Synchrotron Radiation Facilities and Industry

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Today, in an world ever more competitive scientifically and technologically, deep knowledge of materials properties is fundamental.

75 Years of Science with Synchrotron Light

Some synchrotron facilities in world

African Light Source Project (Honorary Member)

ALBA

ALS ADVANCED LIGHT SOURCE

Argonne NATIONAL LABORATORY
APS at Argonne National Laboratory

Australian Synchrotron

HZB BESSY II Light Source
BESSY II at HZB

Canadian Light Source | Centre canadien de rayonnement synchrotron

CHESS CORNELL HIGH ENERGY SYNCHROTRON SOURCE

diamond

Elettra Sincrotrone Trieste

ESRF The European Synchrotron

LNLS

MAX IV

Brookhaven National Laboratory
National Synchrotron Light Source II

NSRRC

PAL POHANG ACCELERATOR LABORATORY
Pohang Light Source-II

DESY.
PETRA III at DESY

Photon Factory
Institute of Materials Structure Science
High Energy Accelerator Research Organization,KEK

HZDR HELMHOLTZ ZENTRUM DRESDEN ROSSENDORF
ROBL Beamline at ESRF (HZDR)

SESAME
(Honorary Member)

SOLARIS NATIONAL SYNCHROTRON RADIATION CENTRE

SLAC NATIONAL ACCELERATOR LABORATORY
SSRL at SLAC

PAUL SCHERRER INSTITUT PSI
Swiss Light Source at PSI

60 Synchrotron and Free Electron Laser facilities around the world

Free Electron Lasers

European XFEL

HZDR HELMHOLTZ ZENTRUM DRESDEN ROSSENDORF
FELBE / TELBE at HZDR

SLAC NATIONAL ACCELERATOR LABORATORY
LCLS at SLAC

PAL POHANG ACCELERATOR LABORATORY
PAL-XFEL

Elettra Sincrotrone Trieste
FERMI at ELETTRA

DESY.
Flash at DESY

PAUL SCHERRER INSTITUT PSI
Swiss FEL at PSI

Construction of Synchrotron Lines

DOUBLE CRYSTAL MONOCHROMATOR (DCM)

MIRROR SYSTEMS

BEAMLINE COMPONENTS

DETECTORS AND DIAGNOSTICS

MOTION CONTROLS

ARPES Beamline at SOLARIS Constructed by FMB Company

Beamline engineering excellence

Complete beamlines & custom engineered systems

- MONOCHROMATORS
- KB MIRROR SYSTEMS
- HIGH HEAT LOAD APERTURES
- BEAMLINE COMPONENTS
- DIAGNOSTICS
- EXPERIMENTAL EQUIPMENT
- OPTICAL ENGINEERING
- COMPREHENSIVE TESTING
- INTEGRATED EPICS SOFTWARE

idt

instrument design technology

beamline engineering excellence

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PREVAC
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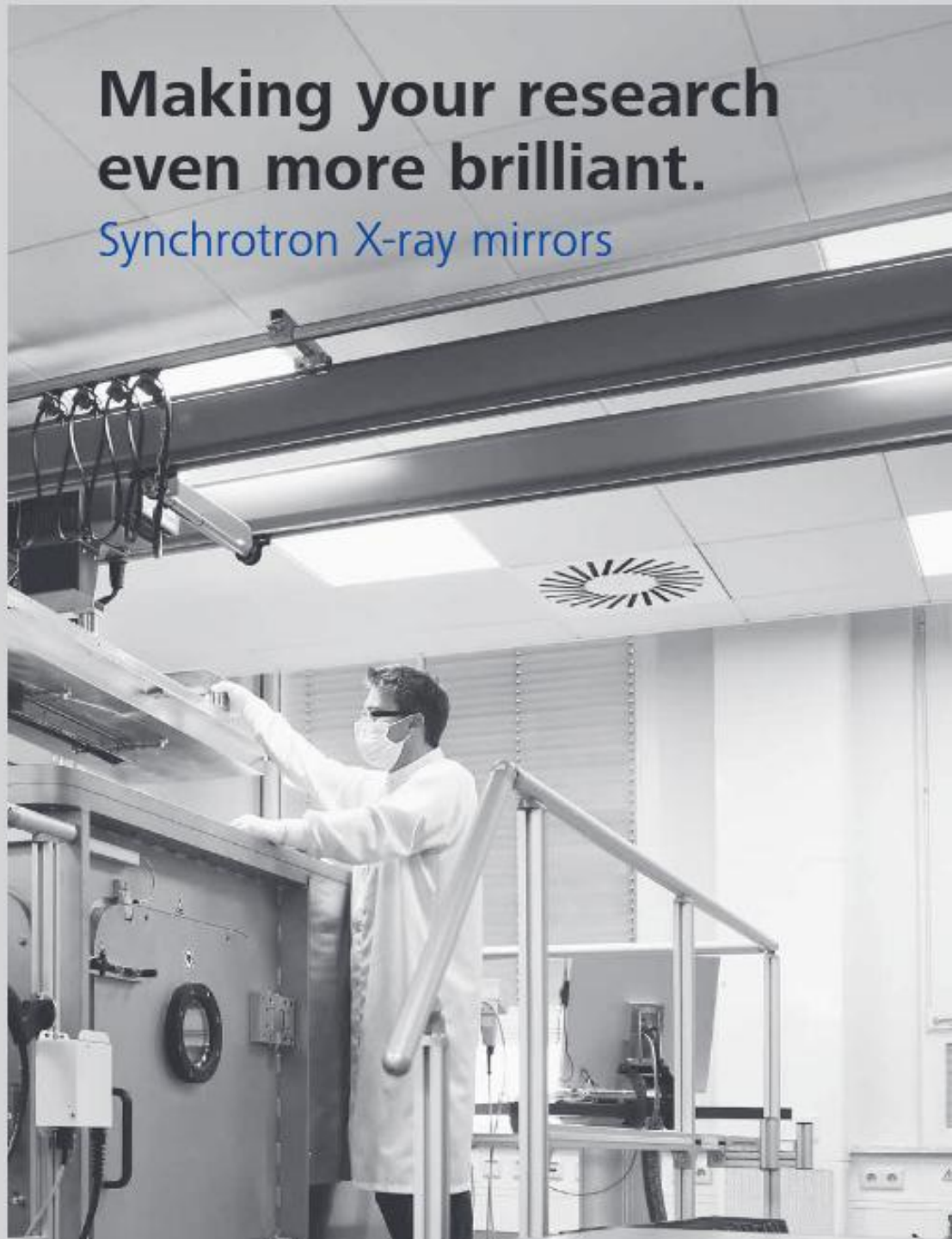
25 YEARS OF
INNOVATIVE
THINKING

ZEISS

Making your research even more brilliant.

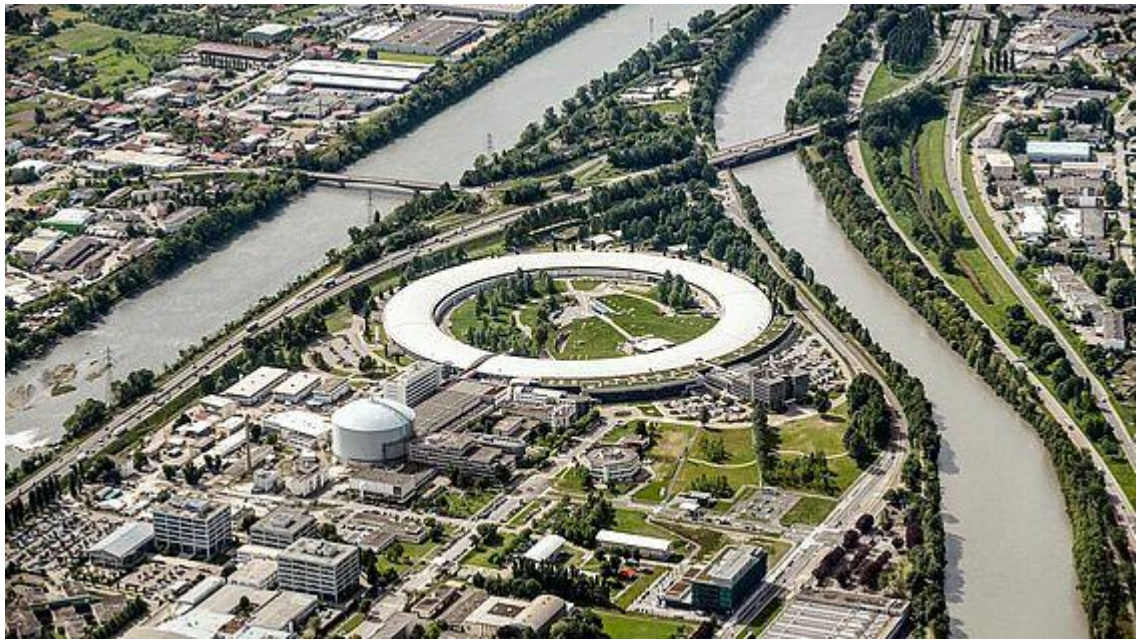
Synchrotron X-ray mirrors



Brilliant research thanks to ZEISS
Mirrors and gratings for synchrotrons

2022 Budget of European Synchrotron Radiation Facility (ESRF)

ESRF
The European Synchrotron



- 43 Beamlines
- 2022 Budget : 90 million Euro
- Number of Staff: 700
- 450 scientist, technicians, and engineer
- About 9000 researchers come each year to the ESRF to carry out experiments.
- 9000 proposal
- 2000 paper annually published
- 330 million Euro for its upgrade

ESRF Member States

France	27.5 %
Germany	25.5 %
Italy	15 %
United Kingdom	14 %
Spain	4 %
Switzerland	4 %
Benesync (Belgium, Netherlands)	6 %
Nordsync (Denmark, Finland, Norway, Sweden)	4 %

ESRF Associates

Portugal	1 %
Israel	1 %
Austria	1 %
Poland	0.6 %
CentralSync (CZ, H, SK)	1.05%

Exploiting the Innovation Potential of Synchrotron Based Researches for Industry

- R&D partnership with industry

To develop union capabilities and industrial supply in high-tech areas such as scientific instrumentation

- Stimulate the industry for using the synchrotron based experiments

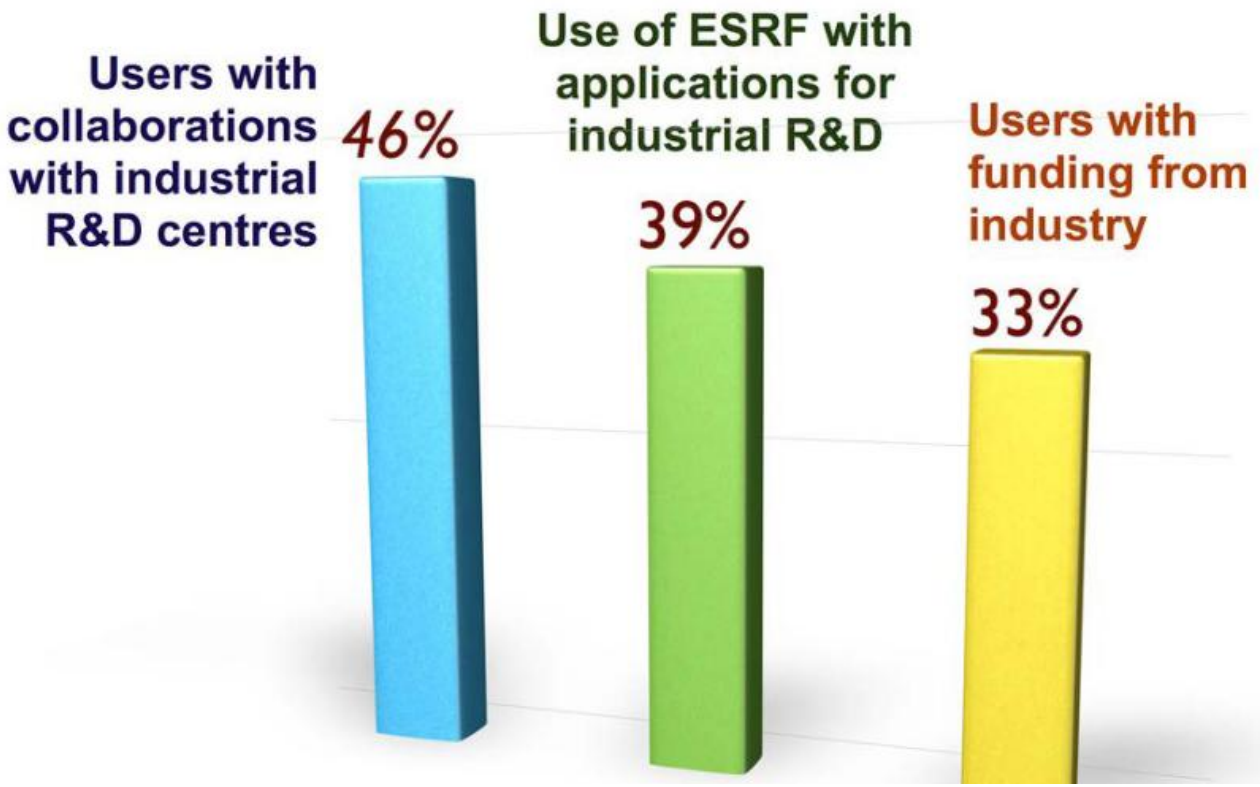
Experimental test facilities, innovation hubs, knowledge-based centers

- Encourage the integration of synchrotron research into local, regional and global innovation systems


Pfizer
MERCK
AstraZeneca
L'ORÉAL
PARIS
gsk
GlaxoSmithKline
ESRF
astex
JM
Johnson Matthey
Inspiring science, enhancing life
BAYER
DAIHATSU
TOYOTA


ESRF
THE EUROPEAN SYNCHROTRON

ESRF Collaboration with Industry



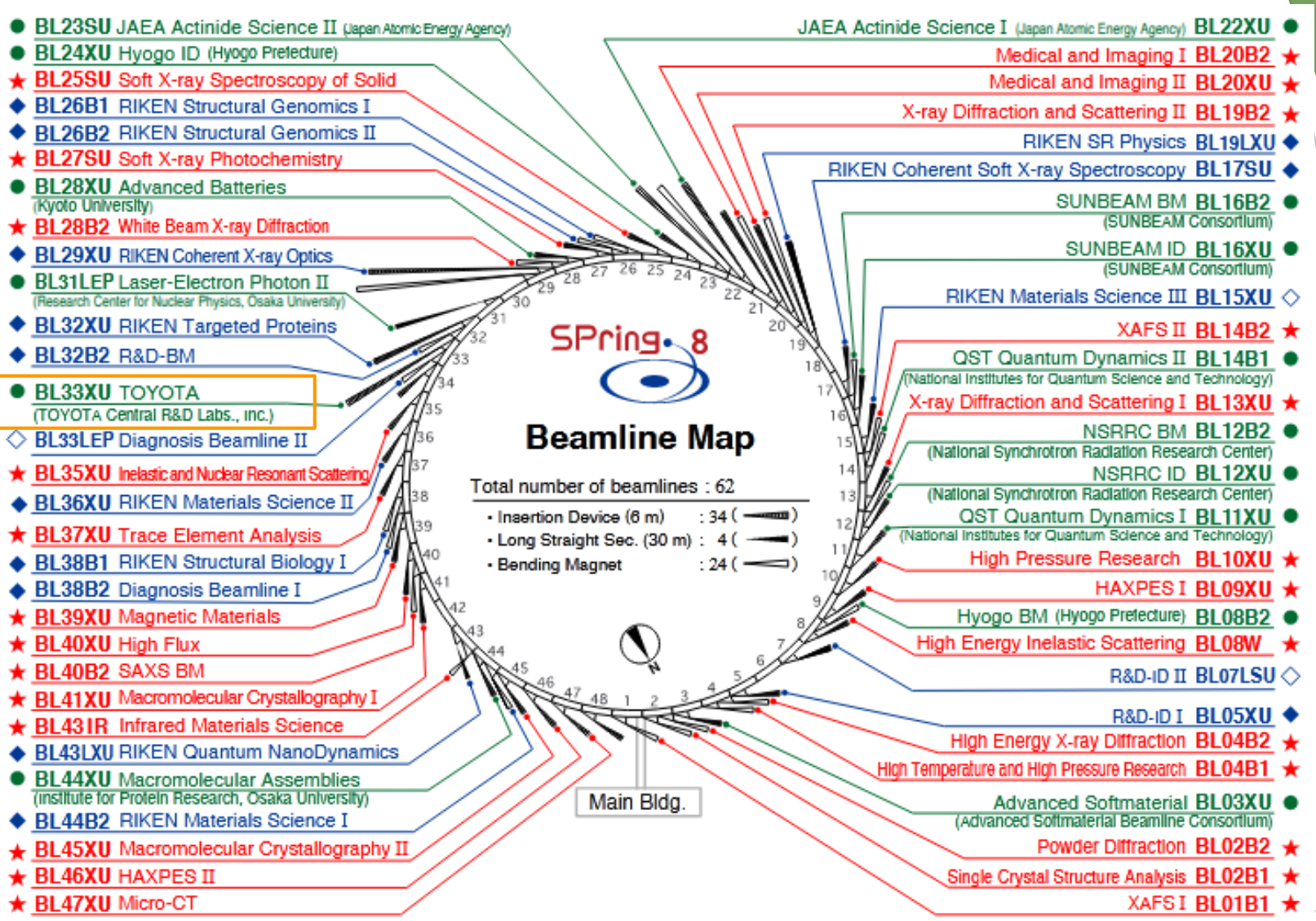
Generated >25MEuros

Used to fund staff positions, purchase new equipment, fund beamlines

- **2011: 2.2MEuros income generated**
- **Beam access by industry represents about 2% of beam time capacity; but about 10% of allocatable income**


X-ray Free
Electron Laser
Spring-8
Storage Ring
8 GeV

TOYOTA Beamline



BL: Beamline
B1, B2: Bending Magnets
XU: X-ray Undulator
SU: Soft X-ray Undulator
W: Wiggler

IR: Infrared Radiation
LEP: Laser-Electron Photon
LXU: Long-length X-ray Undulator
LSU: Long-length Soft X-ray Undulator

