup>2</sup>, Philipp Hönicke<sup>1,3</sup>, Sebastian Risse<sup>3</sup>, Yves Kayser<sup>1,4</sup>,

and Burkhard Beckhoff<sup>1</sup>



- (1) Physikalisch-Technische Bundesanstalt, X-Ray Spectrometry, Abbestr. 2-12, 10587 Berlin, Germany
- (2) Sapienza Università Di Roma, Dipartimento Di Chimica, P.le A. Moro 5, 00185 Roma, Italy
- (3) Helmholtz-Zentrum Berlin, Chemical Energy Division, Hahn Meitner Platz 1, 14109 Berlin, Germany
- (4) Max-Planck-Institute for Chemical Energy Conversion, 45470 Mülheim an der Ruhr, Germany



rhd

UC  
Universit  
Grenoble

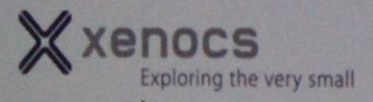
SyMI

12-14th June, Grenoble





Visit our sponsors booths



12-14th June, Grenoble

# 21GRD01 OpMetBat Operando metrology for energy storage materials

## CREATING EXCELLENCE FOR OPERANDO METROLOGY

Impedance Spectroscopy

Raman Spectroscopy

X-ray Spectrometry

X-ray Diffraction

J. Mater. Chem. A 9, 10231 (2021)

Traceable Characterisation Methods

Pilot Line Electrode Coater

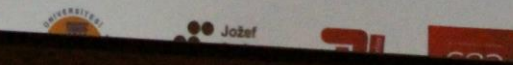
Electrode Preparation

Reproducible Battery Provision

Hybrid Operando Cell

Cell Design

Operando Cell

















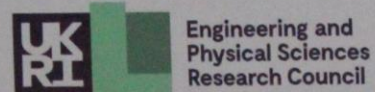
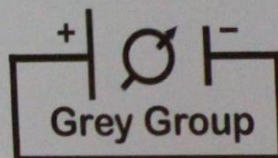
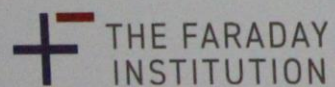






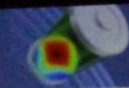
# Investigating Particle Cracking in Single-Crystal $\text{LiNiO}_2$ with Correlated Optical and Electron Microscopy

George Phillips – 14/6/24

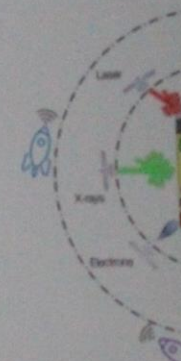


International Operando Battery Days

Grenoble, France, June 12-14<sup>th</sup> 2024



Internat



rhd | instrume  
flexible cell solu

JAC

UGA  
Université  
Grenoble Alpes



SyMMES

CNRS

12-14th J

12-14th June, 2024



Battery Days  
12-14 June 2024









NEUTRONS  
FOR SOCIETY

12-14th June, Grenoble

International Open Access Days  
Grenoble, France, 12-14 June 2017

LEAPS INNOVATION x xenocs  
Empowering the very small

ESRF  
EUROPEAN SYNCHROTRON FACILITY

rd Instruments  
Waters

JACOMEX























## International Operando Battery Days, 2024

### **Spatio-Temporal Analysis of Aging Effects on Lithiation Kinetics and Heterogeneities in Graphite Electrodes**

**Gozde ONEY<sup>1</sup>, Federico Monaco<sup>1</sup>, Saptarshee MITRA<sup>1</sup>, Asma MEDJAHED<sup>2</sup>, Manfred BURGHAMMER<sup>2</sup>, Quentin ARNOUX<sup>3</sup>, Samuel TARDIF<sup>4</sup>, Quentin JACQUET<sup>1</sup>, Sandrine LYONNARD<sup>1</sup>**

<sup>1</sup>Univ. Grenoble, Alpes, CEA, CNRS, IRIG, SyMMES, F-38000 Grenoble

<sup>2</sup>European Synchrotron Radiation Facility, F-38043 Grenoble Cedex, France

