Research infrastructure assistance for research into circular materials and recycling processes

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ReMade @ARI

A hub for materials research

Overview

- ReMade@ARI project introduction
- ESRF, the European Synchrotron, Grenoble, France
- The ReMade opportunity
- Examples of synchrotron research relevant to the circular economy

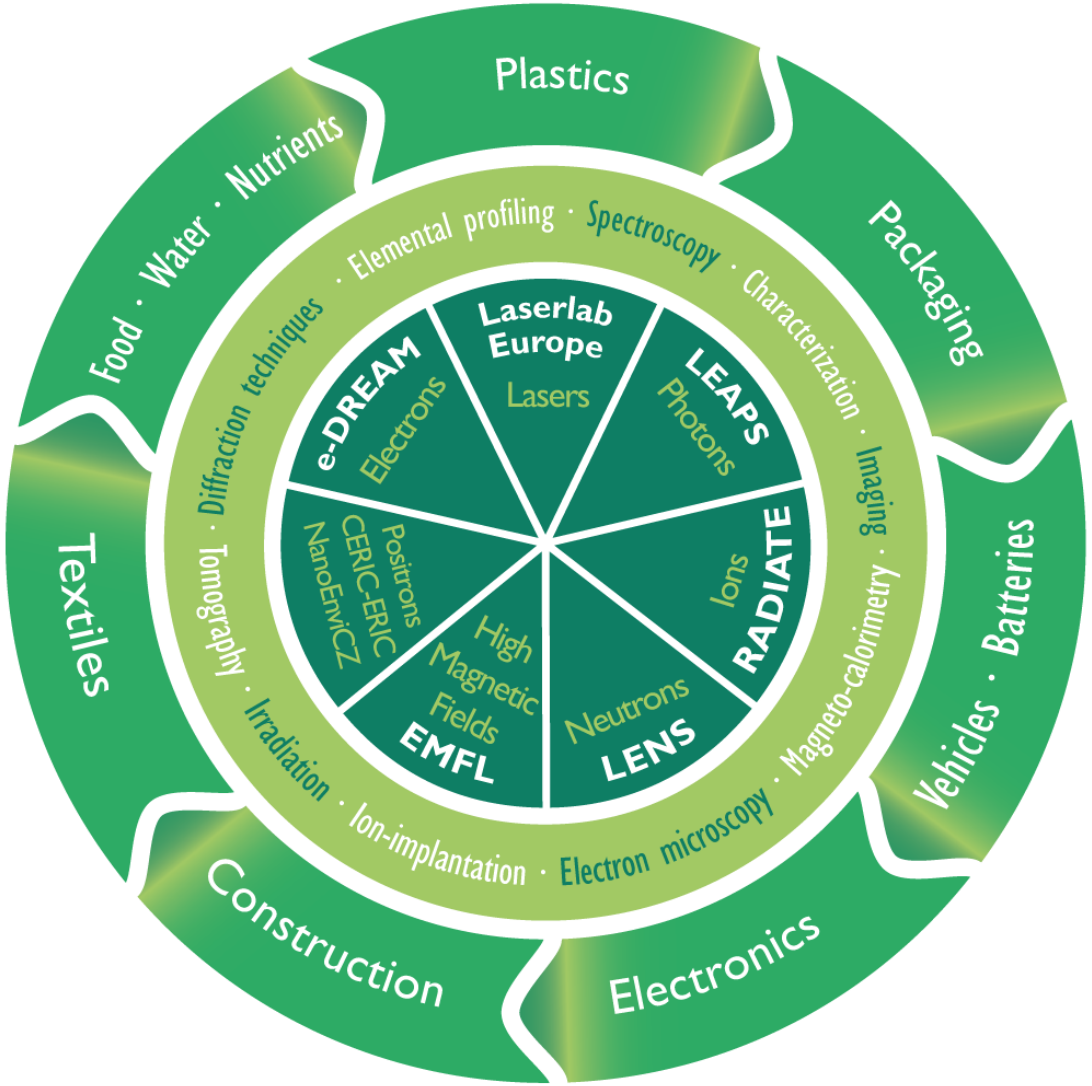
ReMade@ARI = Recyclable Materials Development at Analytical Research Infrastructures