★ : Public Beamlines
● : Contract Beamlines
◆ : RIKEN Beamlines

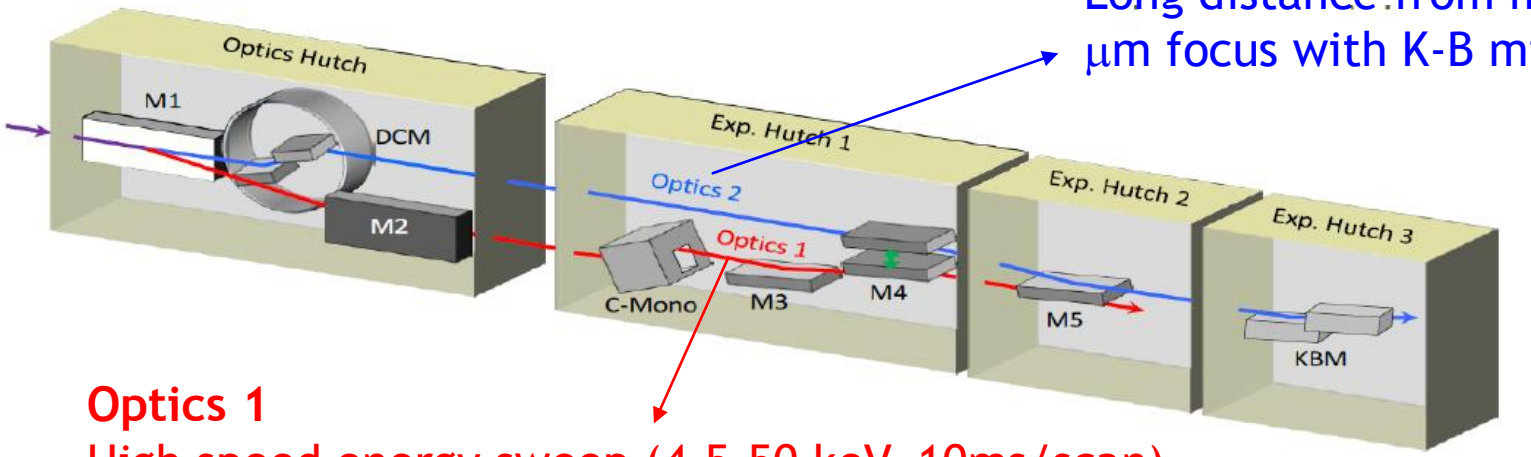
TOYOTA BEAMLINE (BL33XU)

Applications:

Analysis and evaluation of materials used in car industry(Battery, Fuel Cell, Catalyst)

- **High speed time-resolved Measurement**
  several tens of millisecond
- **Operando Measurement**
  under various kinds of atmospheres and temperatures

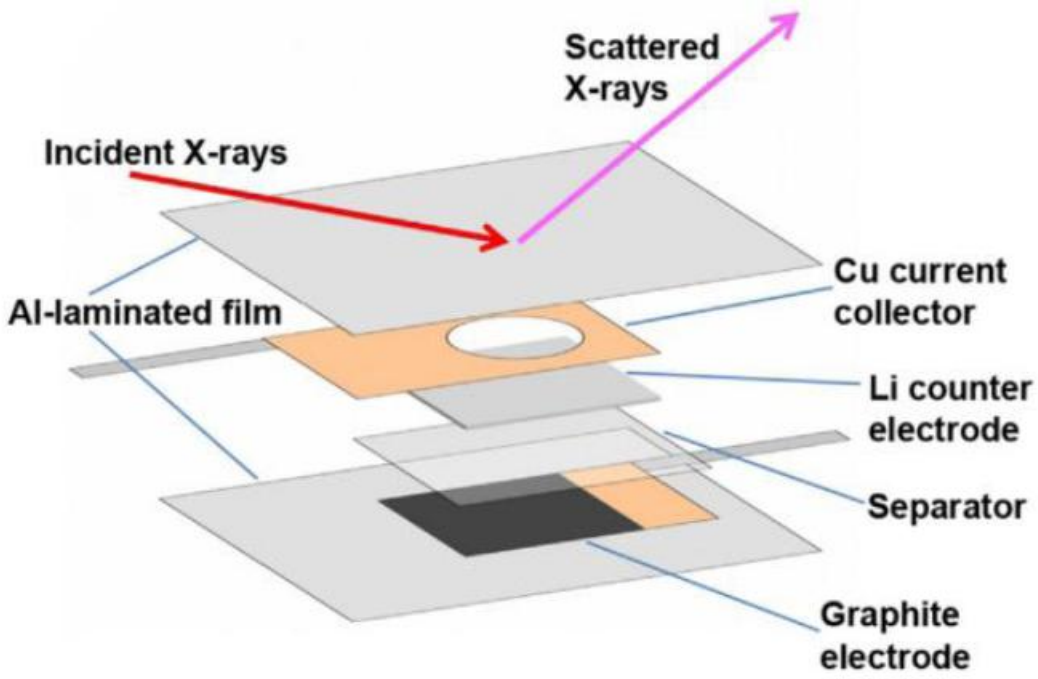
Optics 2
Double Crystal Monochromator (4.5-72 keV)
Long distance from monochromator to sample about 80 m
μm focus with K-B mirror

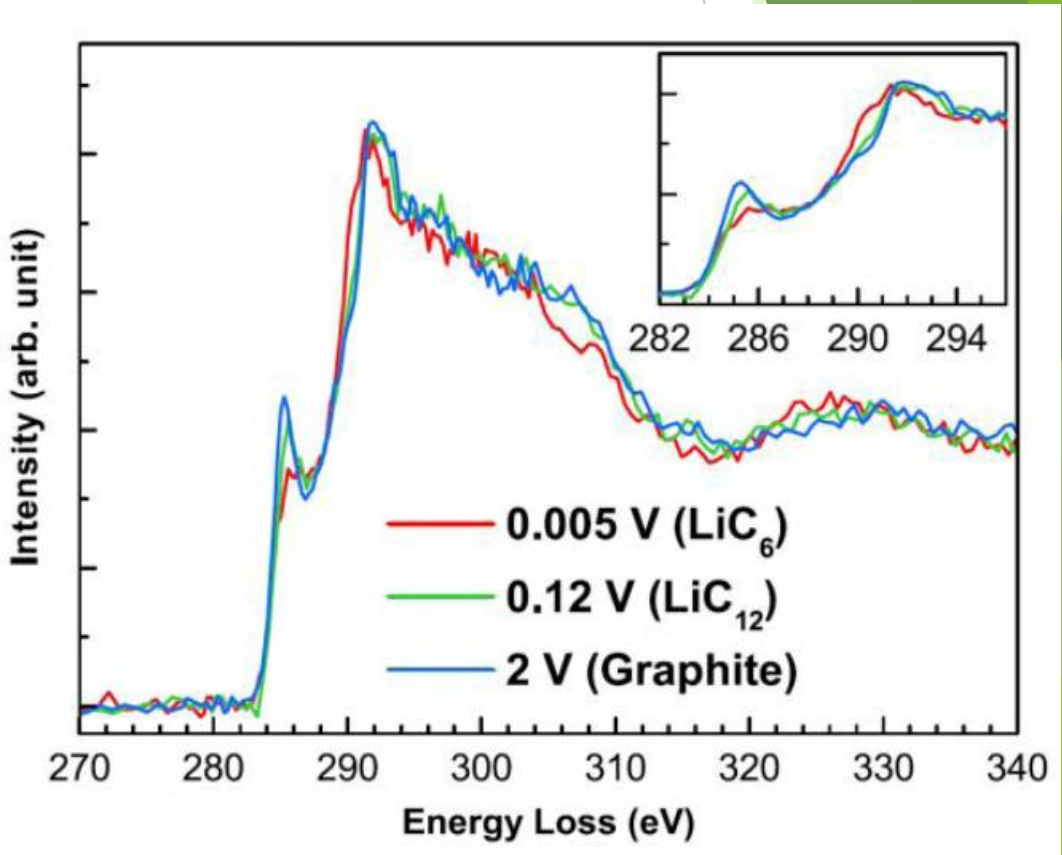


Optics 1
High speed energy sweep (4.5-50 keV, 10ms/scan)
Focus and expand beam with horizontal and vertical mirros

- High speed X-ray absorption fine structure (XAFS) (operando analysis)
- Three dimensional XRD
- SAXS
- X-ray computed tomography/laminography
- X-ray Raman Scattering

Charge and Discharge of Li-ion battery using in situ X-ray Raman Scattering at TOYOTA beamline

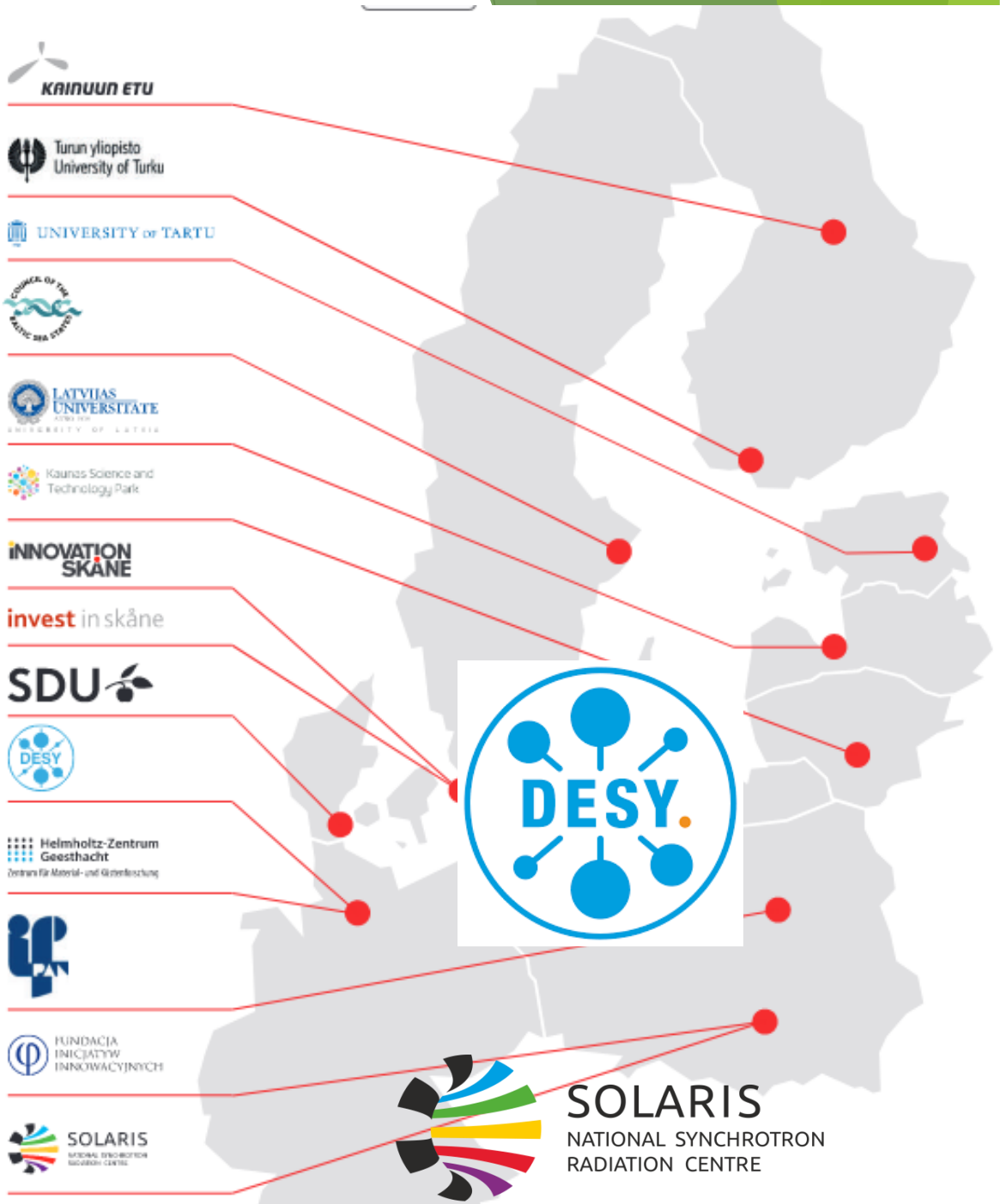




Synchrotron and Business

Synchrotron radiation is used not only for scientific purposes, but is also crucial for the development of many companies.

Companies → RESEARCH for BUSINESS BALTIC TRAM →



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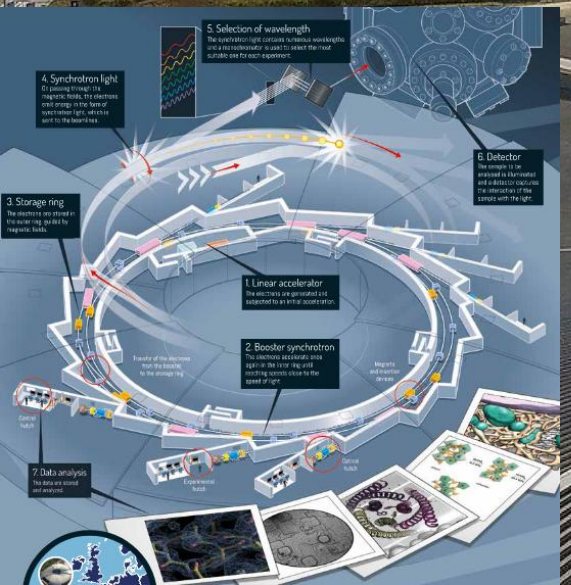
facilitates the use of large research infrastructure, including synchrotrons. The companies under this project can participate in specialized consultations and training offered by the project partners, and will gain access to synchrotron radiation sources to test their innovative ideas.

Barcelona Synchrotron Park (Businesses Park)

30000 Companies



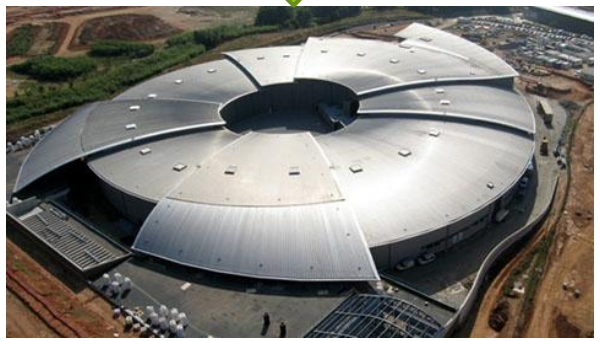
2,100

Over 2,100 industrial and academic scientists per year conducting high impact experiments

ALBA Synchrotron

Barcelona Synchrotron Park Sectors

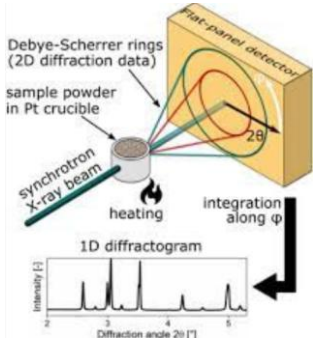
871 Companies
Turnover 15.9 Billion Euro



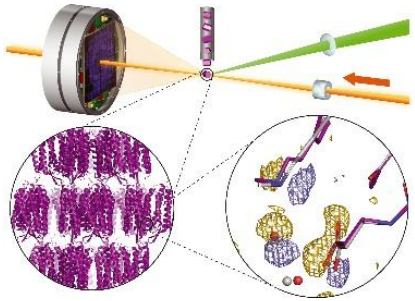
2700 Companies
Turnover 26 Billion Euro

Techniques Available in ALBA

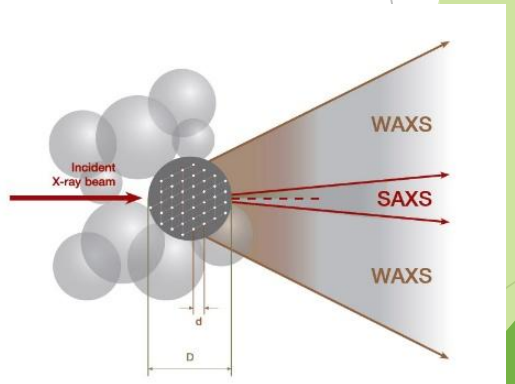
- X-ray Powder Diffraction



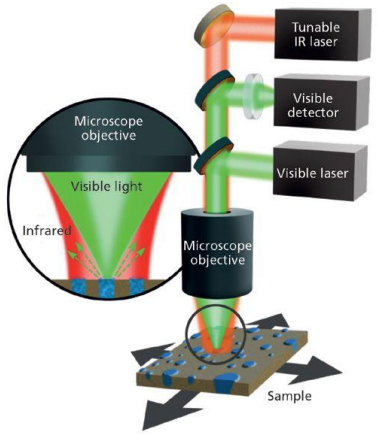
- Macromolecular Crystallography



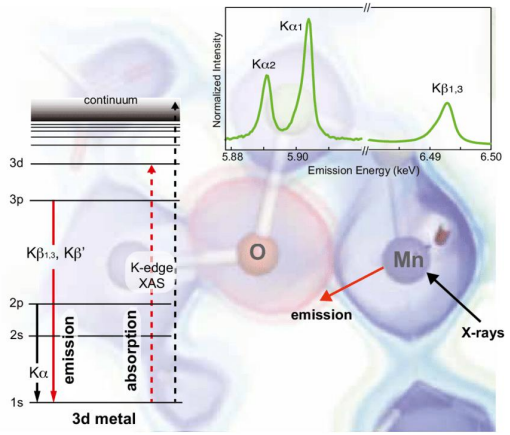
- Small and Wide Angle Scattering (SAX and WAX)



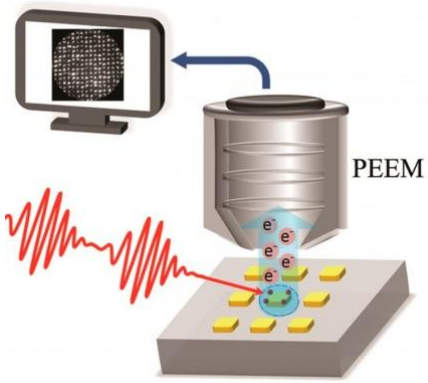
- Infrared Microspectroscopy



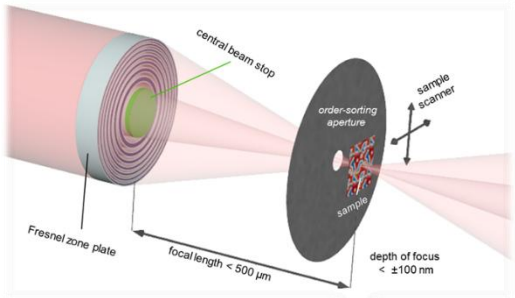
- X-ray Absorption and Emission Spectroscopy



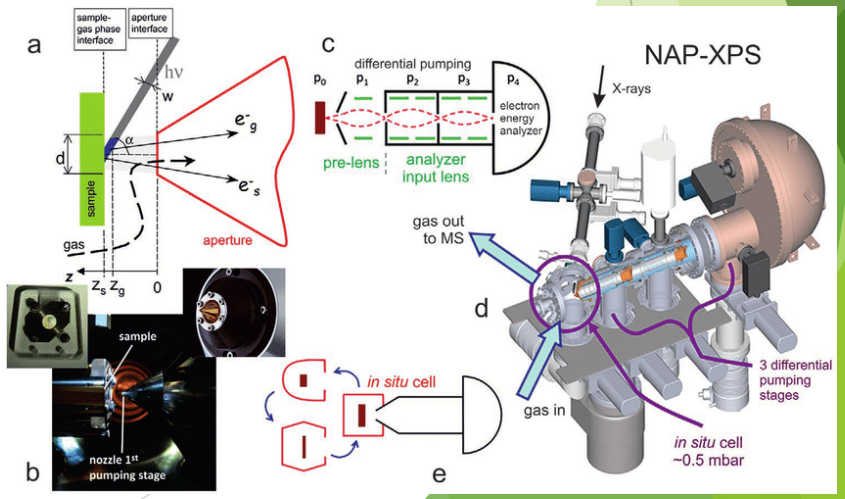
- Photoemission Microscopy

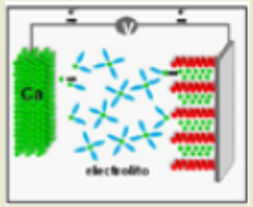


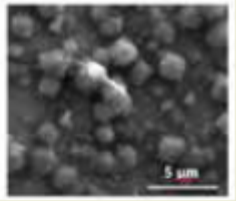
- Soft X-ray Microscopy



- Photoemission near ambient pressure spectroscopy







TOYOTA and CSIC proved viability of calcium-based batteries

The Spanish Research Council (CSIC) in collaboration with TOYOTA Motor Europe (TME) demonstrates the viability of Calcium rechargeable batteries using ALBA techniques.