<sup>3</sup>TotalEnergies OneTech, 69360 Solaize, France

<sup>4</sup>Univ. Grenoble Alpes, CEA, IRIG, MEM, 38000 Grenoble, France

14/06/2024, Grenoble



Federico Monaco

rhd | instrumen  
flexible cell solutions

JAC

UGA  
Université  
Grenoble Alpes

SyMMES  
CNRS

12-14th Jun

12-14th June, Gren



12-14th June



Battery Days  
12-14 June 2024

**nocs**  
Exploring the very sma



Visit our sponsors booths

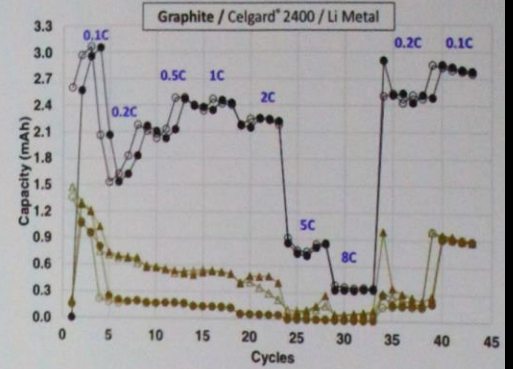


12-14th June, Grenoble

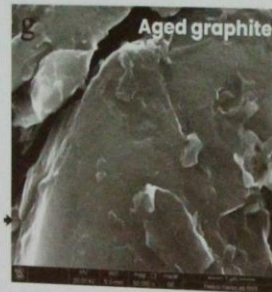
# Graphite aging

➤ Degradation of electrochemical performance with aging

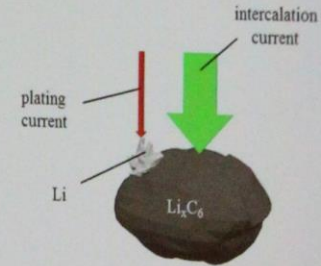
- 1 Loss of cyclable lithium  
**Growth of SEI layer**; temperature, low potential, high SOC  
**Li plating** at low temperatures and high charge rates



Matadi et al 2017 J. Electrochem. Soc. 164 A



Yang et al, 2014, RSC Adv., 4, 26335



C. Uhlmann et al., 2015, Journal of Power Sources, 279

















































sponsors booths

LEAPS  
INNOVATION

nanococs

Exploring the very small



ESRF  
European Synchrotron



NEUTRONS  
FOR SOCIETY

June, Grenoble



# Spatiotemporal tracking of electronic excitations in battery electrodes

Transient reflection imaging

M.-R. Amara<sup>1,4</sup>, A. J. Lovett<sup>2</sup>, T. S. Miller<sup>2</sup>, H. B. de Aguiar<sup>1</sup>, J. Utterback<sup>3</sup> and R. Pandya<sup>4</sup>





*International Operando Battery Days*  
Grenoble, France, 12-14 June 2024

LEAPS



Battery Days  
14 June 2024

DCS  
Powering the very small

Battery Days  
4 June 2024

PCS  
Powering the very small

S  
Y







International Operando Battery Days  
Grenoble, France, 12-14 June 2024

 **LEAPS**  
INNOVATION

 **xenocs**  
Exploring the very small

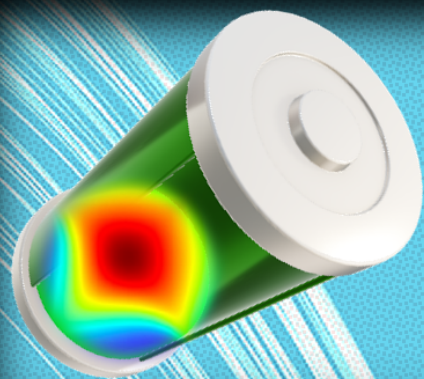
 **ESRF**  
The European Synchrotron

 **ILL**  
NEUTRONS  
FOR SOCIETY



 *International Operando Battery Days*  
Grenoble, France, 12-14 June 2024

 FAPS  XPS



# *International Operando Battery Days*

Grenoble, France, 12-14 June 2024

Day Three

14.06.2024

Tenth Session

o Battery Days



**VISTEC**  
VIDYASIRIMEDHI  
INSTITUTE OF SCIENCE AND TECHNOLOGY



Internat

booths

PS  
ION

CS  
ing the very small



**Dr. O. Fontaine**

Director Molecular Electrochemistry for Energy Laboratory, Thailand  
Junior member of Institut Universitaire de France