CALL: HORIZON-INFRA-2021-SERV-01-04: Research infrastructures services enabling the development of materials for a circular economy

EU funding: 13.7 million EUR + 1.8 million EUR from Switzerland and UK

44 partners;
HZDR is coordinator;

Started on 01 September 2022; Duration 4 years
First ReMade call opened: 03/03/2023
Next TNA deadline: 31/10/2023



Responding to The European Green Deal: **Circular Economy Action Plan**
https://environment.ec.europa.eu/strategy/circular-economy-action-plan_en

ReMade@ARI: Partners

- 48 Analytical research infrastructures from the ARIE network (arie-eu.org) comprising **synchrotrons, neutron sources, lasers, electron microscopes, ion and positron beams, and high magnetic field facilities.** Complementary facilities are provided by CERIC-ERIC, Heyrovský, pELBE and p@FRMII.
- Danish Technological Institute
- University of Bonn

ARIE is a hub of RI networks

- Accelerator-based light sources - *League of Accelerator-based Photon Sources (LEAPS)*
- Ion beams – *RADIATE*
- Laser light sources – *Laserlab-Europe*
- Electron microscopes - *e-DREAM*
- Neutron sources - *League of Advanced European Neutron Sources (LENS)*
- High magnetic field laboratories - *European Magnetic Field Laboratory (EMFL)*
- Proton beams – *INSPIRE*

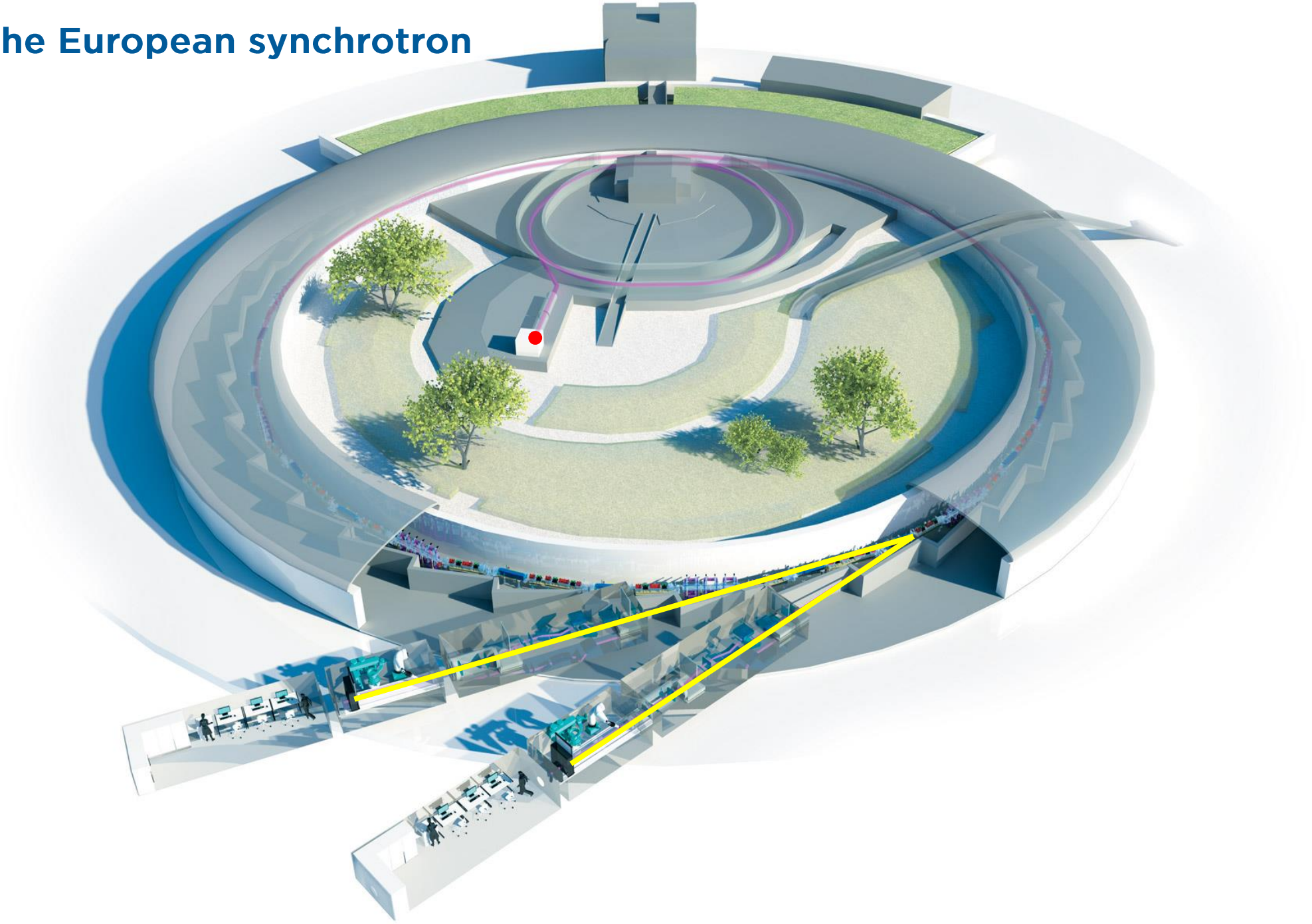
Analytical Research Infrastructures in Europe: **arie-eu.org**