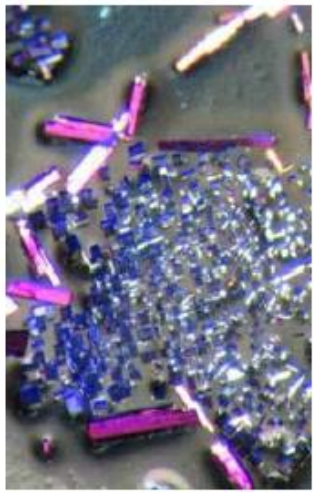
Calcium, a much more abundant and cheaper element than lithium, can act as negative electrode in rechargeable batteries. This is shown in a study elaborated by researchers of Spanish Research Council at the Institut de Ciència de Materials de Barcelona (ICMAB) in collaboration with Toyota Motor Europe (TME), and with the support of the results obtained at ALBA Synchrotron.

This study proves that calcium can be used as negative electrode in rechargeable batteries of high energy density and that they are compatible with electrolytes commonly used in lithium ion technology. These results are the basis of two filed patents and from now on researchers will direct their efforts to the development of materials for the positive electrode able to operate at a high potential to achieve high energy density batteries.

BASF, UPC and ALBA propose a methodology for producing better additives for concrete technology

Using synchrotron light, they have determined how clays and superplasticizers interact in cement pastes. These results pave the way for improving the design of new superplasticizers more resilient to the clays that usually accompany the sands used in concretes as aggregates.

Polycarboxylate (PCE) superplasticizers make concrete more fluid with less water, resulting in a substantial enhancement in workability, allowing the reduction of the water content of the paste. However, polycarboxylate superplastizers are very sensitive to the clays that may contain the sand used for the production of mortars and concretes. Therefore it is of prime importance to understand the interaction between polycarboxylate superplastizers and clays to improve the performance of this complex mixtures.



CRYSFORMA (ICIQ) characterizes polymorphs for the pharmaceutical industry at the ALBA Synchrotron

The company is using X-ray powder diffraction to solve different issues concerning the characterization of the solid state of Polymorphs of Active Pharmaceutical Ingredients.

Polymorphism is the ability of a solid substance to crystallize in more than one crystalline structure, resulting from a different arrangement of the molecules within the crystal lattice. Each of these different crystalline phases are known as polymorphs. Polymorphs of Active Pharmaceutical Ingredients (APIs), although being the same chemical entity, can have different physicochemical properties, which can affect the bioavailability of the final drug, or its processability during the manufacturing process. For this reason polymorphs are of special interest in the pharmaceutical industry.

ENANTIA uses ALBA's X-rays to detect crystalline impurities in drug products

This company collaborates with pharmaceutical companies in the identification and detection of different crystalline phases in drug substances and products.

The majority of drugs are administered as solids and solid state properties influence significantly their performance. The optimal solid form should have chemical and crystalline stability, the right pharmacokinetic profile and be easy to process. When the solid active ingredient exhibits crystalline structure transformations, they [illegible] to be taken into account for performance and intell[illegible] considerations. In the case of generic drugs [illegible] of a specific polymorph, and/or th[illegible] solvates could determine [illegible] In comparison [illegible] as it [illegible]

SAMTACK benefits from synchrotron light for improving food packaging

The company is analysing nanoparticles contained in a new food packaging system that will prevent food oxidation and extend its lifetime.

Samtack, founded in 1988 and based in Esparreguera (Barcelona), is a manufacturer of glues and adhesives specialized in the sector of graphic arts and packaging. Samtack has developed a new flexible multilayer system, in collaboration with the University of Zaragoza and the Complutense University of Madrid, that contains Selenium nanoparticles and is capable to increase food shelf life.

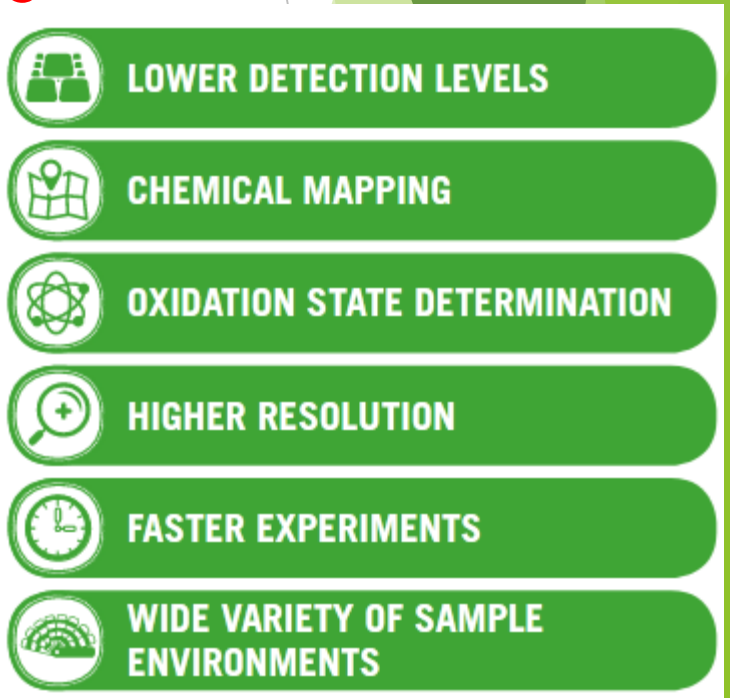
Free radicals are formed spontaneously from oxygen, moisture and UV radiation and initiate oxidation reactions quickly. As free radicals are very small, they are capable of traversing the plastic layers. The new multilayer system developed by Samtack contains Selenium nanoparticles in one of the layers, and these Selenium nanoparticles are capable to absorb free radicals and therefore to prevent food from oxidation.

What is the attraction of synchrotron X-ray studies to industrialists?

Industrialists come synchrotron because the level of detail which is resolved by synchrotron based experiments is far superior to standard laboratory techniques

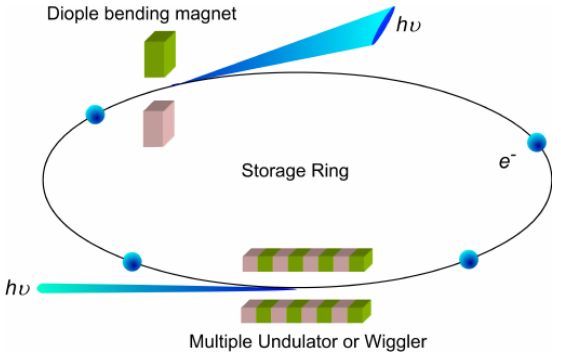
Synchrotron offers industry cutting-edge techniques for characterizing materials and processes at micro and nanoscopic levels by using synchrotron light, providing results focused on a company's specific needs.

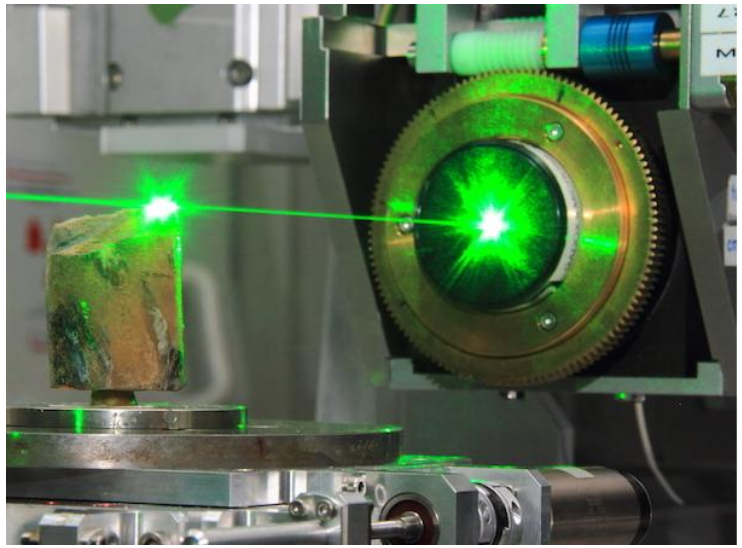
Research with synchrotron X-rays boosts industrial innovation

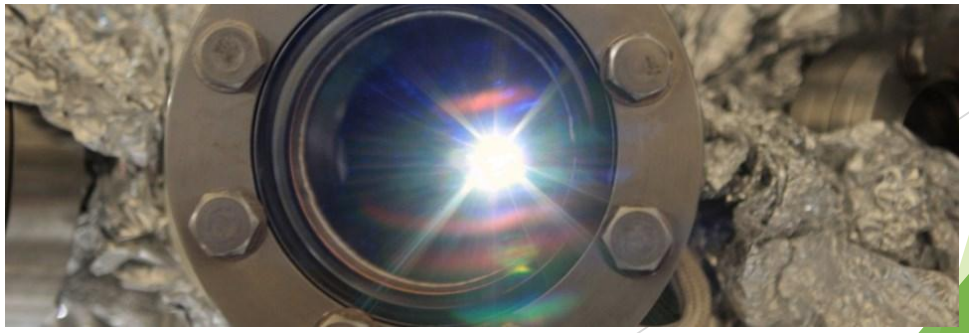


Properties of Synchrotron Radiation

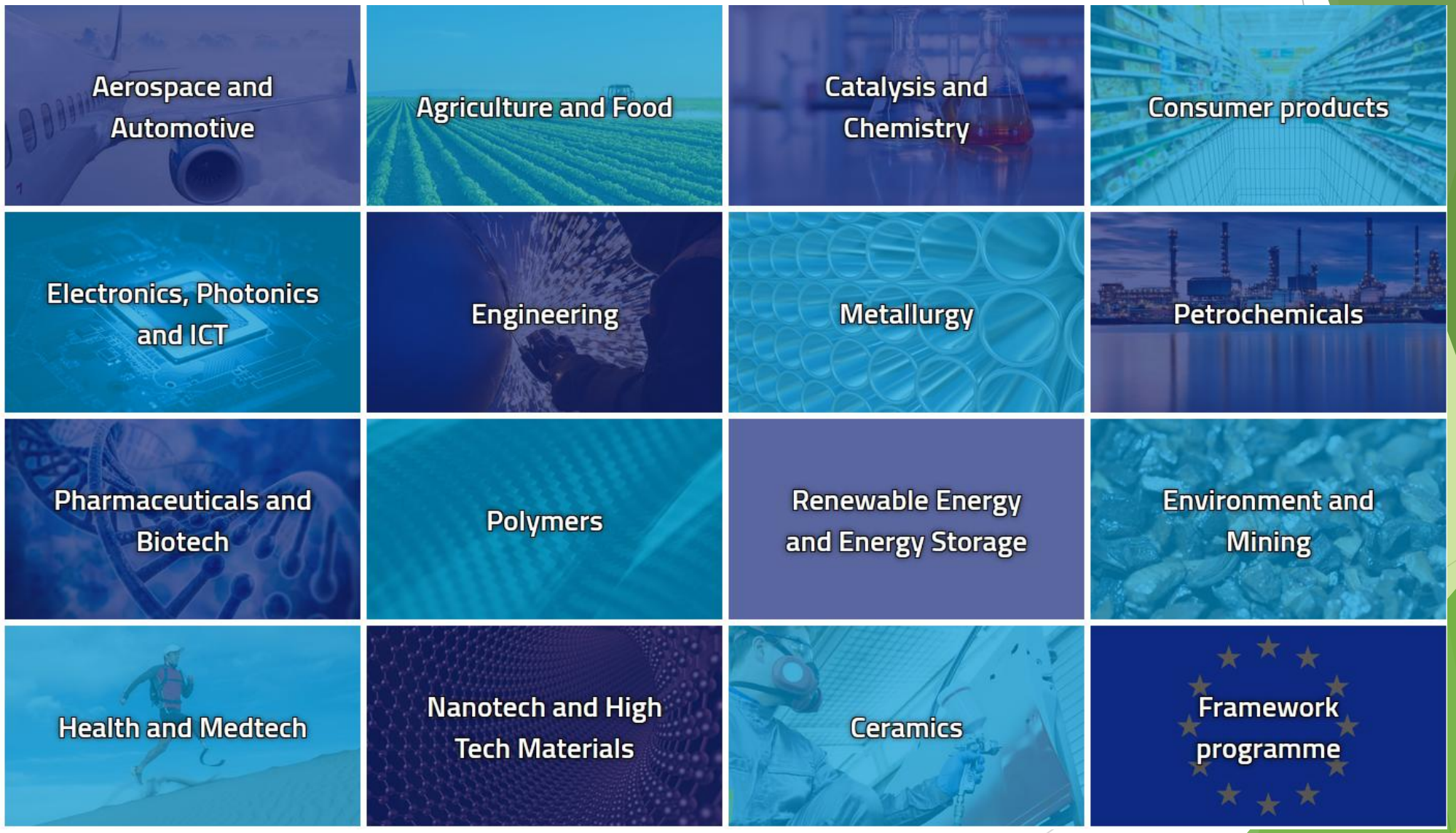
- Its extreme Intensity (ten of millions times higher than any laboratory source
- Covers a wide range of energy (IR to hard X-ray)
- Strongly collimated
- Can be polarized linearly, circularly, and elliptically
- higher quality experimental data (higher signal-to-noise ratio), faster and more precise results