rhd instru  
flexible

UGA  
Université  
Grenoble Alpes

SyMMES

renoble

12-1

12-14th June, Gre



Days  
2024

ery small

12-14th June, Gre



ays  
24

y small





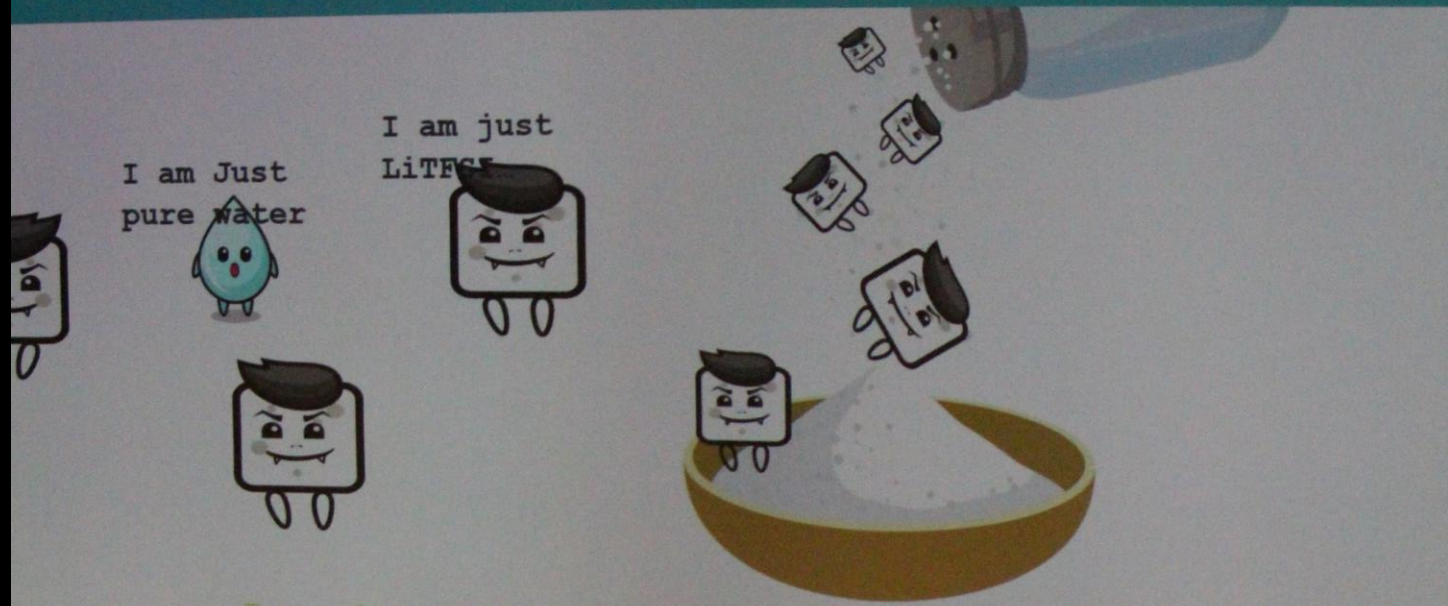








# Water-in-Salt



Rossukon Jommongkol, O. Fontaine  
O'batt- June 2024









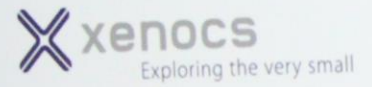




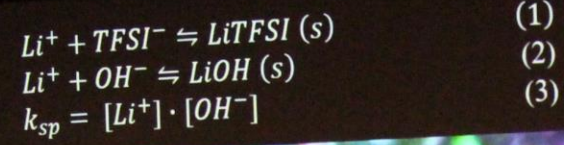




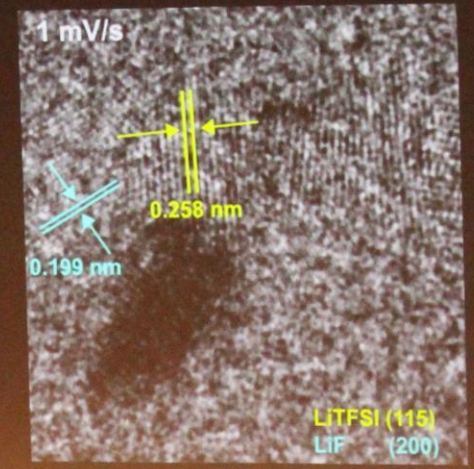
Visit our sponsors booths



12-14th June, Grenoble



LiTFSI commercial



R. Jommongkol, O. Fontaine et .al., Small. 2023









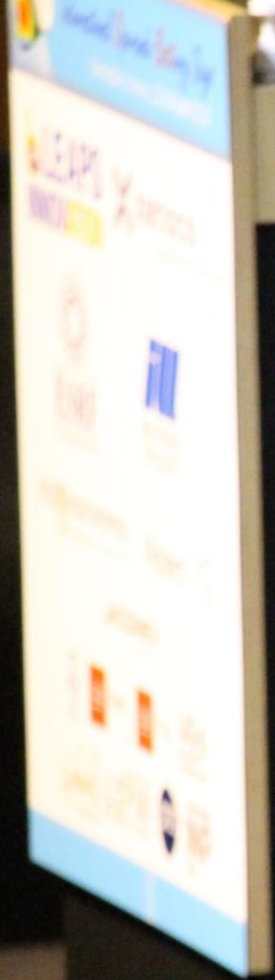
















# Investigation of 3D structure evolution in lithium-sulfur batteries with computed X-ray tomography

Antoine Klein, S. de Angelis, M. Sadd, S. Checchia, M. Olsson, S. Xiong, and A. Matic

ando Battery Days

rs booths

APS  
TION

ocs  
oring the very small

RF  
ctivation

NS  
ETY

Grenoble



Intern



rhd inst  
Research

UGA  
Université  
Grenoble Alpes

SyMMES

*12-14th June, Grenoble*



*12-14th June, Grenoble*







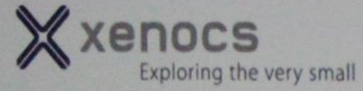


*12-14th June, Grenoble*



# Energy density

Visit our sponsors booths



The European Synchrotron

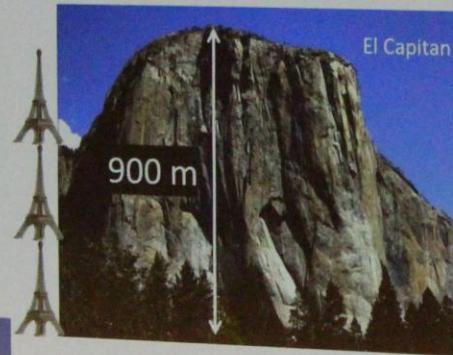


NEUTRONS FOR SOCIETY

12-14th June, Grenoble



Tesla Model S



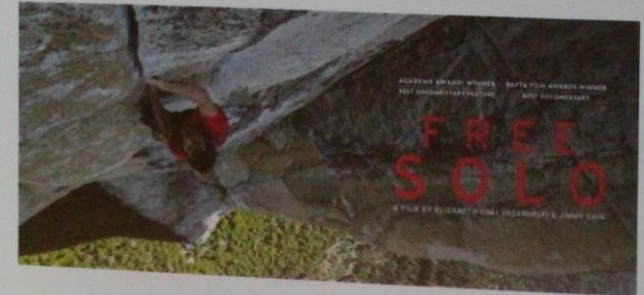
El Capitan

900 m



200 Wh/kg battery pack

Climb "El Capitan" 600 times



















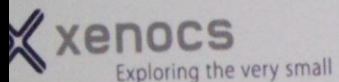








our sponsors booths



# ULTRATHIN-FILM ELECTRODE APPROACH FOR PROBING ANODE-FREE ALL-SOLID STATE BATTERIES AT OPERATING CONDITIONS BY X-RAY SPECTROSCOPIES