Free access and assistance for use of the research infrastructures

- Open to academic researchers and industry
- 50,000 hours of trans-national access (TNA) at >50 facilities
- Provides access to materials characterisation or complex experiments for materials of recycling process development
- Ideal for first time users of RIs or to test complementary techniques
- Guidance from facility experts on project feasibility and selection of techniques
- One application can request multiple techniques at multiple facilities
- Support beyond the usual service offer of the RIs (18 junior scientists)
- Additional tailored access for industry
- Opportunity for users from countries with few national facilities to access a large range of RIs

About the ESRF

ESRF: The European synchrotron



ESRF: established by an inter-governmental convention signed by 13 member states

21 PARTNER COUNTRIES

13 Member states:

France	**27.5%**
Germany	**24.0%**
Italy	**13.2%**
United Kingdom	**10.5%**
Russia	**6.0%**
Benesync	**5.8%**
(Belgium, The Netherlands)	
Nordsync	**5.0%**
(Denmark, Finland, Norway, Sweden)	
Spain	**4.0%**
Switzerland	**4.0%**

8 Scientific associate countries:

Israel	**1.75%**
Austria	**1.75%**
Poland	**1.0%**
Portugal	**1.0%**
India	**0.66%**
Czech Republic	**0.6%**
South Africa	**0.3%**
Hungary	**0.25%**

Budget: ~ 100 M€ per year
700 staff
44 beamlines
9000 scientific visits per year
2000 publications per year
30% of research is with industry

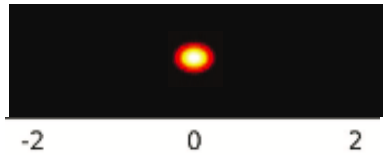
ESRF-EBS – First in a new generation of high-energy synchrotrons