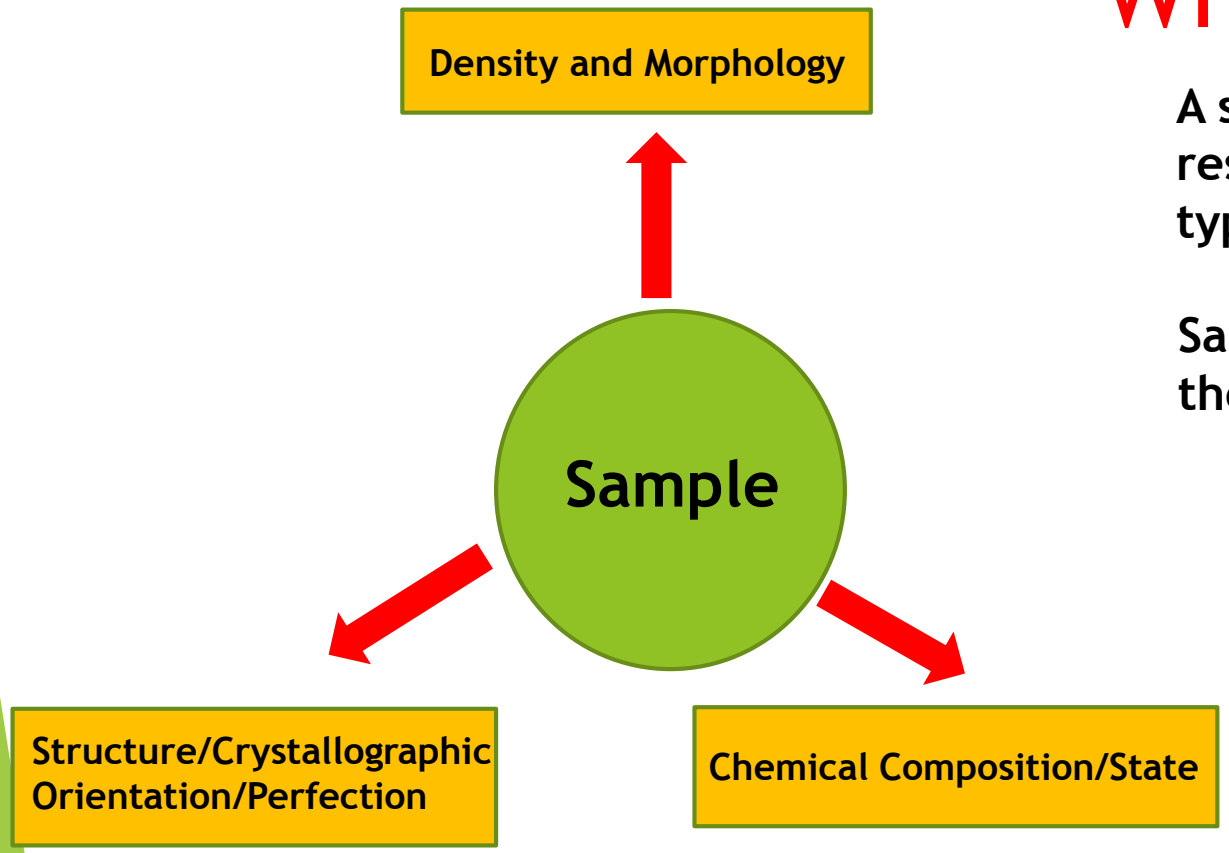




Industrial Applications



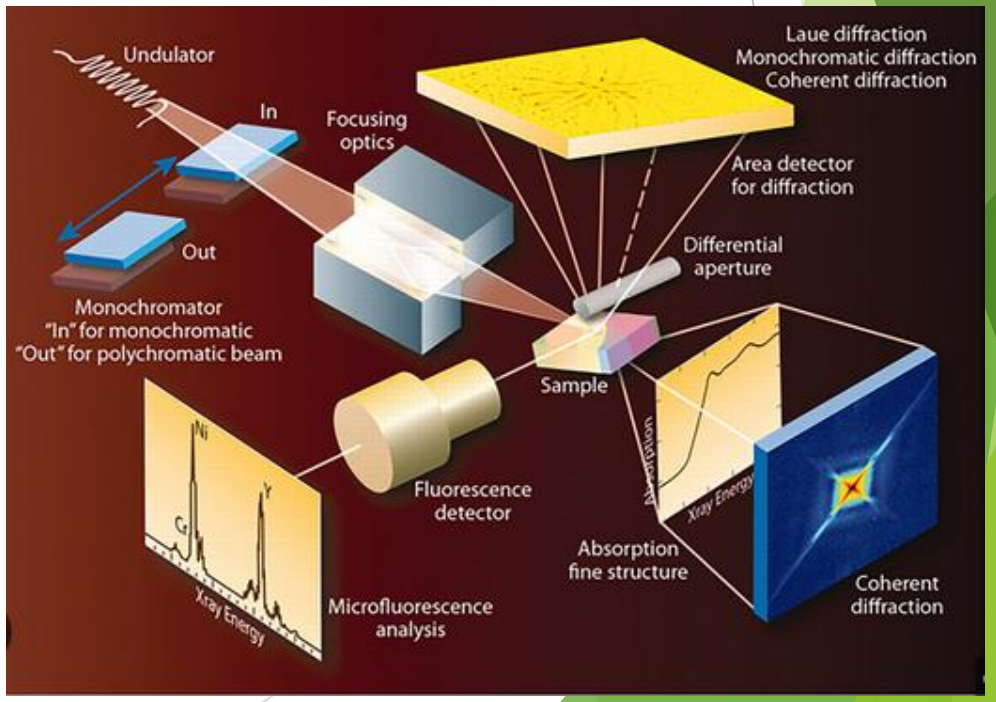
Synchrotron based X-ray Imaging

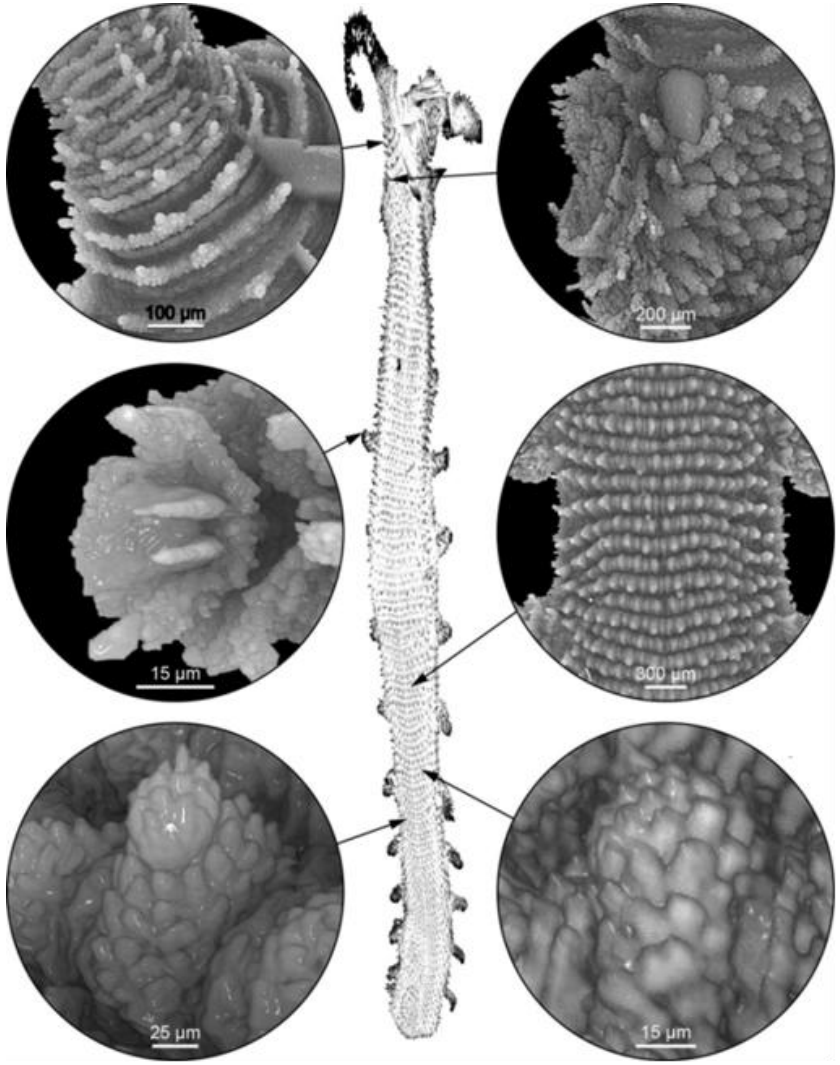


Why?

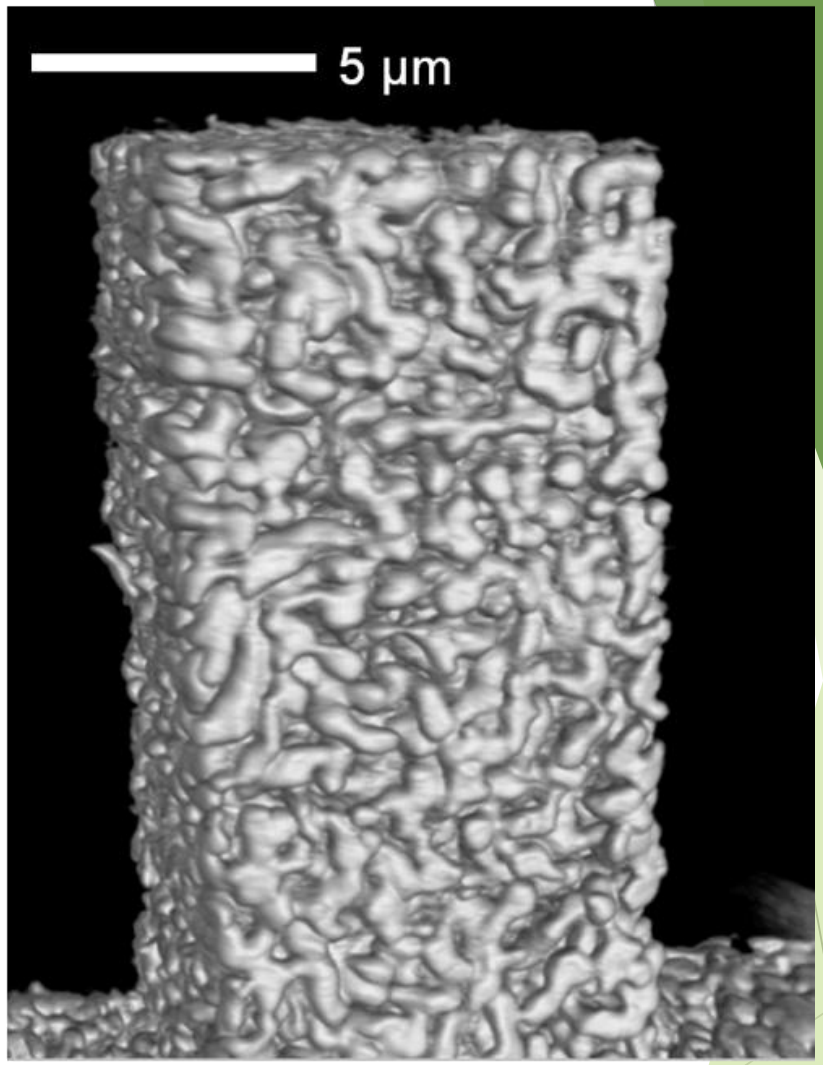
A substantial fraction of synchrotron radiation-based research is being carried out today with an 'imaging-type' approach.

Samples are not homogeneous, and that a 'local', in the μm or sub-μm range, characterization is needed.

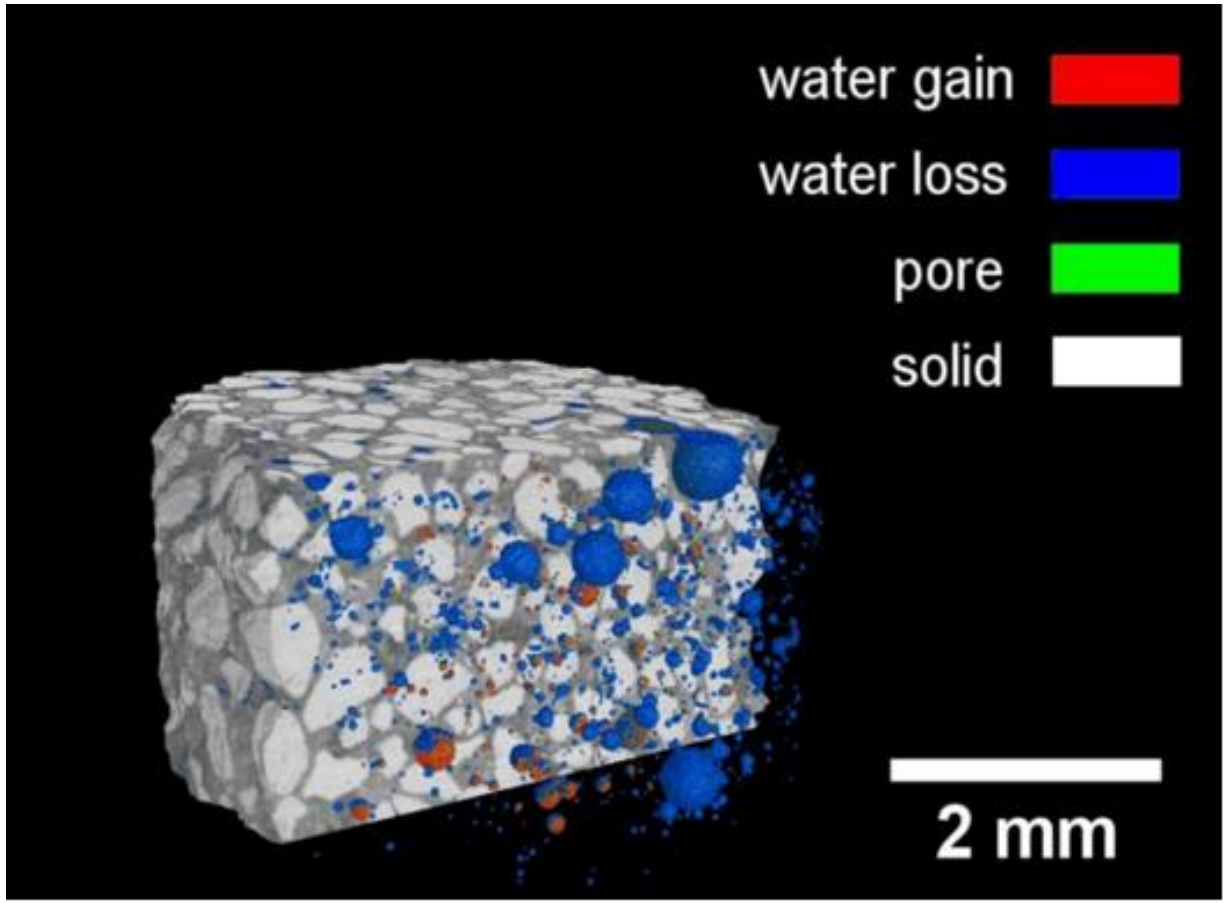




3D reconstruction of a 100 million years old, exceptionally well preserved amber-embedded worm (total length of ca. 13 mm) found in Myanmar. Recorded at the micro tomography endstation of beamline P05/IBL. Source: I.S. Oliveira et al., *Current Biology* **26**, 2594 (2016)



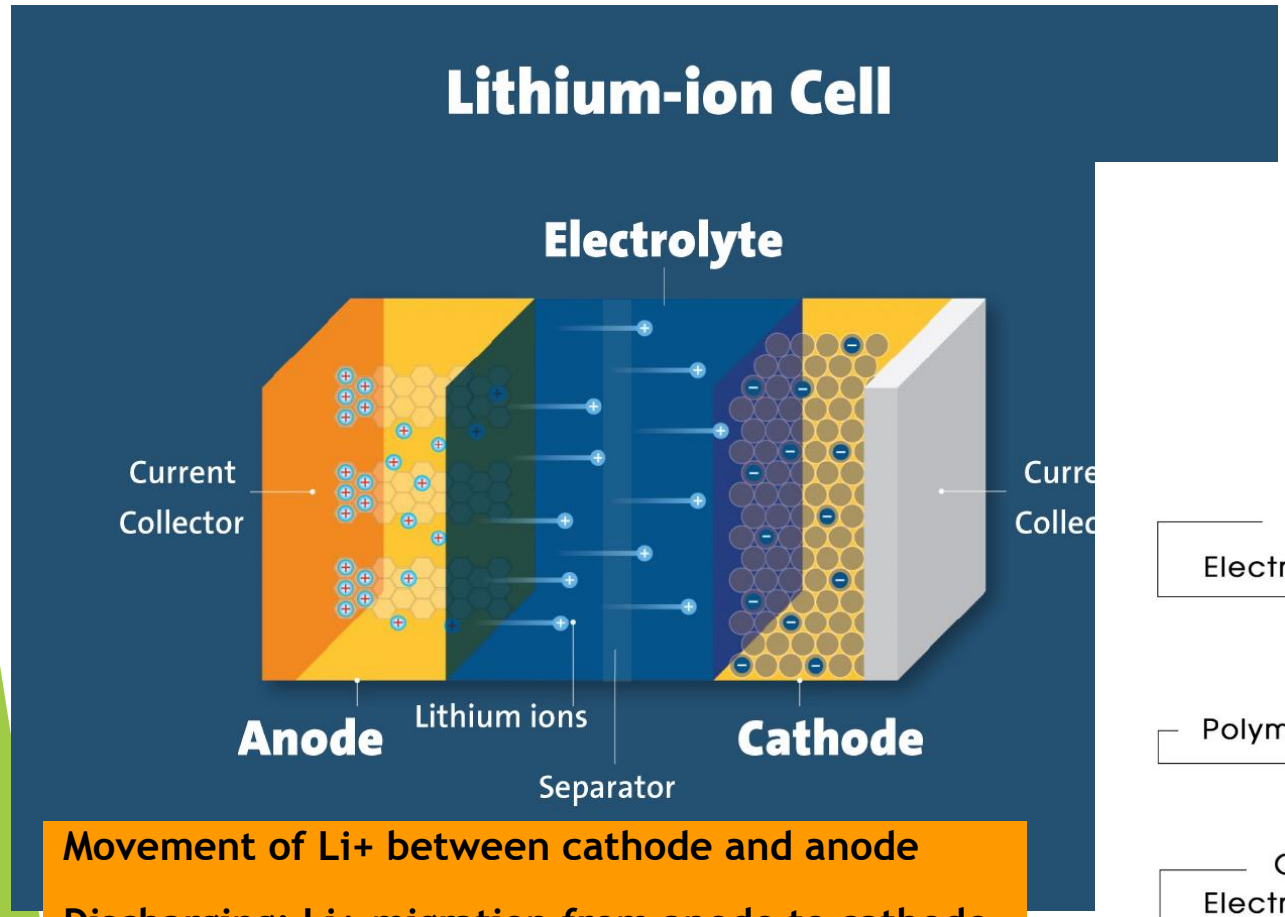
3D reconstruction of a nanoporous gold column, with its filament structures having a typical cross section of 400 nm. Recorded at the nano tomography endstation of beamline P05/IBL. Source: Prof. Erica Lilleodden, HZG



Changes of water distribution in concrete monitored during the the process of concrete drying. Recorded at the micro tomography endstation at beamline P07/HEMS. Source: F. Yang et. al., *Proc. SPIE Developments in X-Ray Tomography X*, **99670L** (2016)