## Interface Design Department

E. Kataev, B. Aktekin, Z. Chalkley, P. Feng, L. Riegger, R. Garcia-Diez, R. Felix-Duarte, R.G. Wilks, Y. Xu, A. Henß, Y. Lu, J. Janek and M. Bär









*Grenoble*







noble







FOR SOCIETY  
Ann Mazzi, Dr. Nazia  
12:30 AM, 09/06/2016

FOR SOCIETY





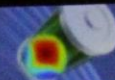








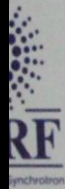




Booths

APS  
ATION

MOCS  
Exploring the very small



ONS  
CIETY

e, Grenoble

**Study of ion transport in Li-ion batteries electrodes by operando spectroscopic ellipsometry**

Alex Morata, Beatrice Laurenti, Juan Carlos Gonzalez-Rosillo, Fernanda Monteiro, Francesco Chiabrera, Marc Nuñez, Valerie Siller, Albert Tarancón

rhd ins

UGA  
Université  
Grenoble Alpes

SyMMES

12-14th June, Grenoble













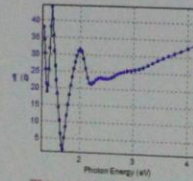
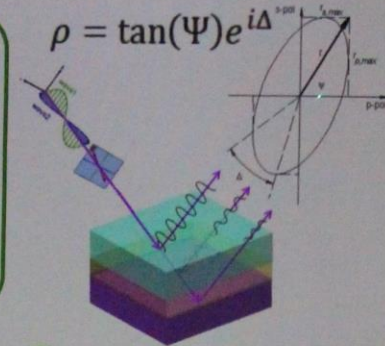
# Introduction - Spectroscopic Ellipsometry and TERS

Visit our sponsors booths



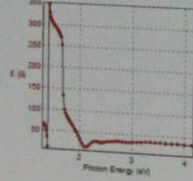
12-14th June, Grenoble

Spectroscopic ellipsometry is a highly accurate **non-destructive** technique that uses polarized light to provide the **thickness and the optical properties** of a thin film. Ideal technique for studying thin-film materials and systems



**Optical properties**

- Composition
- Crystallinity



**Multi-layers**

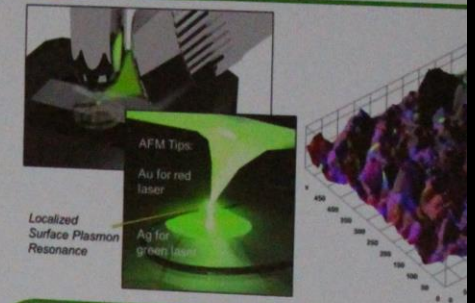
- Depth profiles
- Interfaces
- Thickness

**MATERIALS PROPERTIES**

**DEVICE PERFORMANCE**

J. Mater. Chem. A, 2014, 2, 20552

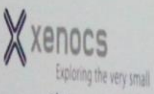
**TERS** combines the sensitivity of **Raman spectroscopy** with high spatial resolution of **scanning probe microscopy (SPM)** and enables chemical imaging of surfaces at nanometre length-scale.



**Confinement of the EM-field in the tip apex (SPR + lightning rod effect)**

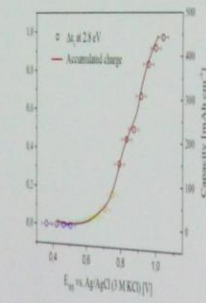
- Enhancement up to  $10^7$
- Detection sensitivity down to the molecule level
- Spatial resolution at several nanometres even on a sub-nanometer scale

Visit our sponsors booths



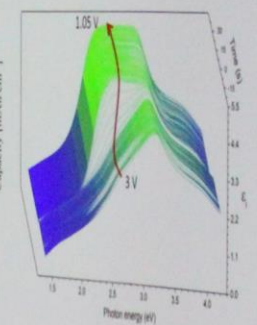
12-14th June, Grenoble

### Tracking Li content



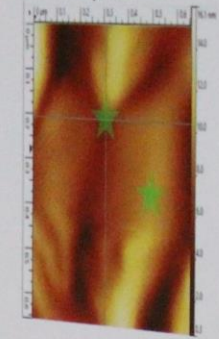
- SE provides a measurement of the **state-of-charge** of a cathode.
- **Volume changes** can be tracked

### Determination of diffusion coefficients



- The **diffusion coefficient** of the material can be obtained by measuring the evolution during a transient step and using **FEM simulation**.