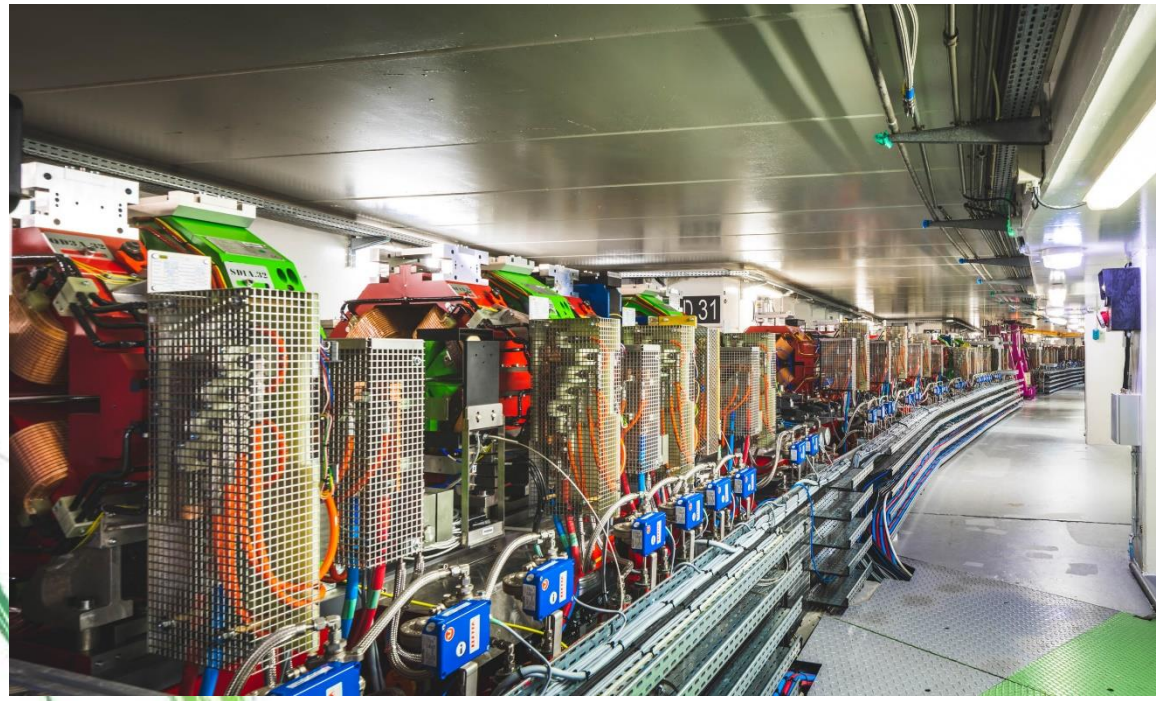
ESRF-EBS
Extremely Brilliant Source

- **Opened to users 25/08/2020**
- **Investment of 150 MEUR**
- **Fourth generation source + new beamlines + data infrastructure**