Renewable Energy and Energy Storage

- Lithium-ion Batteries for Energy Storage Applications

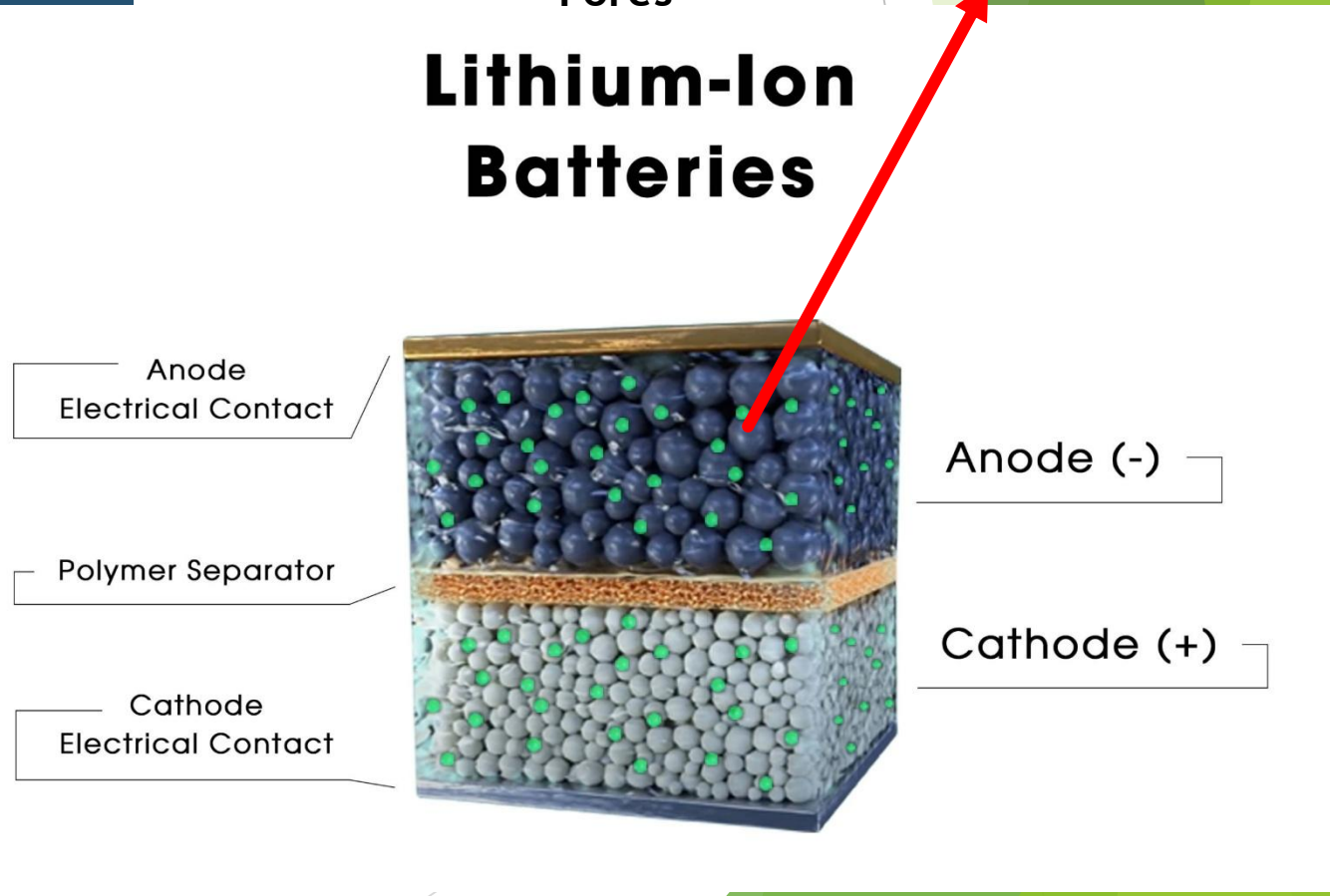


Movement of Li+ between cathode and anode

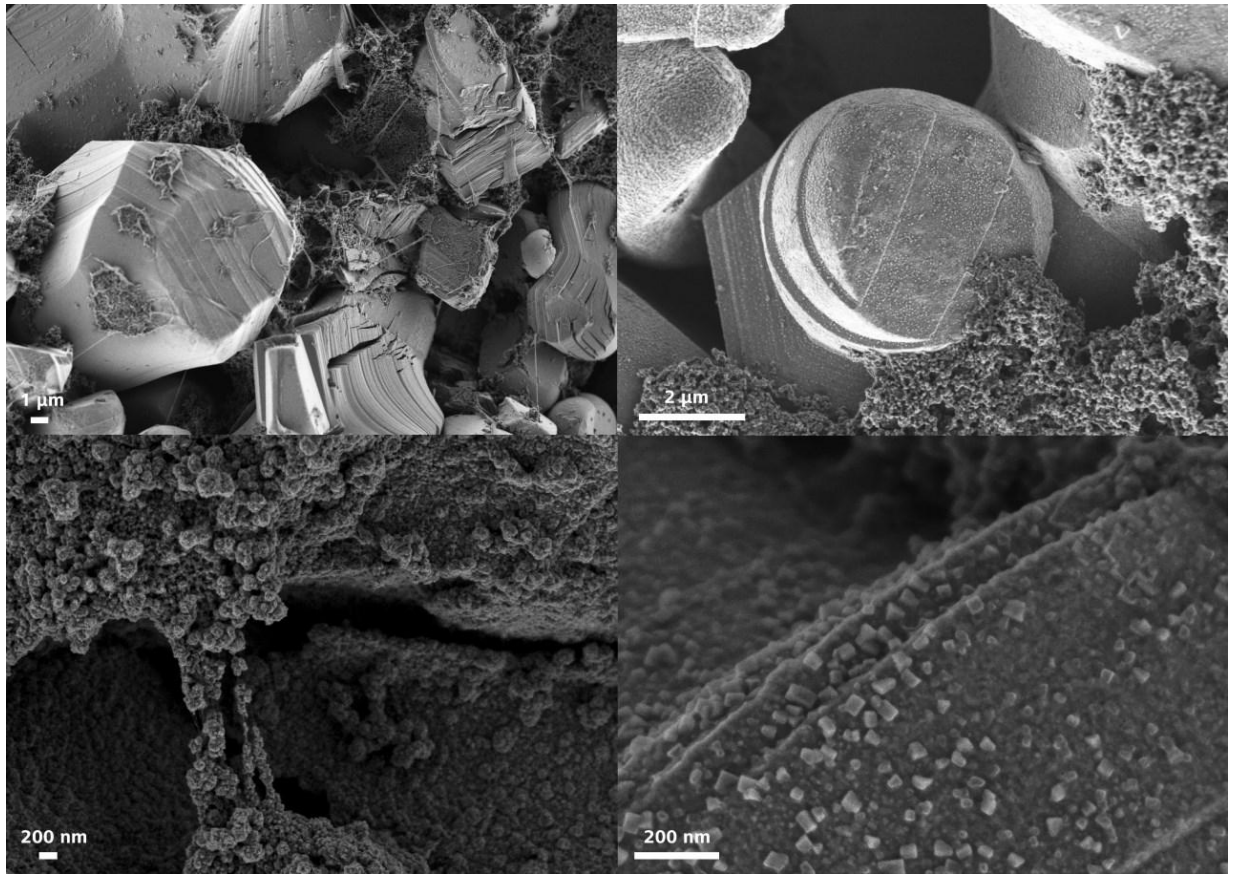
Discharging: Li+ migration from anode to cathode

Charging: Li+ migration from cathode to anode

Active Material ($LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$) NMC
Conductive Carbon
Binder (polyvinylidene Fluoride)
Pores



Scanning Electron Microscopy of Lithium-ion Battery



ZEISS FE-SEM instrument

- **The performance the Lithium-ion batteries depends strongly on the Microstructure of the electrodes**
- **The distribution of heavy elements (NMC) and light elements (CBD) in the composite electrode in nanometer**

X-ray holotomography with nanometric resolution to study the three structural components of different electrodes with a variable amount of CBD. The technique exploits phase contrast, which allows one to properly

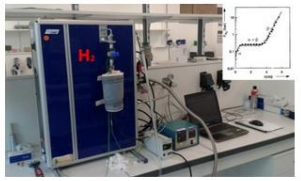
LRCS Company

Lithium-ion Batteries

(Resp. M. Morcrette)

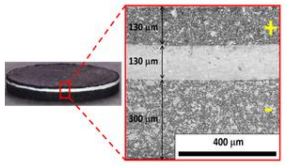
Hydrogen Storage

(Resp. R. Janot)



All Solid State Batteries

(Resp. V. Seznec)

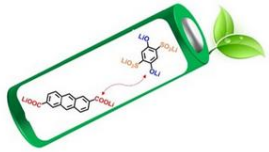


Sodium-ion Batteries

(Resp. C. Masquelier)

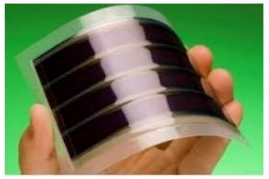
Organic Batteries

(Resp. M. Becuwe)



Photovoltaïc Devices

(Resp. F. Sauvage)

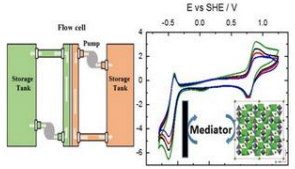


Lithium-Sulfur Batteries

(Resp. M. Morcrette)

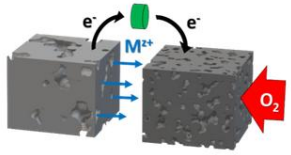
Redox-Flow Batteries

(Resp. E. Baudrin)



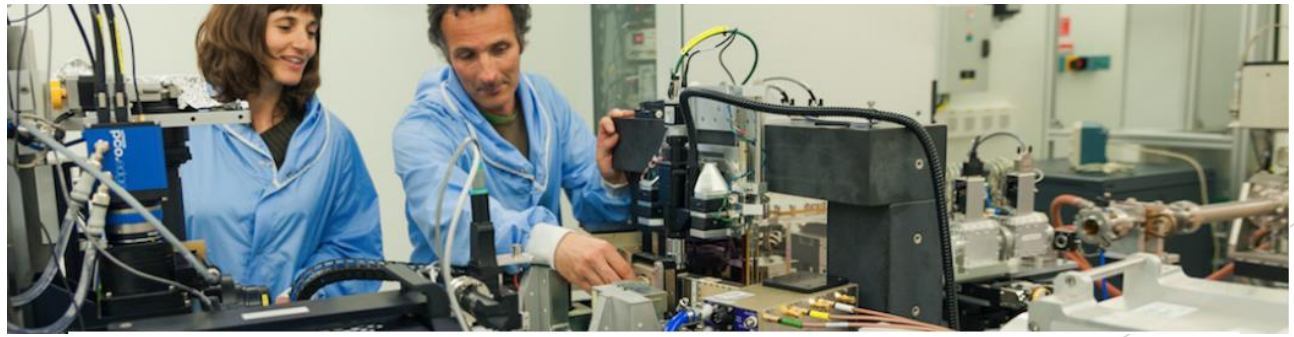
Metal-Air Batteries

(Resp. A. Franco)

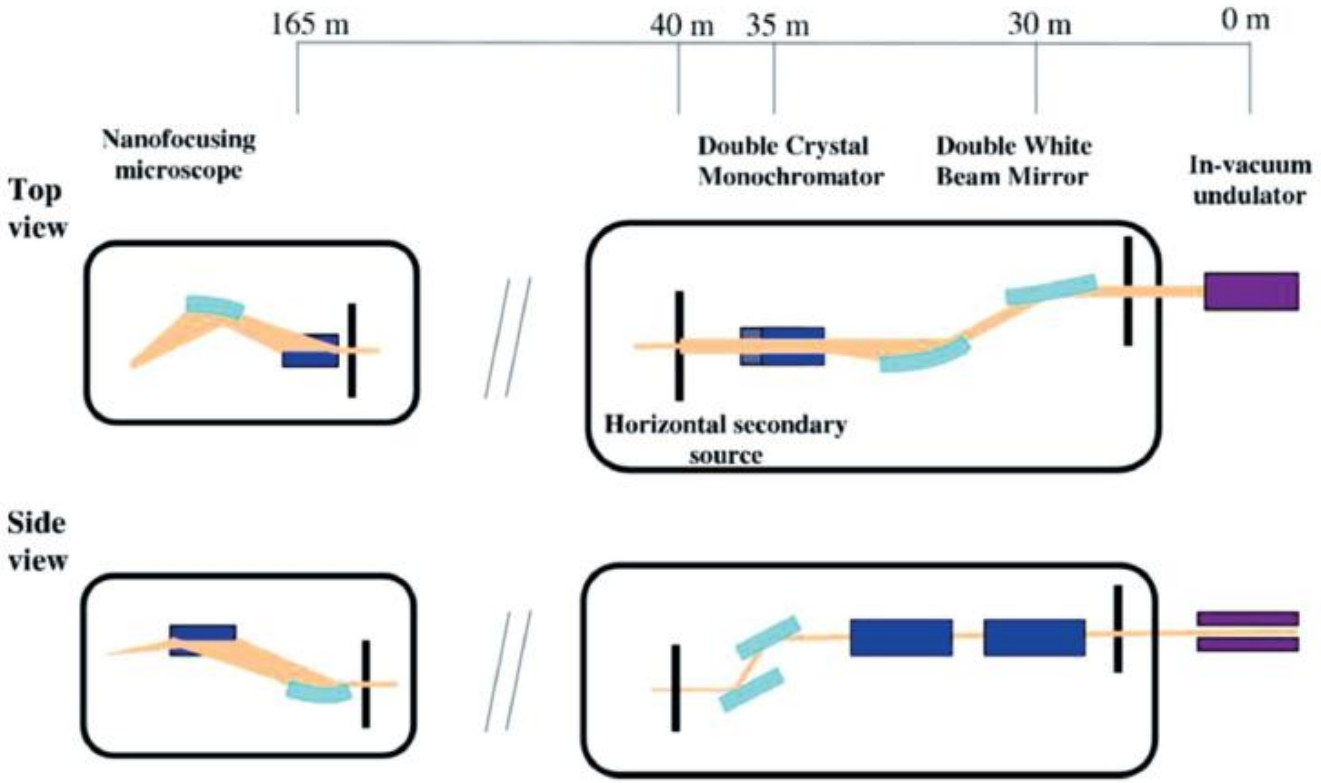


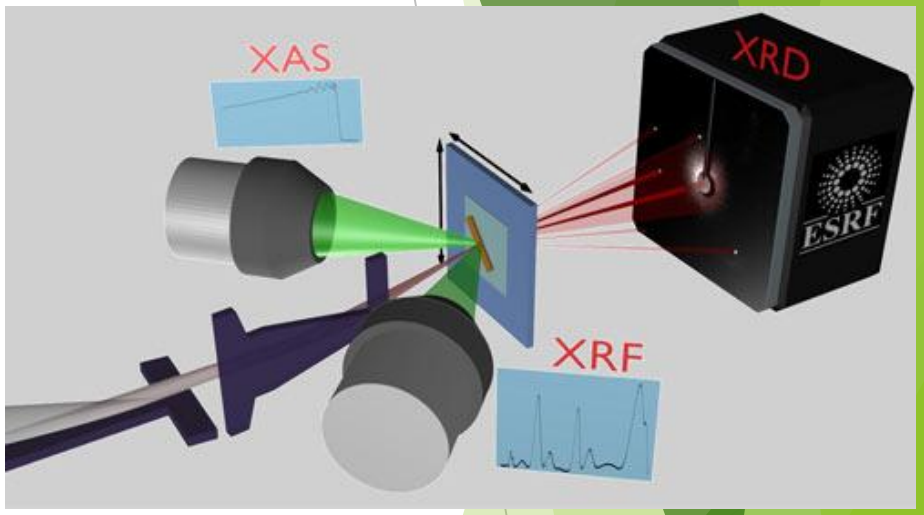
RENAULT Company

ID16B Beamline at ESRF : NANO-ANALYSIS BEAMLINE



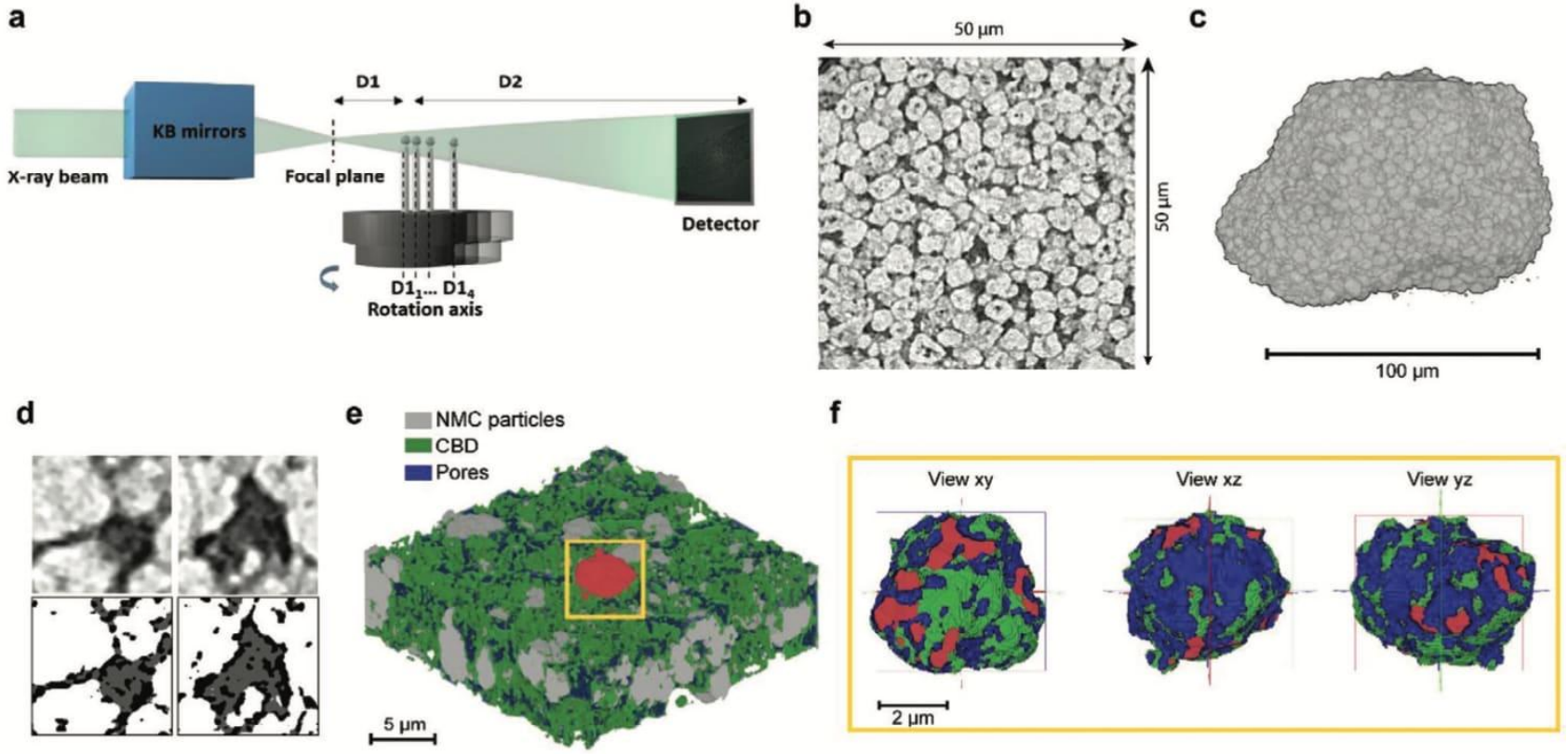
General optical layout of ID16B Beamline at ESRF