### Preferential diffusion paths



- **TERS** is a promising technique to study phenomena **otherwise prohibited with conventional Raman**
- **First operando measurements** already showing interesting phenomena at the **nanoscale**



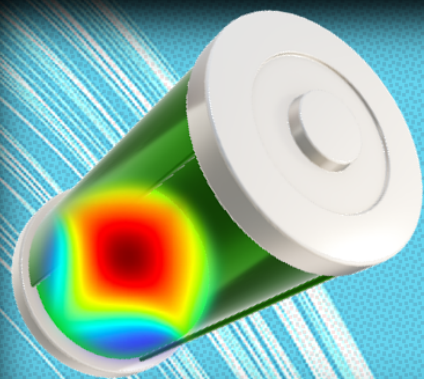
12-14th June











# *International Operando Battery Days*

Grenoble, France, 12-14 June 2024

Day Three

14.06.2024

Prize and Ending



  
NEUTRONS  
FOR SOCIETY

12-14th June, Gre



 *International Operando Battery Days*  
Grenoble, France, 12-14 June 2024

 **LEAPS**  
INNOVATION

 **xenocs**  
Exploring the very small

 **ESRF**  
The European Synchrotron

 **NEUTRONS**  
FOR SOCIETY

**xenocs**  
Exploring the very small



Our sponsor

12-14th June, Grenoble

rhd



International Open-Access Battery Days



Exploring the very small

# Our sponsors



12-14th June, Grenoble



International Operando Battery Days  
Grenoble, France, 12-14 June 2024

LEAPS INNOVATION X xenocs

ESRF NEUTRONS FOR SOCIETY

rhd instruments Waters

JACOMEX

UGA

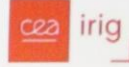
SyMMES LePMI GTS INP



# Our sponsors



12-14th June, Grenoble



enoble



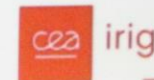
GLEADS  
BEST ORAL AWARD  
International Journal of History of Art







12-14th June, Grenoble



International Operando Battery Days  
Grenoble, France, 12-14 June 2024







Exploring

Our sponsors

12-14th June, Grenoble




International Opened Beta

enoble





  
NEUTRONS  
FOR SOCIETY

12-14th June, Grenoble

  
LEPMI  
Grenoble - Chambéry

  
SyMMES  
SYNTHÈSE, MÉTHODES, MODÈLES  
SYNTHÈSE, MÉTHODES, MODÈLES

  
cea

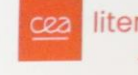
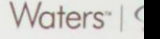
  
flexible cell solution











14th June, Grenoble



International Opened Battery Days  
October 1-3, 2018

LEAPS INNOVATION x xenocs  
Exploring the very small

ESRF ILL  
NEUTRONS FOR SOCIETY

rhd instruments Waters | CEA

JACOMEX

cea liten cea irig UGA

SyMMES LePMI CNRS INP

FOR SOCIETY

12-14th June, Cambridge



Operando Battery Days

12-14th Grenoble



Days  
e 2024  
S  
e very small





12-14th June, Grenoble





12-14th June, Grenoble



International Operando Battery Days  
Grenoble, France, 12-14 June 2018









 *International Operando Battery Days*  
Grenoble, France, 12-14 June 2024

 **LEAPS**  
INNOVATION

 **xenocs**  
Exploring the very small



12-14th June, 2024



 *International Operando Battery Days*  
Grenoble, France, 12-14 June 2024

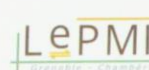
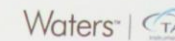
 **LEAPS**  
INNOVATION

 **xenocs**  
Exploring the very small

**xenocs**  
Exploring the very small



# Our sponsors



12-14th June, Grenoble



**xenocs**  
Exploring the very small

**ESRF**  
The European Synchrotron

**ILL**  
NEUTRONS  
FOR SOCIETY

12-14th June, Grenoble

# Our sponsors

**LEAPS  
INNOVATION**

**xenocs**  
Exploring the very small

**ESRF**  
The European Synchrotron

rhd **instruments**  
flexible cell solutions

**JACOMEX**  
pure safety

Waters | **TA**

**LEPMI**  
Grenoble - University

**SyMMES**  
Synchrotron-Materials-Mechanics-Engineering

**cea** **irig**

**cea** **liten**

**CNRS**

**UGA**  
Université  
Grenoble Alpes

**UN  
SAV  
MO**

International Operands Battery Days  
2014

**LEAPS  
INNOVATION** **xenocs**  
Exploring the very small

**ESRF**  
The European Synchrotron

**ILL**  
NEUTRONS  
FOR SOCIETY

rhd **instruments** Waters | **TA**

**JACOMEX**

**cea** **irig** **cea** **liten** **CNRS** **UGA**  
Université Grenoble Alpes

**SyMMES** **LEPMI** **CNRS** **INP**







**PACK**  
THE ORIGINAL  
NOVA 100%  
NOVA 100%  
NOVA 100%  
NOVA 100%

**PACK**  
THE ORIGINAL  
NOVA 100%  
NOVA 100%  
NOVA 100%  
NOVA 100%

the

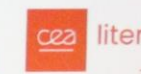
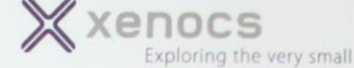
o'hair