3rd generation - emittance H/V ~ 1000

4th generation – H/V ~ 1

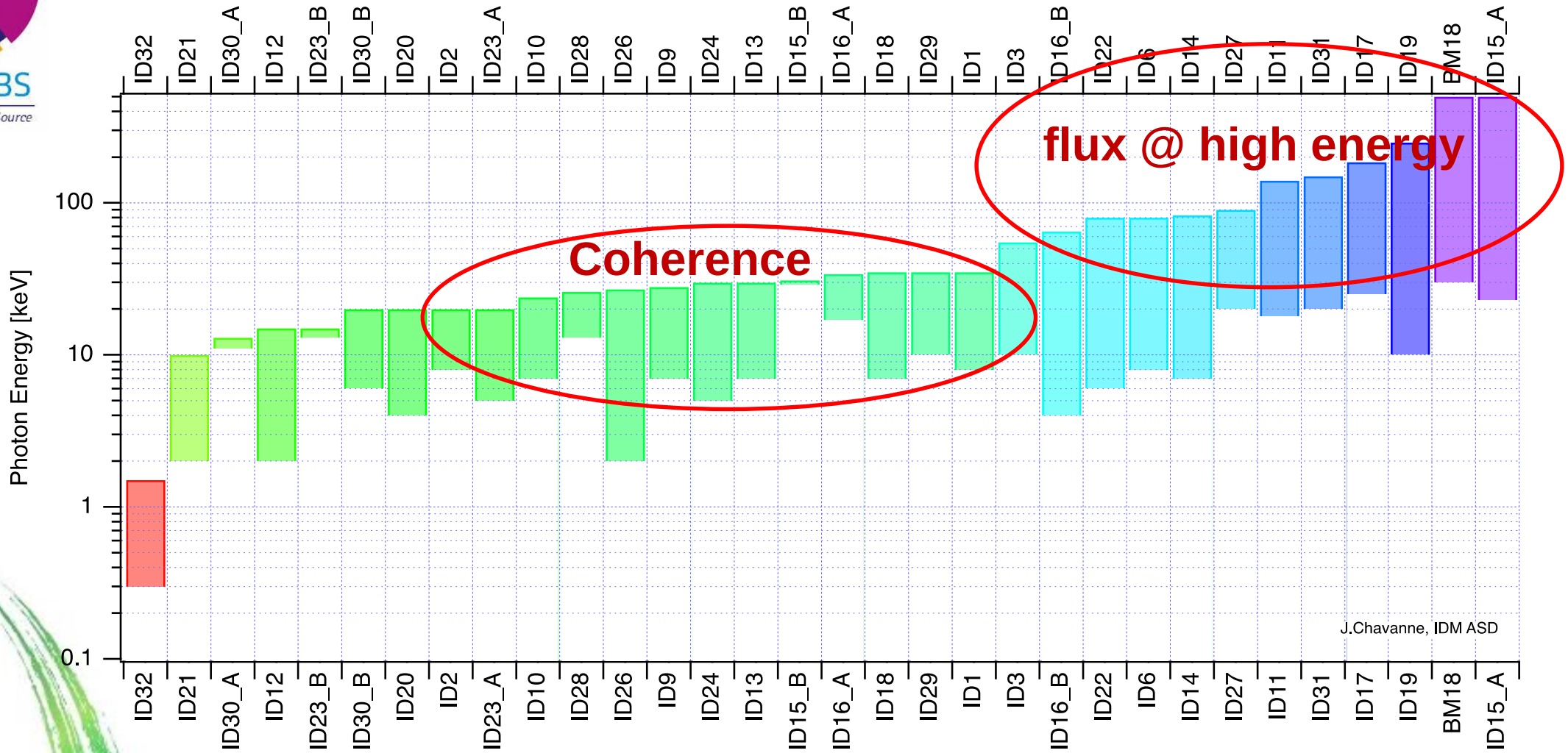




ESRF
The European Synchrotron

ESRF-EBS – First in a new generation of high-energy synchrotrons

- **Brilliance *100 and coherence *30**
- **Opportunity for new experiments**
- **Faster experiments / more data**