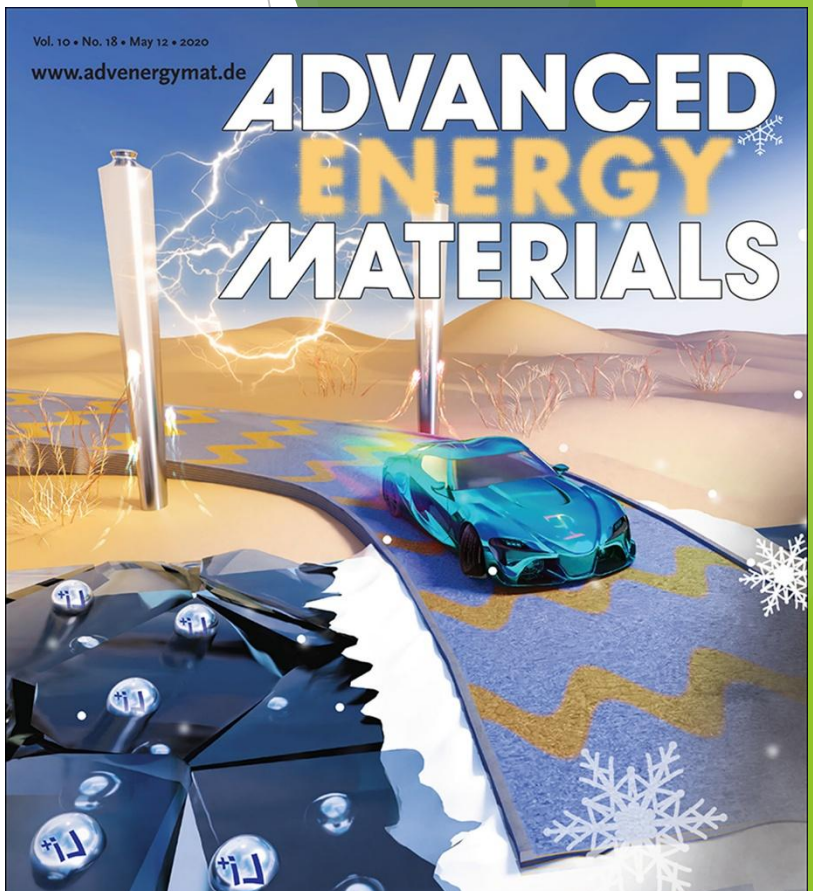




- nano X-ray fluorescence (XRF)
- Nano X-ray absorption spectroscopy (nano-XAS)
- Nano-tomography
- 2D/3D nano X-ray diffraction (nano-XRD)
- Nano X-ray excited optical luminescence (nano-XEOL)
- Nano X-ray beam induced current (nano-XBIC)

Tomography Techniques as a powerful Tool for the Microstructure of the Electrodes



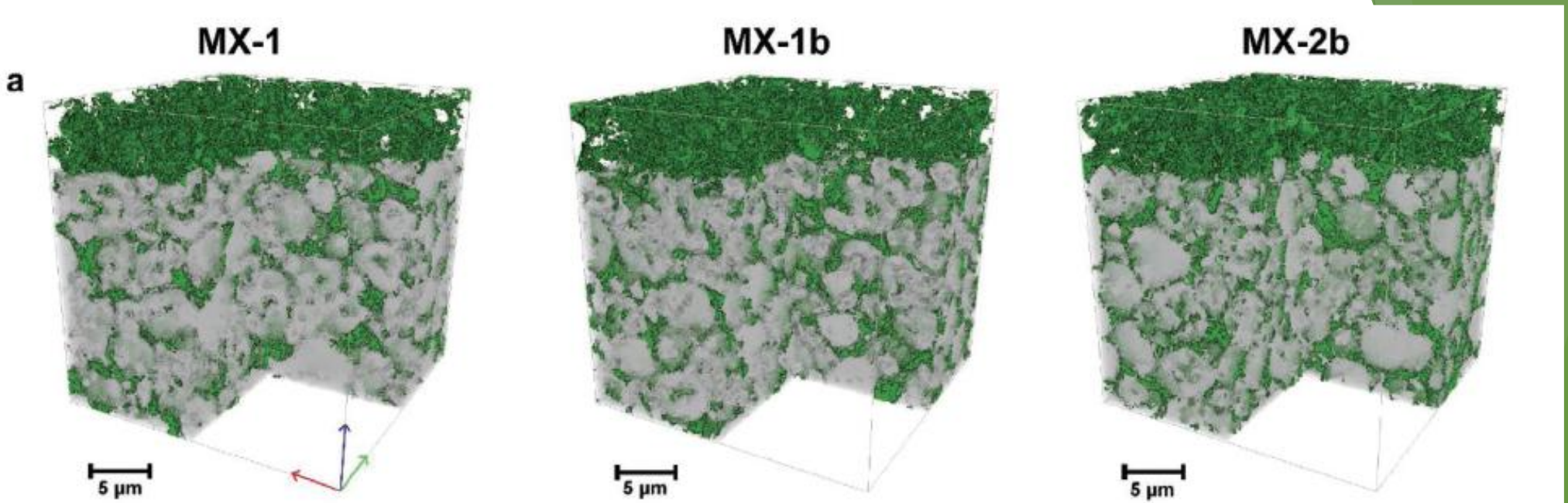


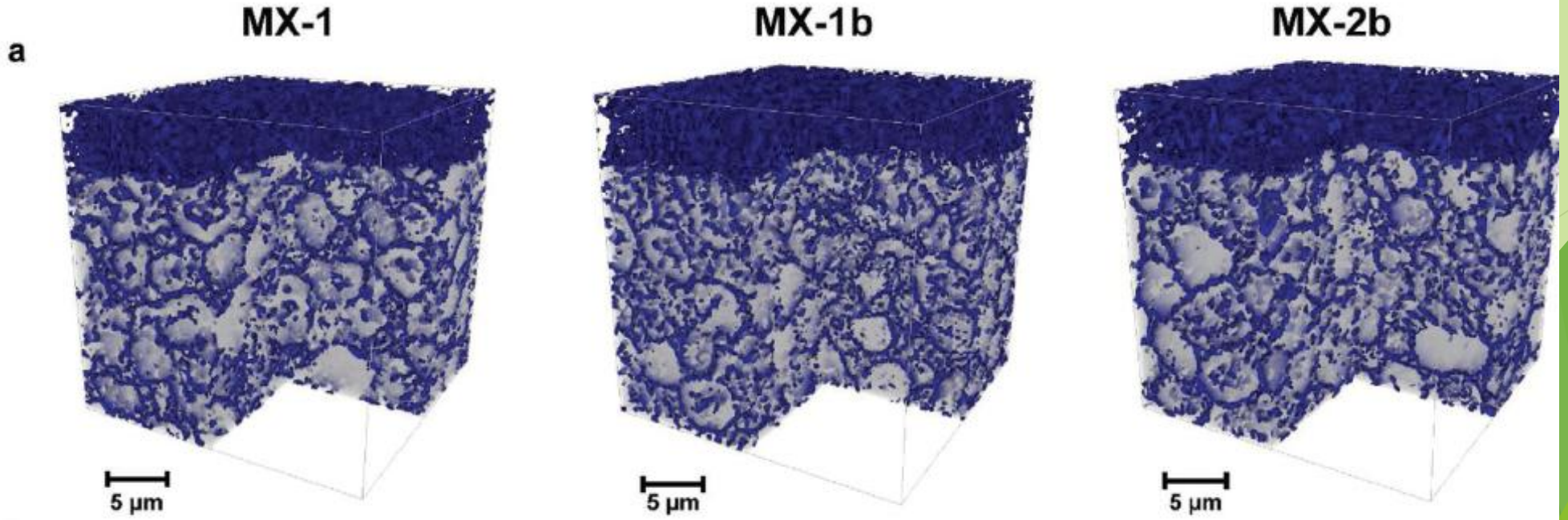
ADVANCED ENERGY MATERIALS

Full Paper

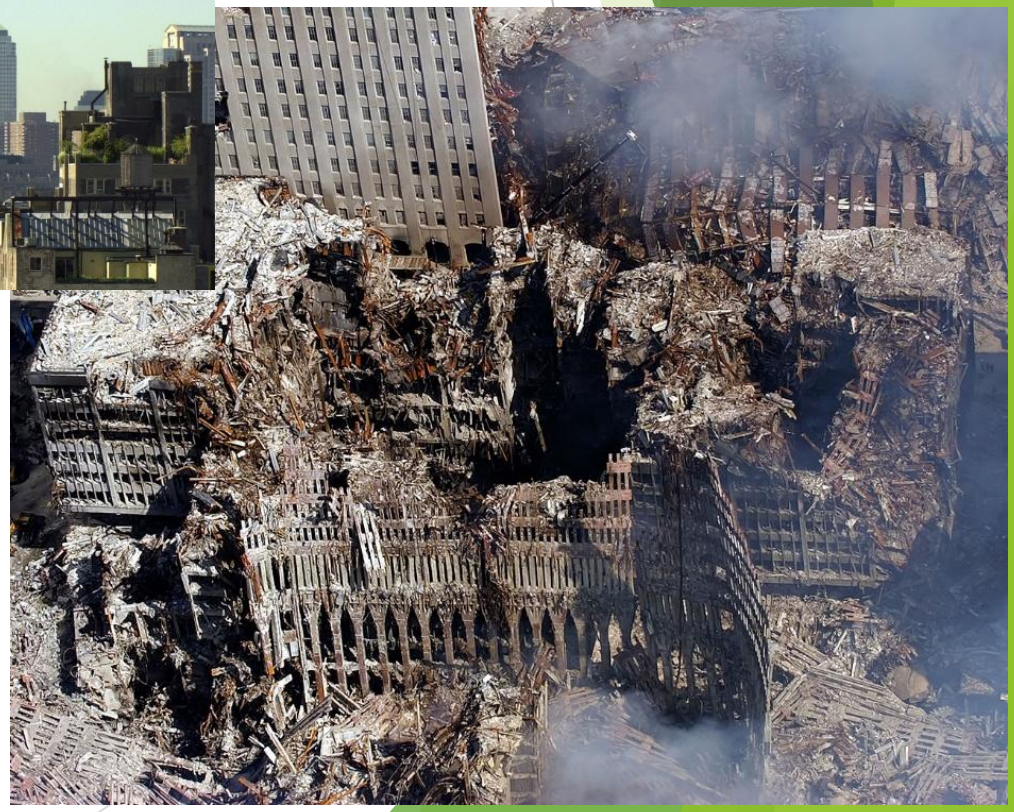
3D Quantification of Microstructural Properties of $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$ High-Energy Density Electrodes by X-Ray Holographic Nano-Tomography

Tuan-Tu Nguyen, Julie Villanova, Zeliang Su, Rémi Tucoulou, Benoît Fleutot, Bruno Delobel, Charles Delacourt, Arnaud Demortière

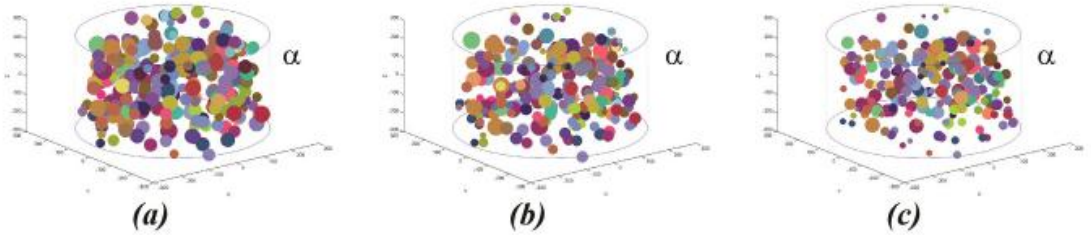

MX-1
MX-1b
MX-2b
a
5 µm
5 µm
5 µm


MX-1
MX-1b
MX-2b
a
5 µm
5 µm
5 µm

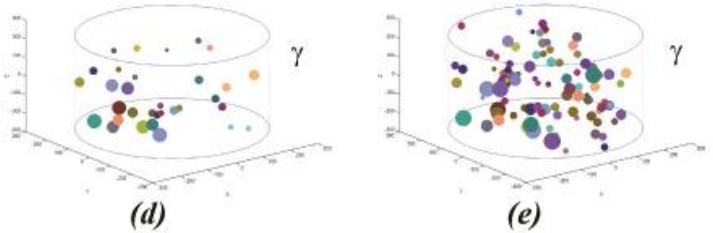
Making A fire-Resistant Steel



Twin Towers collapse

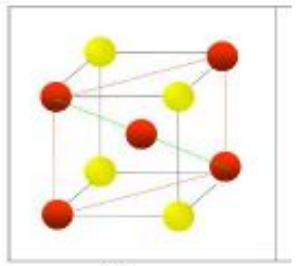


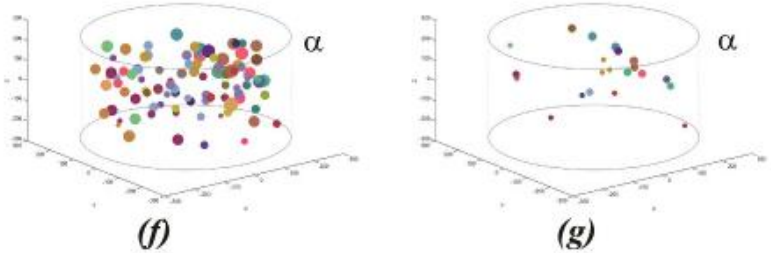
1103 K

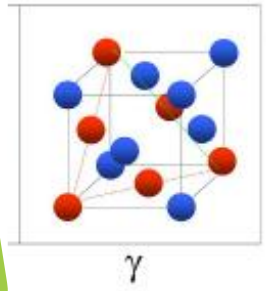


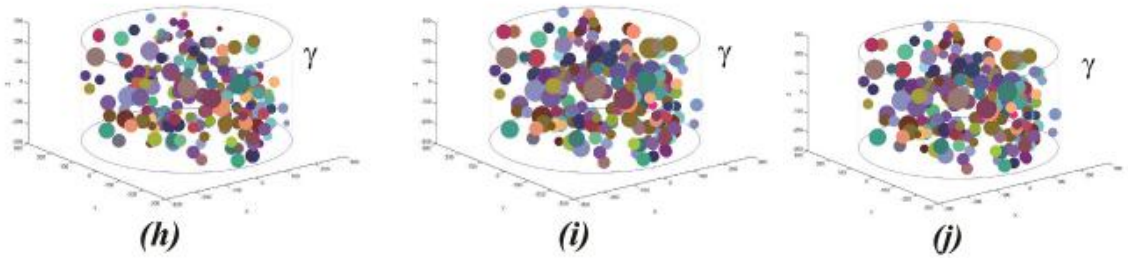
1109 K

1113 K









1117 K

1119 K

1121 K

ID11 Material Science Beamline at ESRF

In Situ Observation of the nucleation process inside steel as it is submitted to temperature variation especially in the phase transitions