*International Operando Battery Days*  
Grenoble, France, 12-14 June 2024



12-14th June, Grenoble



International Open-air Battery Days





*International Operando Battery Days*





INNOVATION

xenocs  
Exploring the very small



ESRF  
The European Synchrotron



NEUTRONS  
FOR SOCIETY

Our sponsors

rhd instruments  
flexible cell solutions

LePMI  
Grenoble - Chambéry

SyMMES  
SYNCHROTRON YVES COENNEVILLE  
MUSEUM OF SCIENCE AND TECHNOLOGY

cea

12-14 June, Grenoble



International Open-Access Battery Days  
Grenoble, France, 12-14 June 2024

LEAPS INNOVATION x xenocs  
Exploring the very small

ESRF  
The European Synchrotron

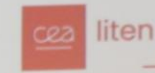
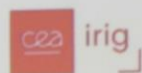
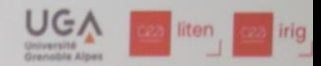
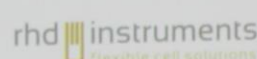
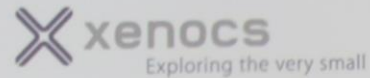
rhd instruments Waters | cea

JACOMEX

cea ifren cea ifrig UGA

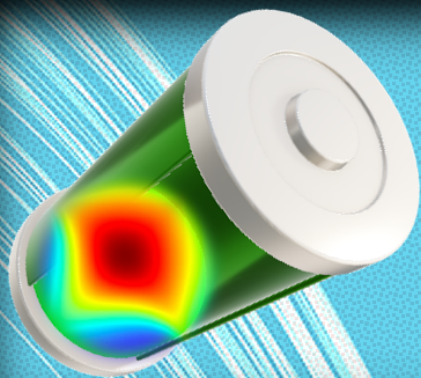
SyMMES LePMI CTS INP