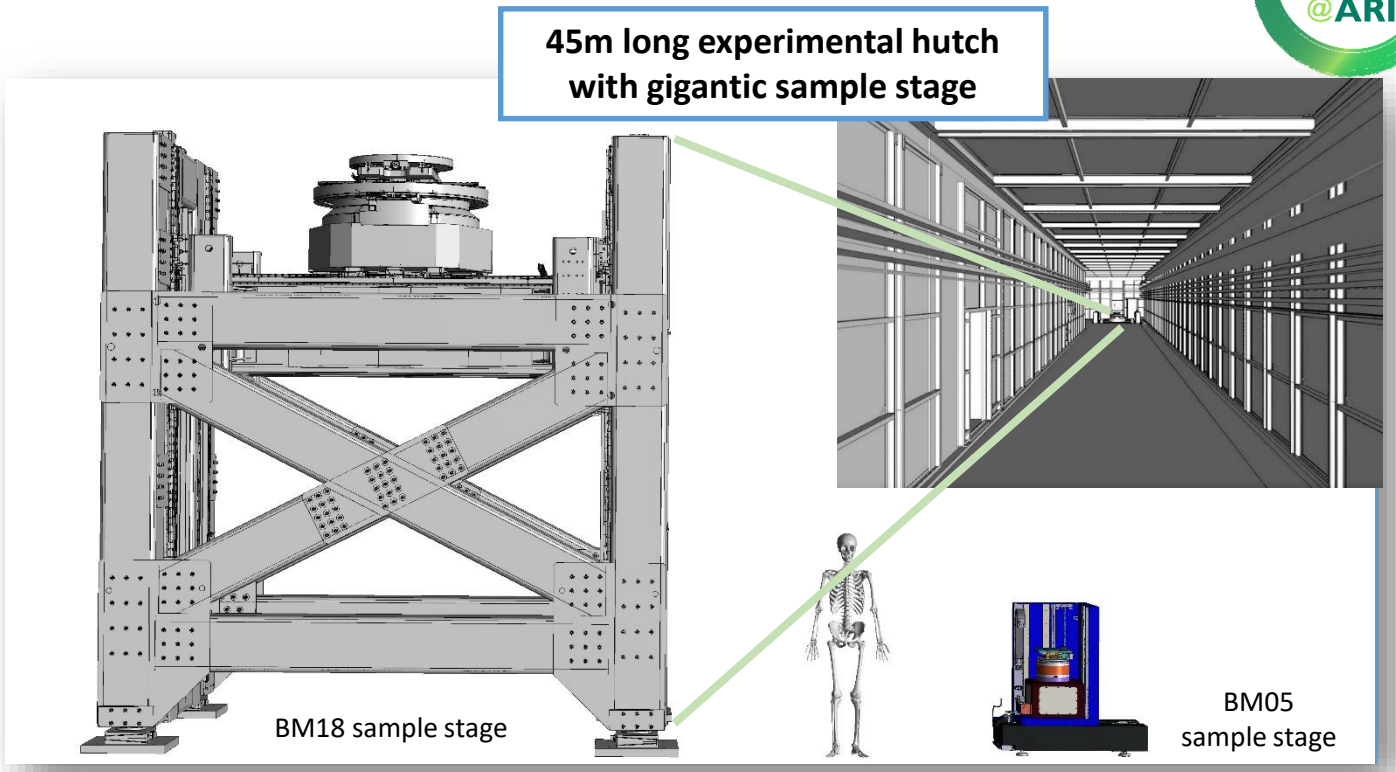


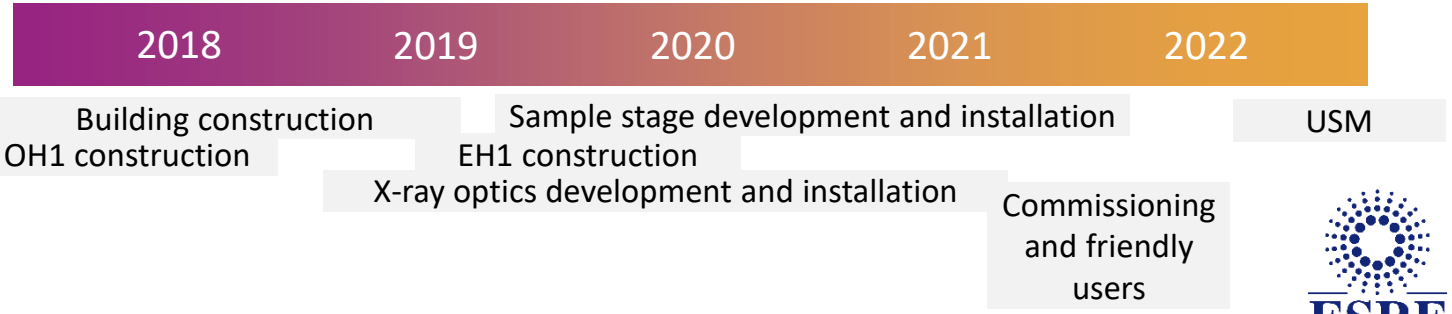
BM18 hierarchical phase-contrast tomography - Project coordinator: Paul Tafforeau