Controlling the nucleation means controlling the properties of the material

SCIENTIFIC REPORTS

Preferential Nucleation during Polymorphic Transformations

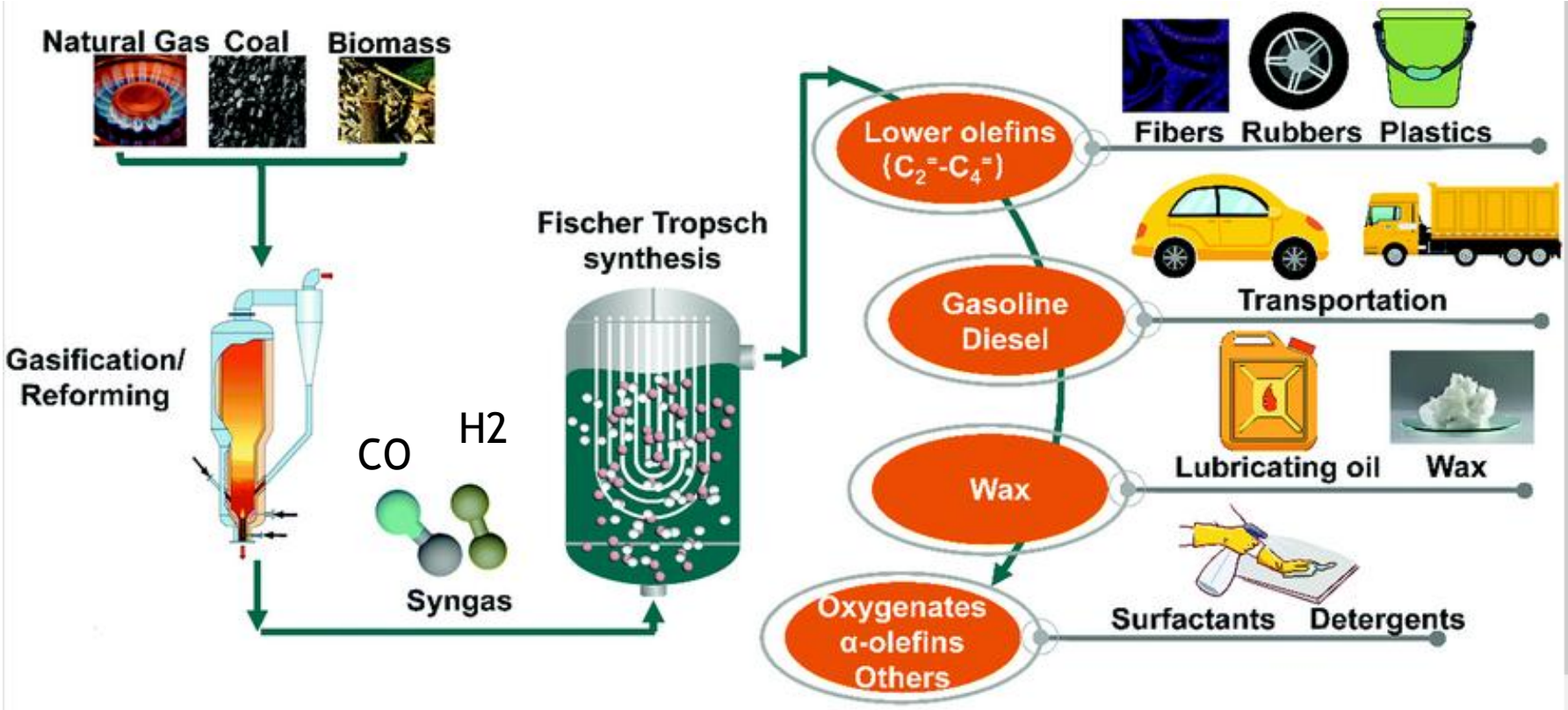
H. Sharma†, J. Sietsma & S. E. Offerman

Petrochemical Applications of Synchrotron Facility

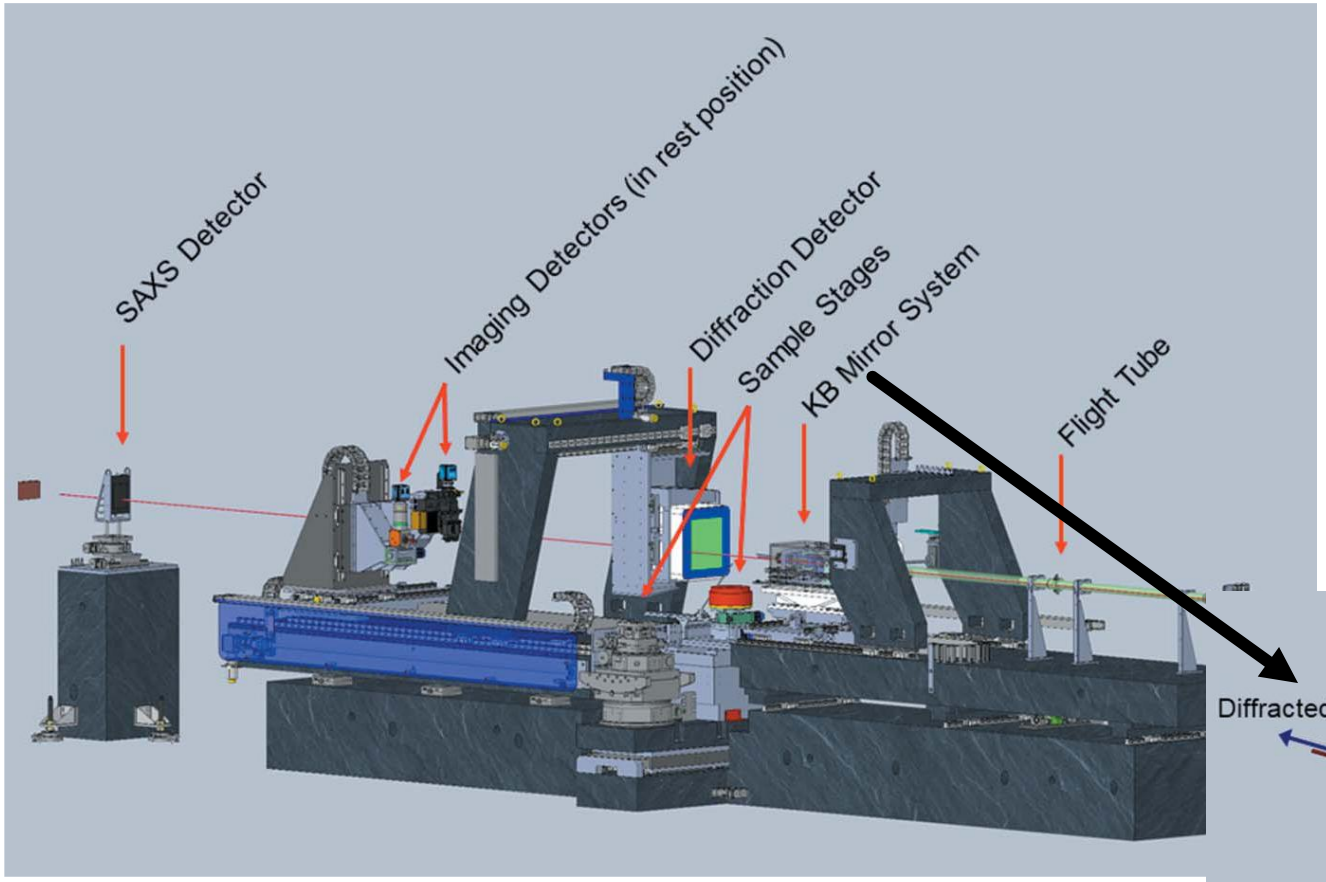
- Infrared microscopy analysis of fluid inclusions
- Analysis of trace elements of crude oil to determine the path followed by oil during migration.
- 3D in situ distribution and flow of oil and water in oil-wet matrices using microtomography.
- Studying the conditions of high temperature and high pressure that take place in future oil reservoirs, which are likely to be located much deeper than current ones.
- Studying the behaviour of the mixed fluids (oil, water, gas and sand) that originate in the reservoir when submitted to changes of temperature and pressure during the transport to the surface through small-angle X-ray scattering.
- Elimination of S, N, Ni, V from crude oil is of major environmental importance. XANES is able to provide clues as to the local coordination of these elements and even more interesting on solid heavy oil deposits.
- Identifying structural and electronic characteristics of catalytic materials under in situ/operando conditions.

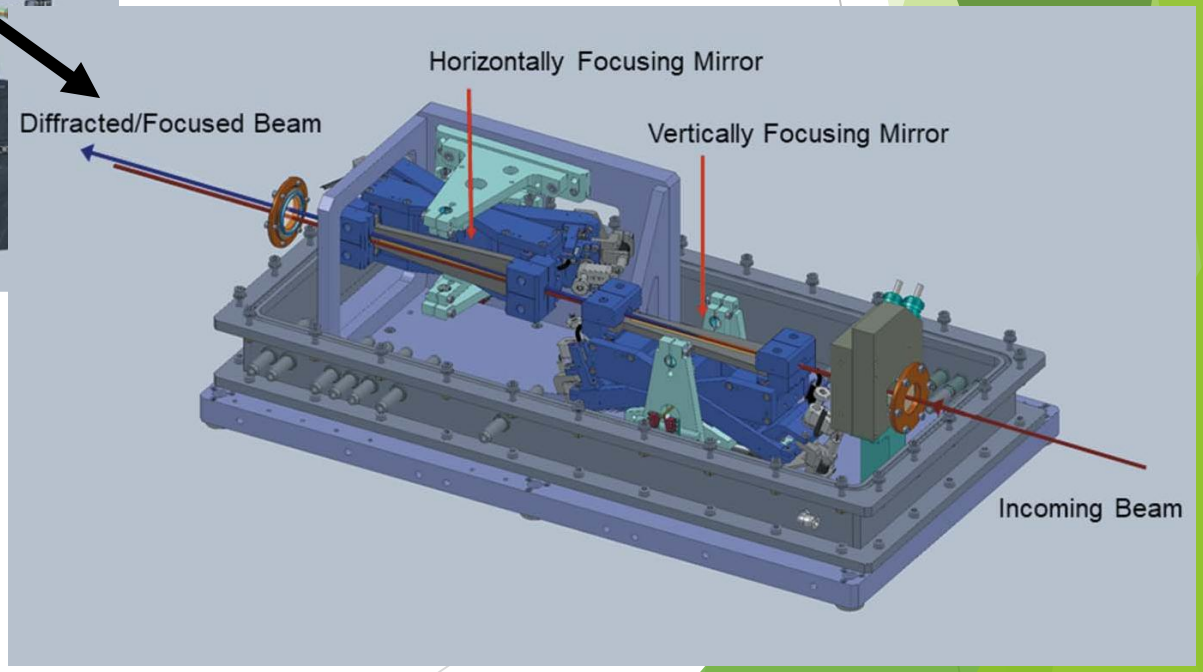
Fischer-Tropsch Synthesis

A heterogeneous catalytic process that converts biomass-derived syngas (mixture of CO and H_2) to synthetic liquid fuels and valuable chemicals. FT synthesis has received renewed interest in recent years due to the necessity to decrease global dependency on fossil fuels



ID15A-Material Chemistry and Material Engineering





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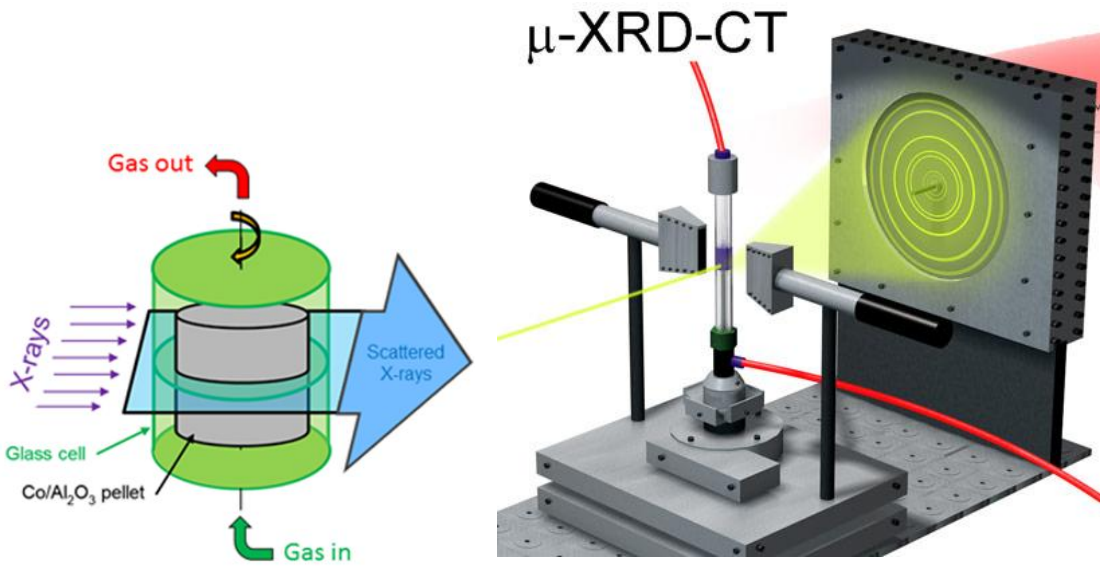
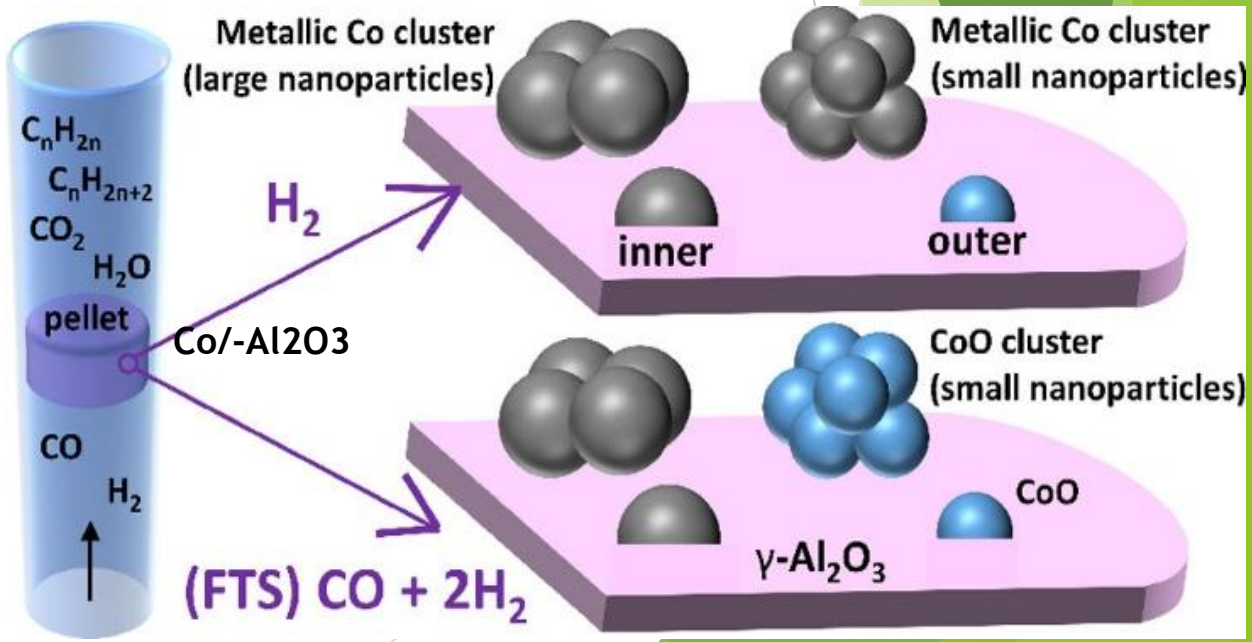
Real-Time Scattering-Contrast Imaging of a Supported Cobalt-Based Catalyst Body during Activation and Fischer–Tropsch Synthesis Revealing Spatial Dependence of Particle Size and Phase on Catalytic Properties

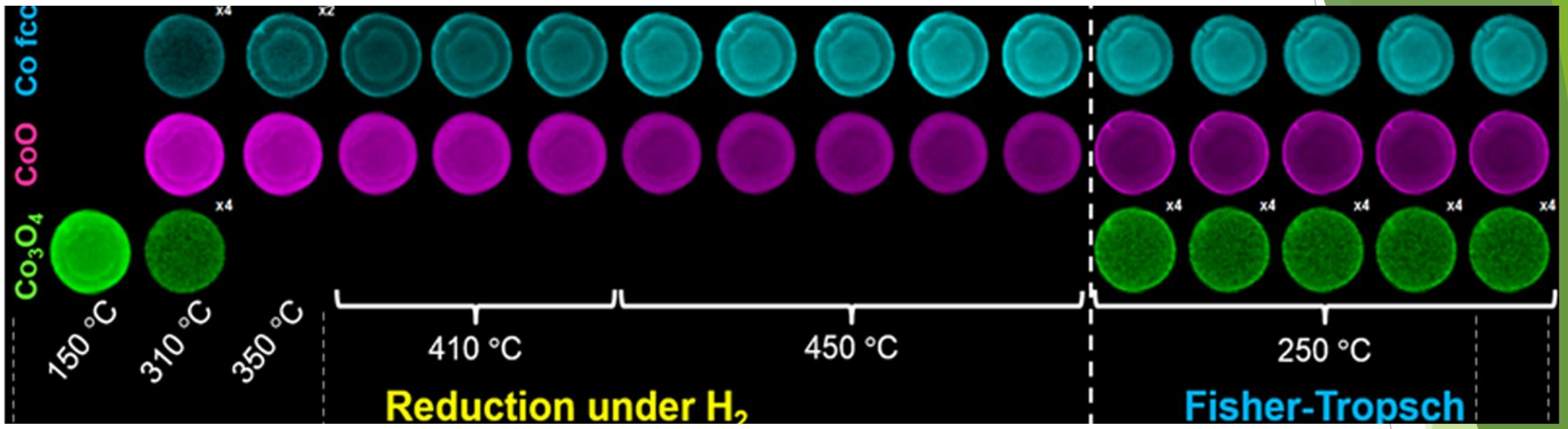
Pierre Senecal[†‡], Simon D. M. Jacques[†§], Marco Di Michiel[∥], Simon A. J. Kimber[∥], Antonis Vamvakeros[†‡], Yaroslav Odarchenko[†‡], Ines Lezcano-Gonzalez[†‡], James Paterson[⊥], Ewen Ferguson[⊥], and Andrew M. Beale[*†‡]

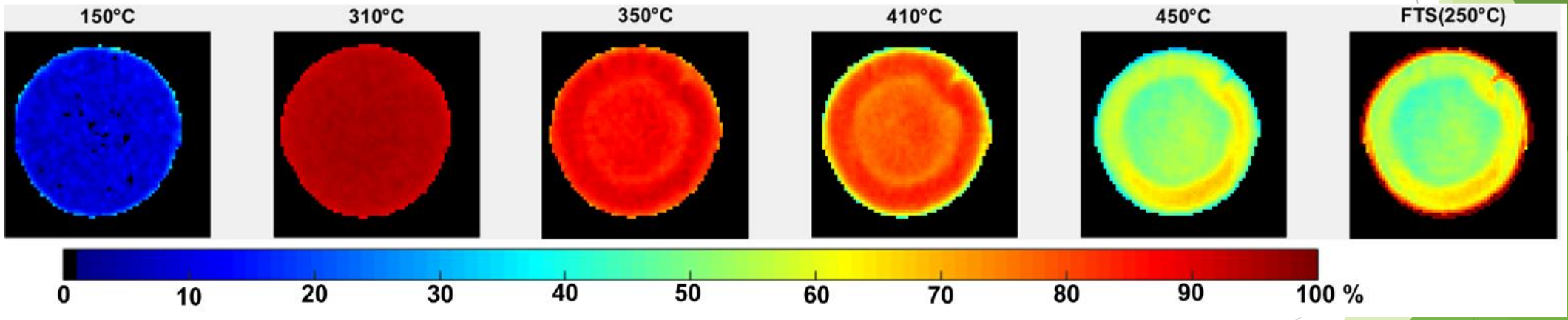
View Author Information

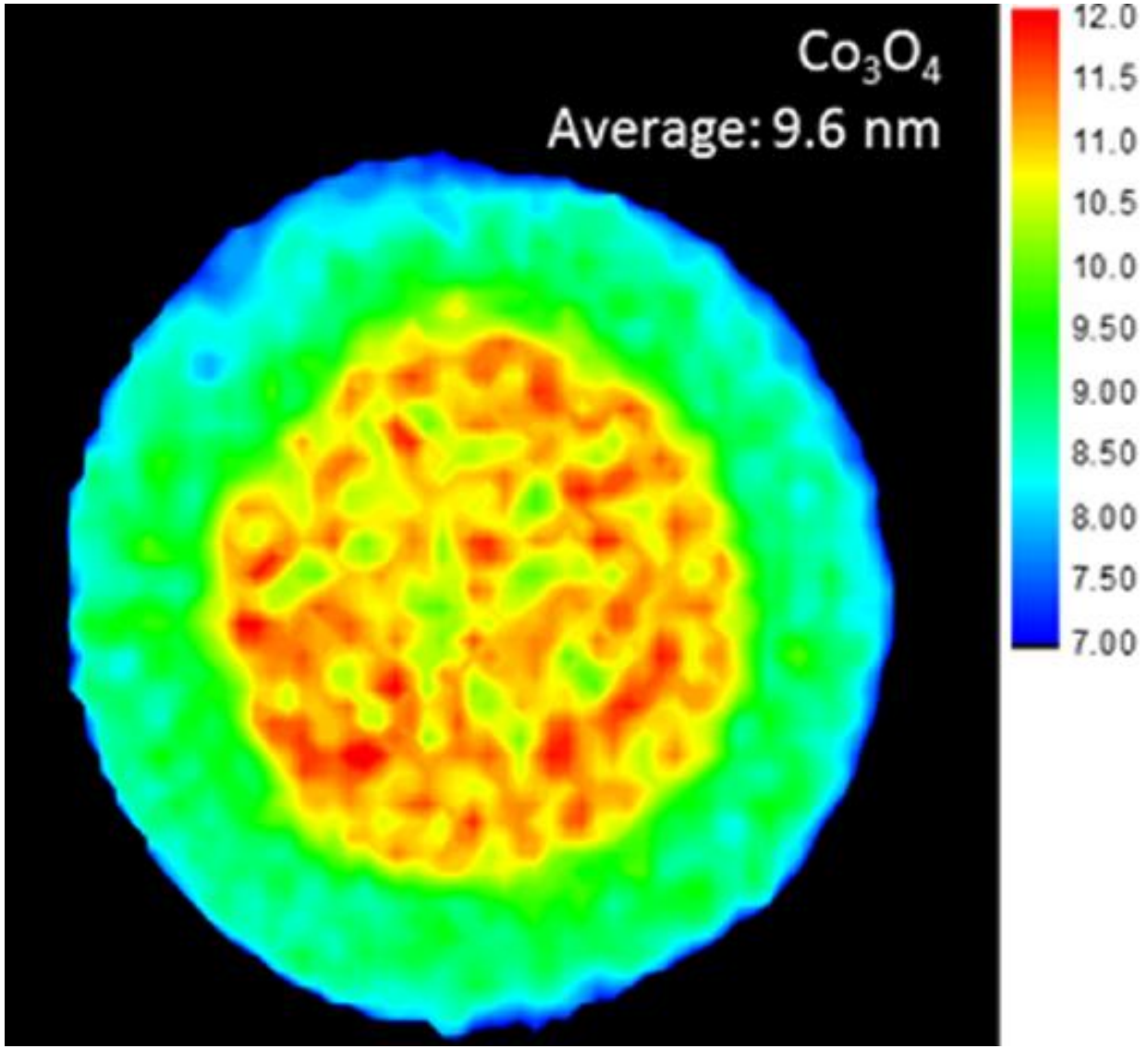


X-ray diffraction tomography




Co fcc
CoO
Co_3O_4
x4
x2
x4
x4
x4
x4
x4
x4
150 °C
310 °C
350 °C
410 °C
450 °C
250 °C
Reduction under H_2
Fisher-Tropsch