# Our sponsors



12-14th June, Grenoble





# *International Operando Battery Days*

Grenoble, France, 12-14 June 2024

Day Three

14.06.2024

On the Sides



NEUTRONS  
FOR SOCIETY

12-14th June



*International Operando Battery Days*  
Grenoble, France, 12-14 June 2024

**LEAPS INNOVATION** **xenocs**  
Exploring the very small

**ESRF**  
The European Synchrotron

**ILL**  
NEUTRONS FOR SOCIETY

**rhd instruments**  
Flexible cell solutions

**Waters**

**JACOMEX**

  
NEUTRONS  
FOR SOCIETY

12-14th June, Gre



International Operando Battery Days  
Grenoble, France, 12-14 June 2024

PS  
TION

xenocs  
Exploring the very small

  
NEUTRONS  
FOR SOCIETY

struments  
Waters®

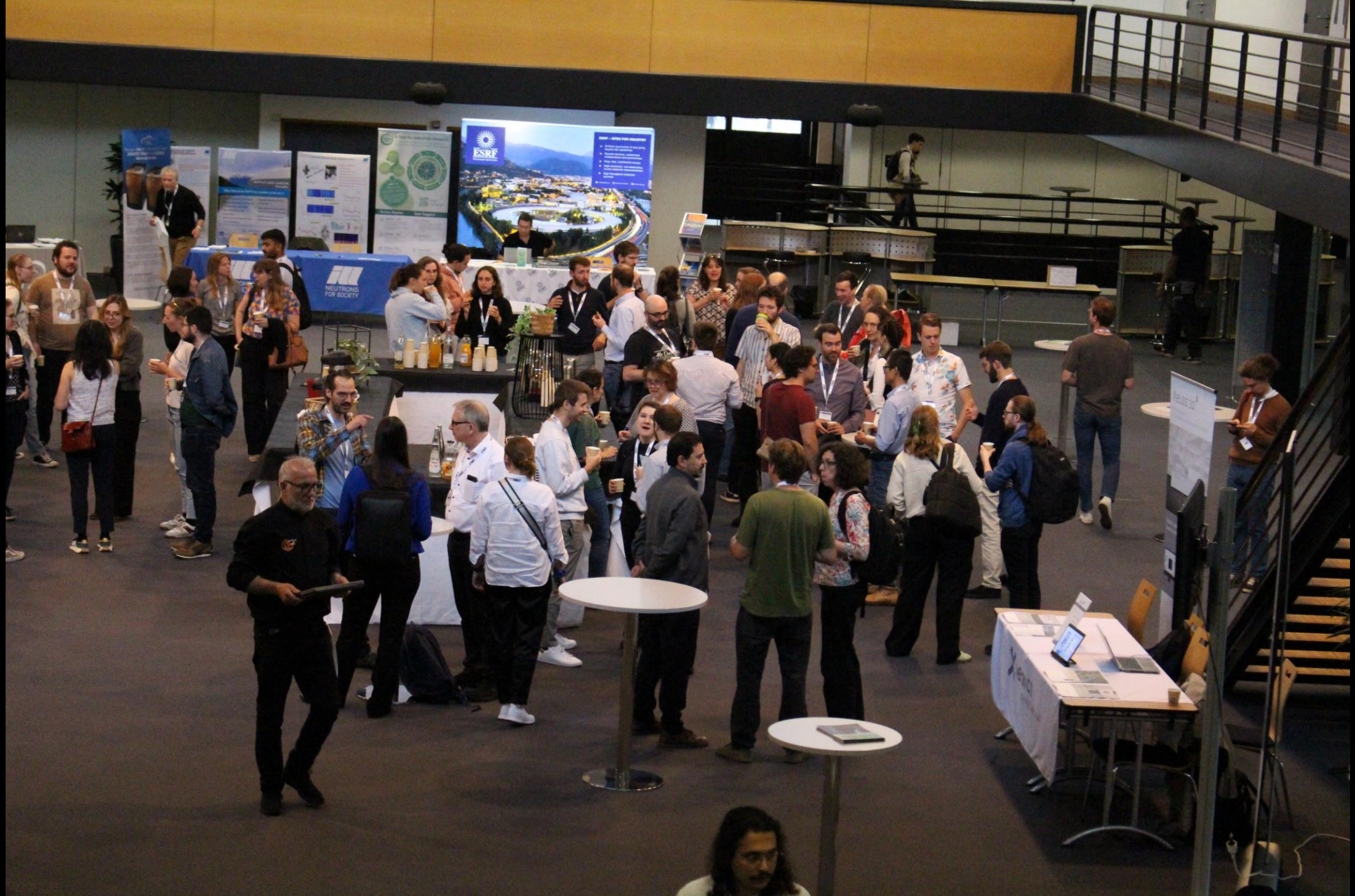
JACOMEX

liten

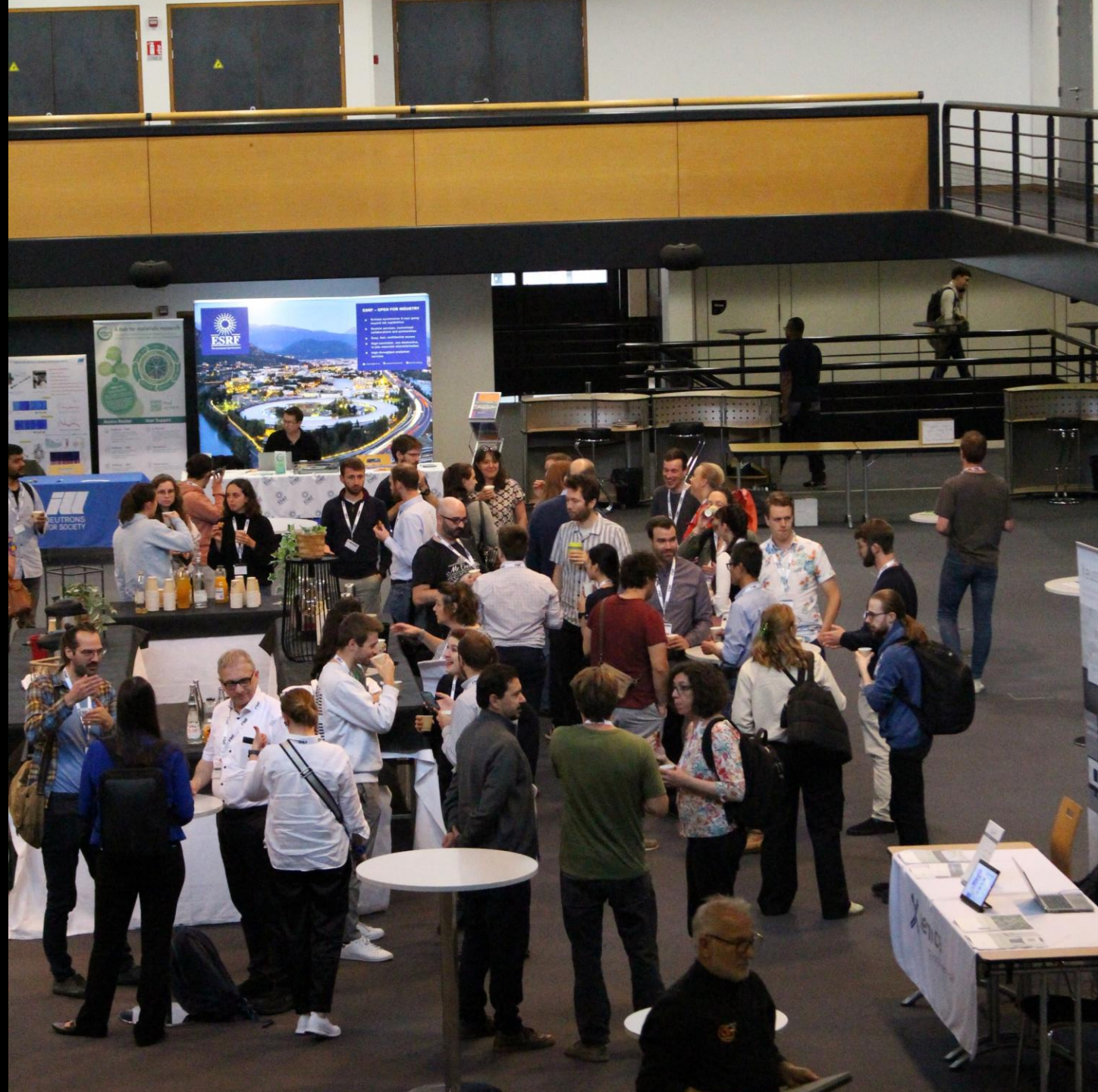
CHAMBRE DE COMMERCE & D'INDUSTRIE DE GRENOBLE

Xenocs  
ESRF  
Program  
Everything happens  
in the WTC  
Best talk and best  
poster prizes for  
young scientists