MAIN TECHNIQUES
- Hierarchical tomography
- Propagation phase-contrast imaging

BEAMLINE SPECIFICATIONS
- Energy range: 25-350 keV (polychromatic)
- 220m long beamline, up to 38m for propagation phase-contrast
- Sample size up to 2.5m and 300 kg
- High level of automation and high throughput

EBS & REFURBISHMENT IMPROVEMENTS
- Smallest possible X-ray source of the EBS
- Beam of 35cm with highest coherence worldwide for high-energy X-ray imaging.
- Large resolution range (0.7 - 200 µm)





BM18 hierarchical phase-contrast tomography - Project coordinator: Paul Tafforeau

Biomedical imaging

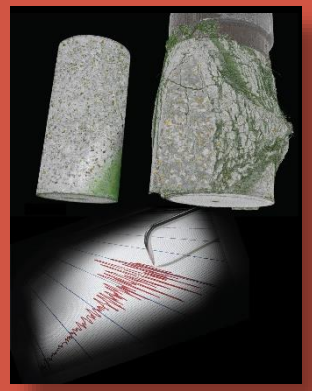
- A new scale in human body knowledge
- Understanding effects of diseases

Natural and cultural heritage

- Understanding the evolution of life on earth
- Non-invasive structural study of archaeological specimens and art pieces

High sensitivity phase-contrast tomography in large and complex samples

Geology



- Origin of earthquakes
- Mechanisms of volcanoes
- Climate change

Material sciences

- Non-destructive control of large devices (batteries, complex mechanical parts)
- Additive manufacturing (in-situ and ex-situ)

Industrial applications

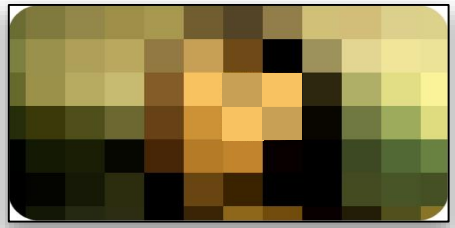
- Testing high-value objects
- Analysis of 3D structures of industrial products
- Industrial processes