150°C
310°C
350°C
410°C
450°C
FTS(250°C)
0
10
20
30
40
50
60
70
80
90
100 %



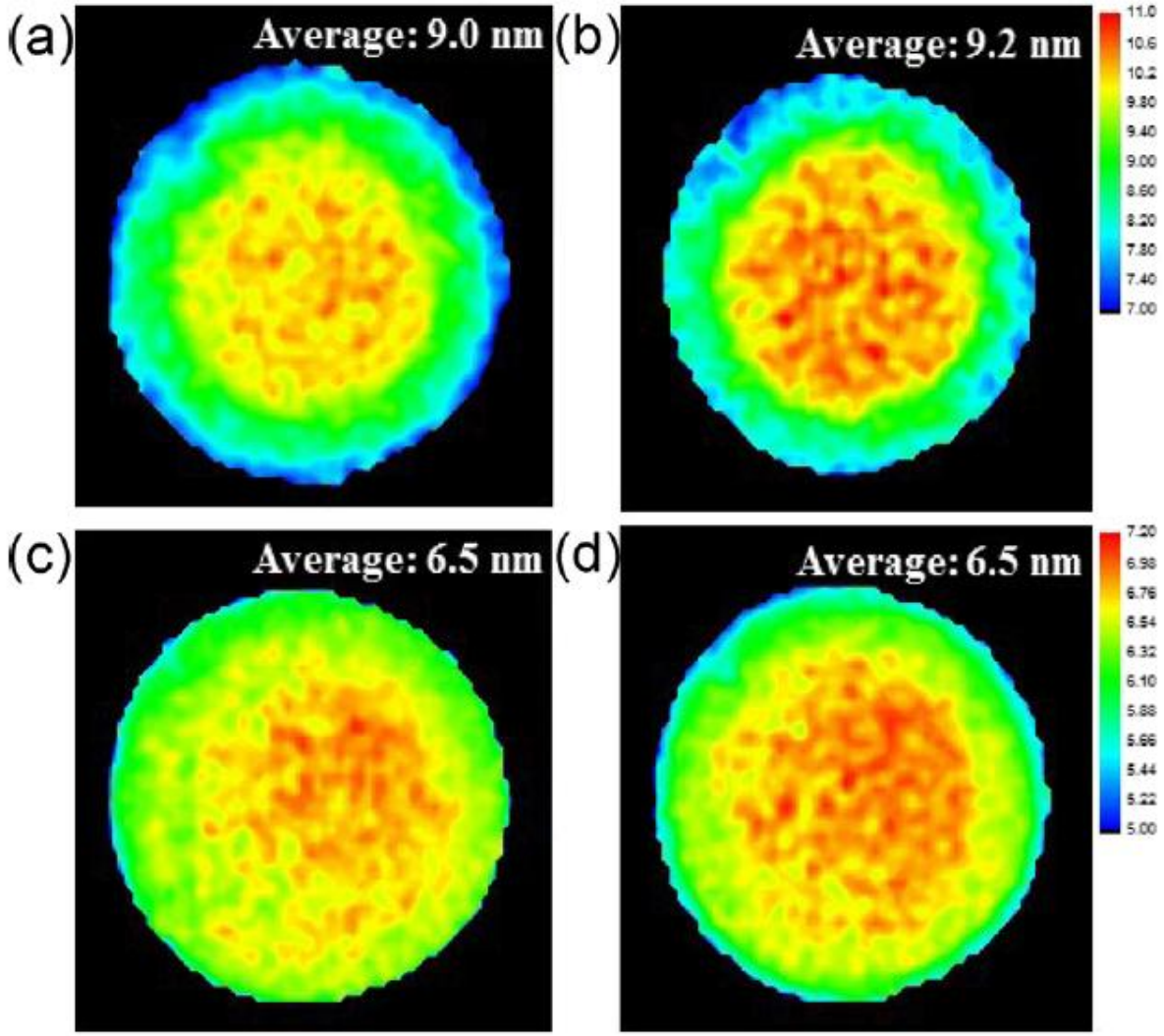
Co_3O_4 nanoparticle size distribution map recorded at 150 °C during reduction in H_2.

H2 reduction

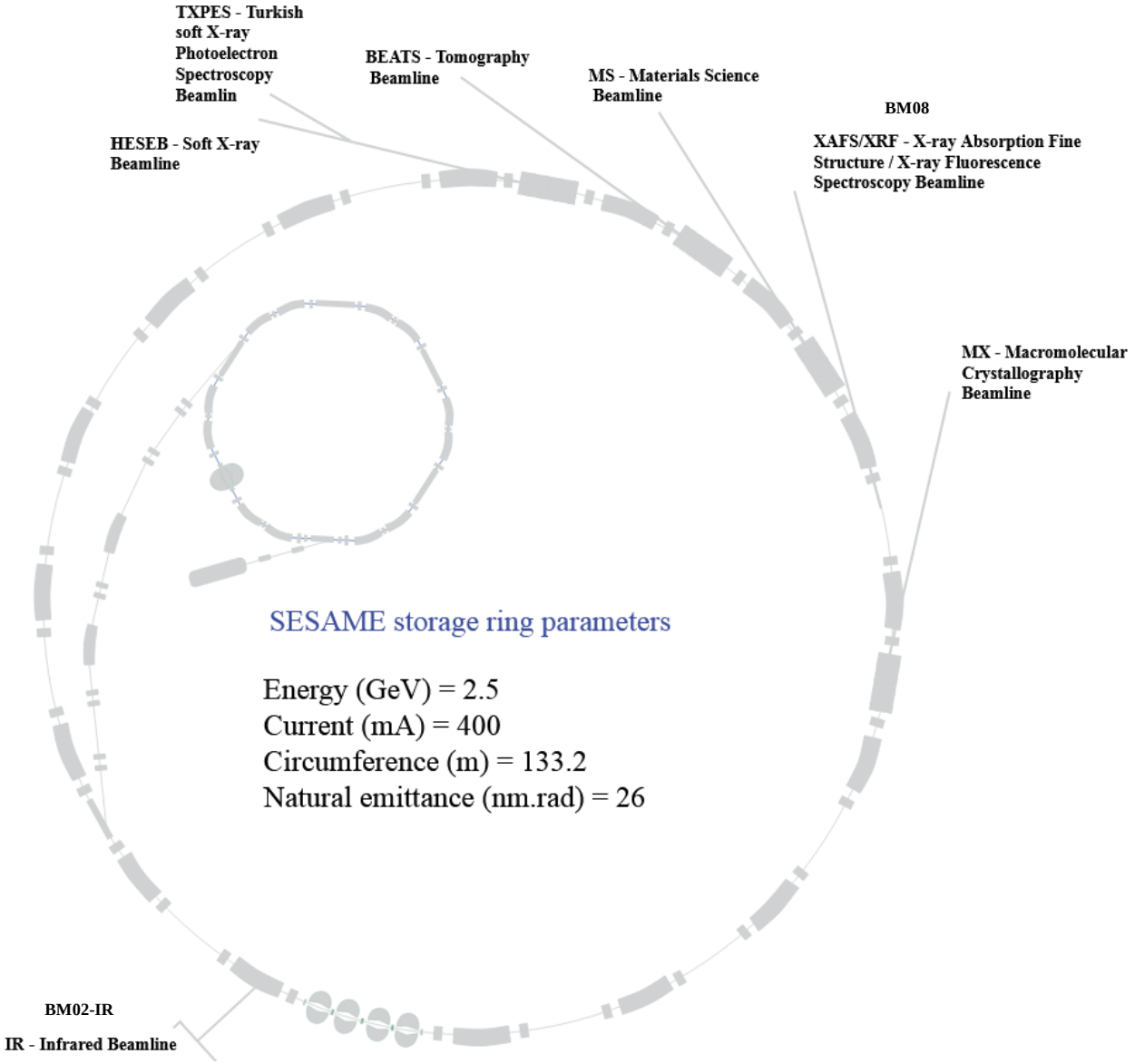
FTP

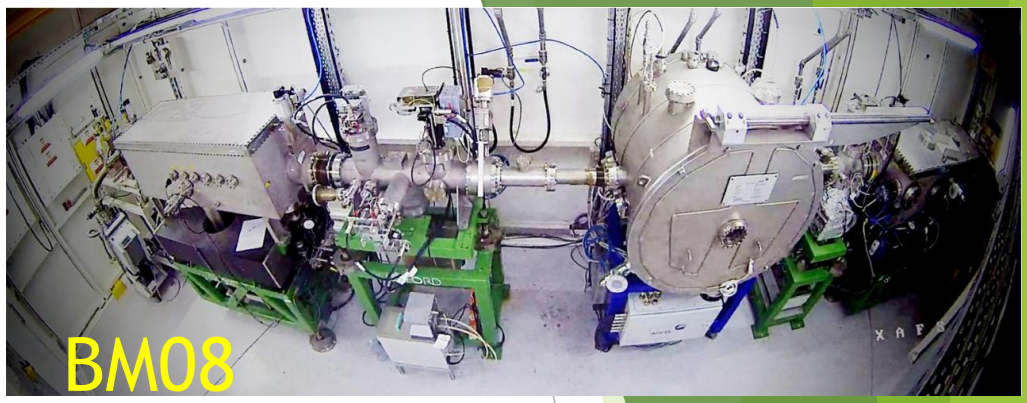
Co nanoparticle

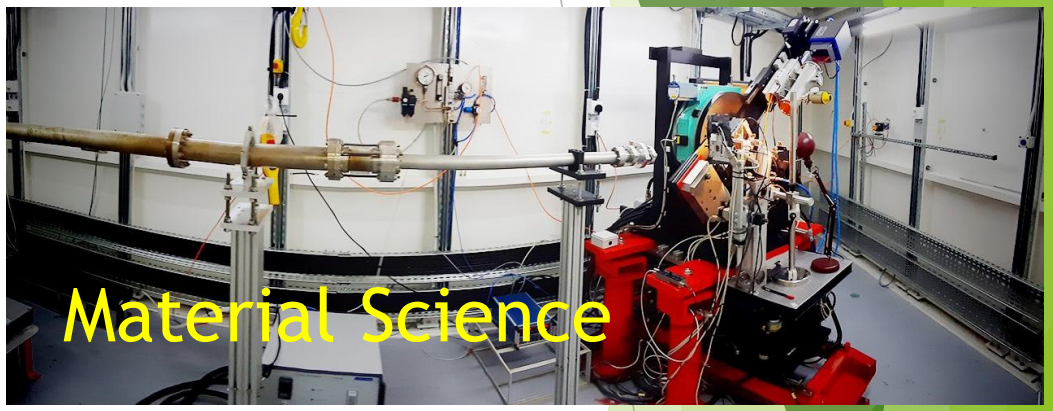
CoO nanoparticle



SESAME Beamlines

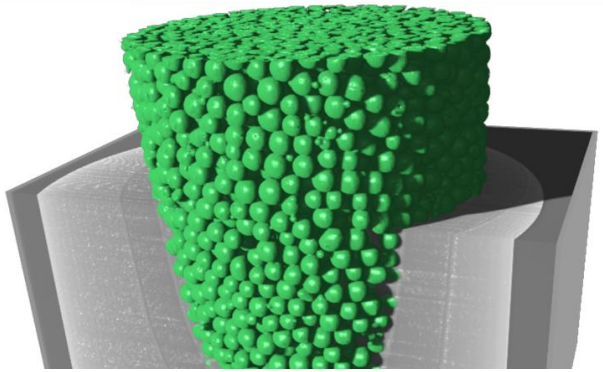

TXPES - Turkish soft X-ray Photoelectron Spectroscopy Beamlin
BEATS - Tomography Beamline
MS - Materials Science Beamline
BM08
XAFS/XRF - X-ray Absorption Fine Structure / X-ray Fluorescence Spectroscopy Beamline
HESEB - Soft X-ray Beamline
MX - Macromolecular Crystallography Beamline
SESAME storage ring parameters
Energy (GeV) = 2.5
Current (mA) = 400
Circumference (m) = 133.2
Natural emittance (nm.rad) = 26
BM02-IR
IR - Infrared Beamline


BM08


Material Science

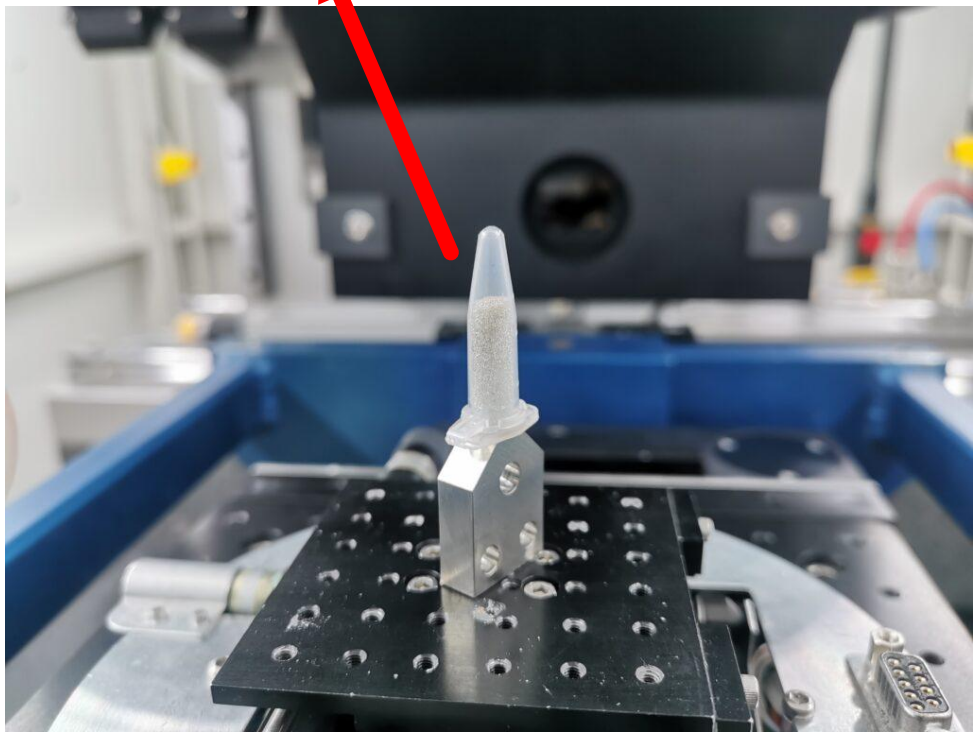

IR Beamline

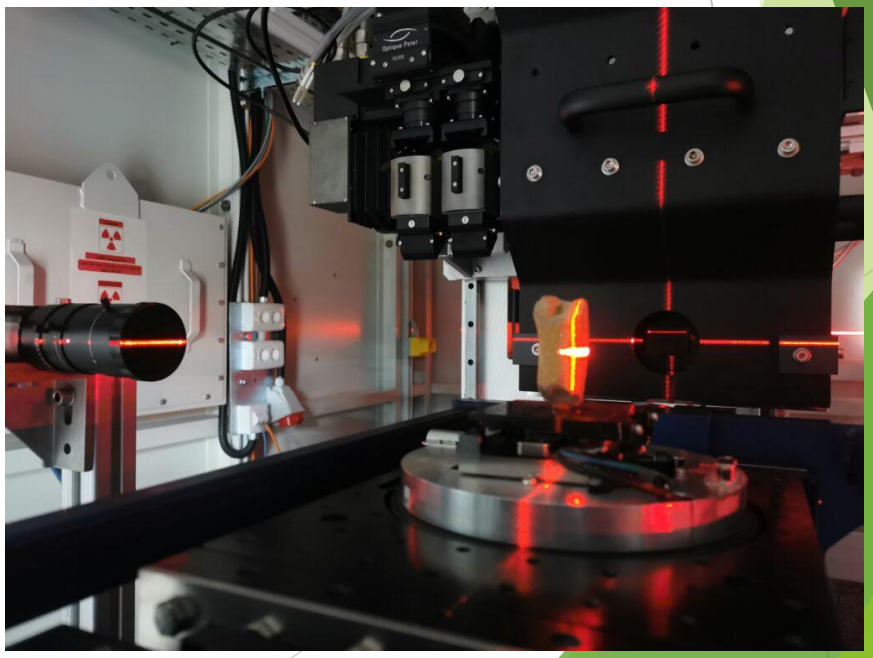
Beamline for Tomography at SESAME



First 3D tomography images from BEATS

News 24 May 2023





HESEB Helmholtz-SESAME Soft X-Ray Beamline for SESAME

This initiative was launched by the Helmholtz research centre DESY in Germany with the plan to build a new state-of-the-art beamline in the soft X-ray range at SESAME to be used for a large number of top-class scientific applications. The goal is to create new research opportunities at SESAME and to enhance the cooperation in photon science between German and European research groups and the SESAME members.

Within the four-year project (2019-2022), a Helmholtz consortium of five German research centres (DESY, FZJ, HZB, HZDR and KIT) has designed a high-performance beamline for soft X-rays and planned its realization at SESAME. The undulator photon source was developed and built by HZB, while the contract for the technical design, construction and installation of the beamline was awarded to the company FMB Berlin, which has already successfully built and installed several such beamlines at various facilities worldwide.

Thanks for your Attention