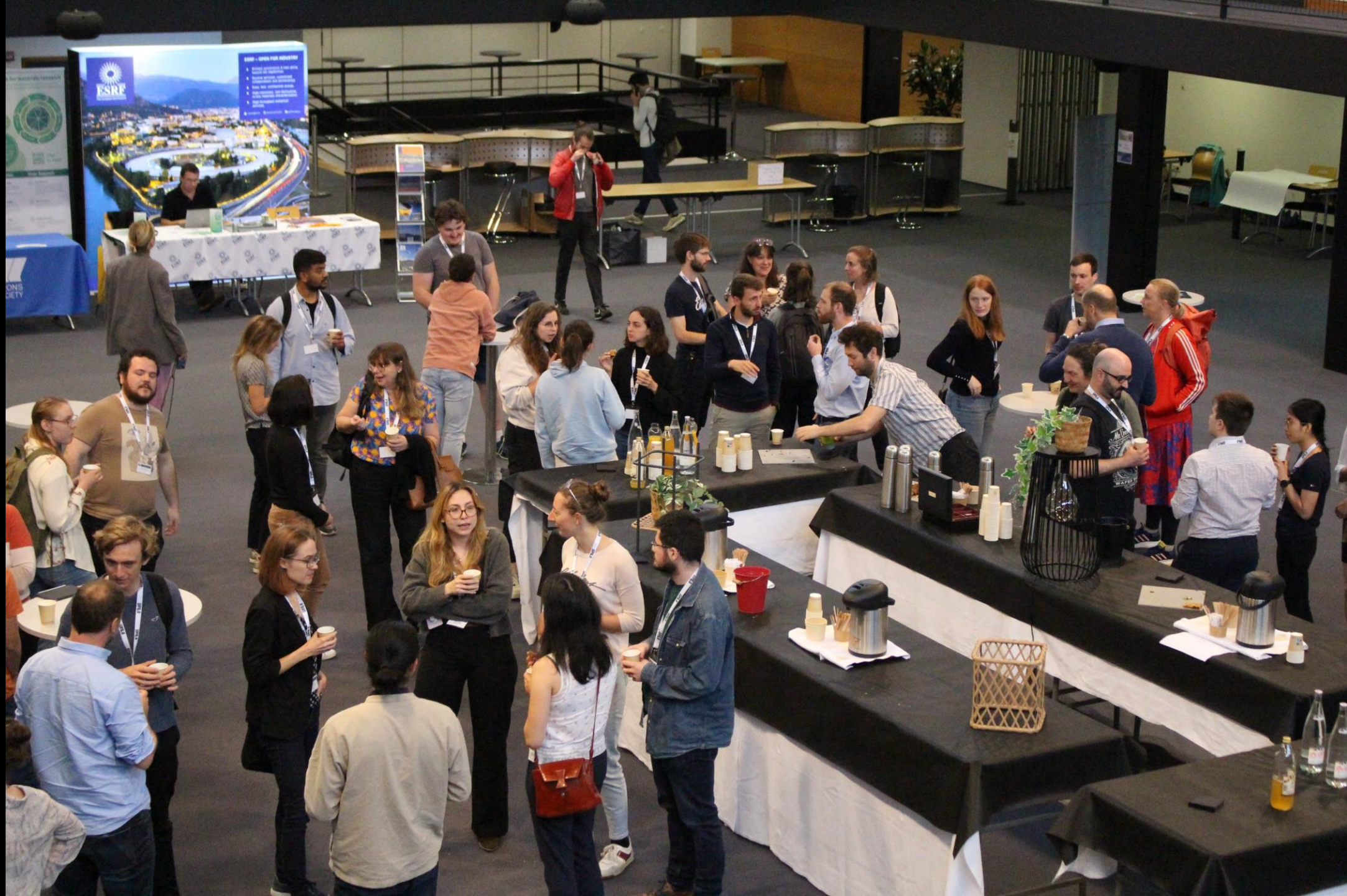


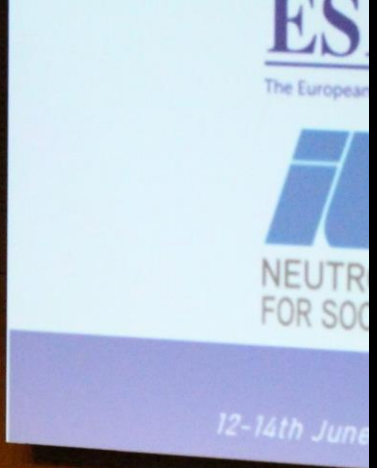














International Operands Battery Days  
Grenoble, France, 12-14 June 2014

**APS ACTION** **xenocs**  
Combining the very best

**ESRF** **ILL**  
NEUTRONS FOR SOCIETY

rhd instruments Waters | TA

**JACOMEX**

**cea** **irig** **UGA**  
**SyMMES** **LePMI** **cnrs** **INP**





APS  
INNOVATION

RECOGNIZES

*Corentin Tournis*

**BEST ORAL AWARD**

*International Operando Battery Days*  
Lyon, France, 12-14 June 2018





rhd instruments  
flexible cell solutions

JACOMEX  
pure safety

Waters | T

LePMI  
Grenoble - Chambéry

SyMMES

cea irig

cea liten

cnrs

UGA  
Université  
Grenoble Alpes



rhd instruments  
flexible cell solutions

JACOMEX  
pure safety

Waters | TA  
INSTRUMENTS

LePMI  
Grenoble - Chambéry

SyMMES  
SYNTHÈSE, MÉTHODES  
MOLECULAIRES

cea irig

cea liten

CNRS

UGA  
Université  
Grenoble Alpes

UNIVERSITÉ  
SAVOIE  
MONT BLANC

GRENOBLE  
INP  
UGA







# Our sponsors