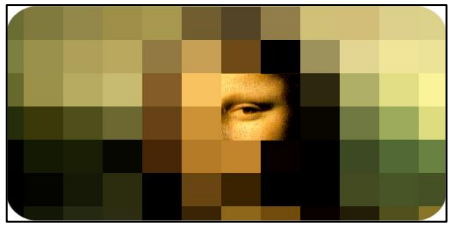
ESRF: A powerful microscope

Look inside

2D or 3D

See the details





micron to nm, imaging and spectroscopy

High speed

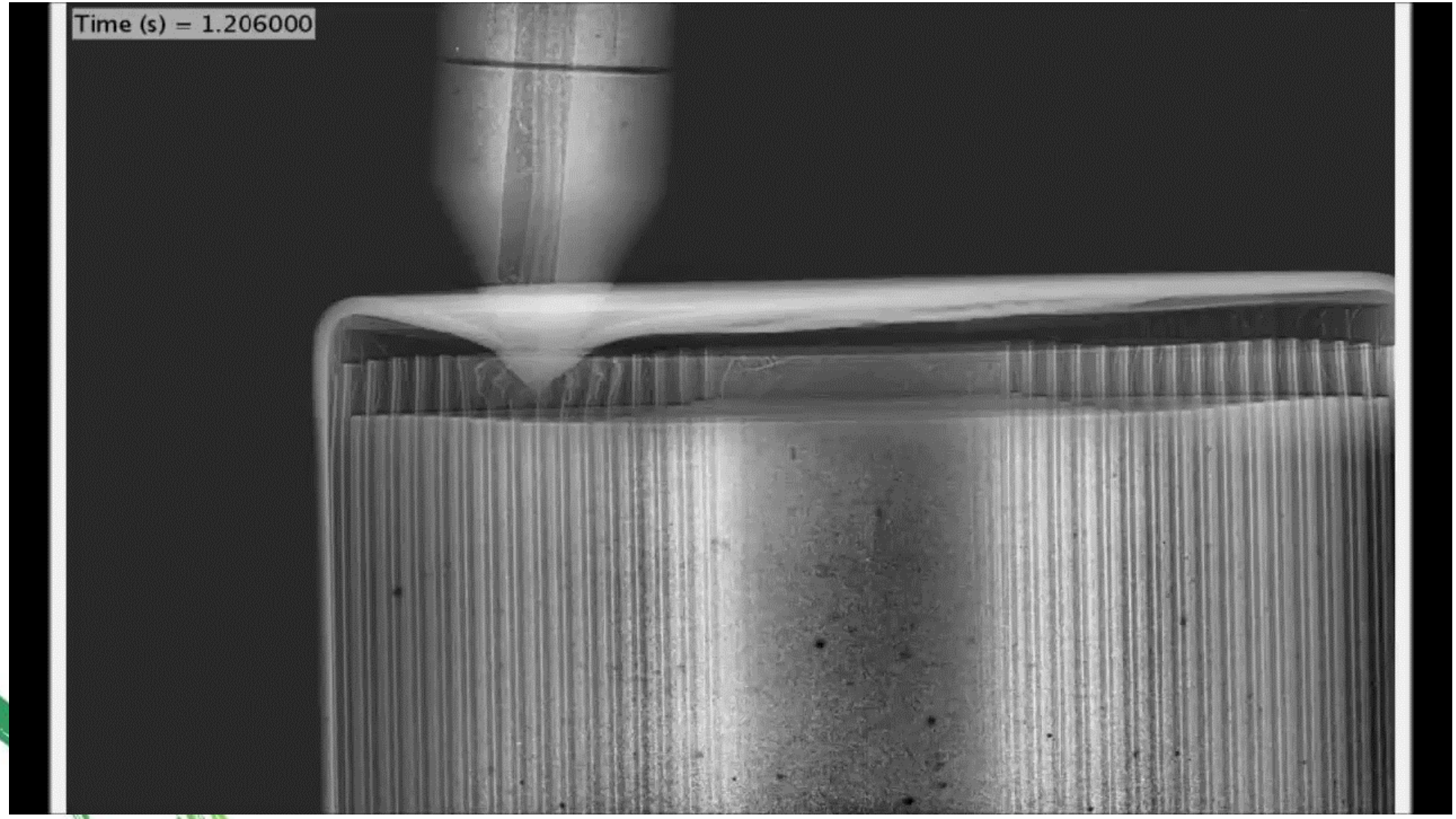
s, ms, … fs

Under real conditions

In situ, operando

Example of fast imaging: Li-ion battery thermal runaway

kHz radioscopy with 75 keV X-rays



Beamline ID19
Polychromatic beam
Peak: 75 keV
pco. dimax, 1250 fps
LuAG: Ce

D. P. Finegan, et al, Identifying the cause of rupture of Li-ion batteries during thermal runaway Advanced Science, vol. 5, no. 1, 1700369 (2018)

Beamline ID19

Example of high-throughput: X-ray fluorescence (XRF) trace element analysis

STREAMLINE

3500 samples
~ 2 days of autonomy

- System designed in collaboration with the University of Montpellier
- For use in academic research or as a service to industry
- Production tested for a client in the mining sector using a series of several thousand samples
- Ready for use and its first commercial access was through the University of Montpellier
- Expect future customers in raw materials

Beamline BM23

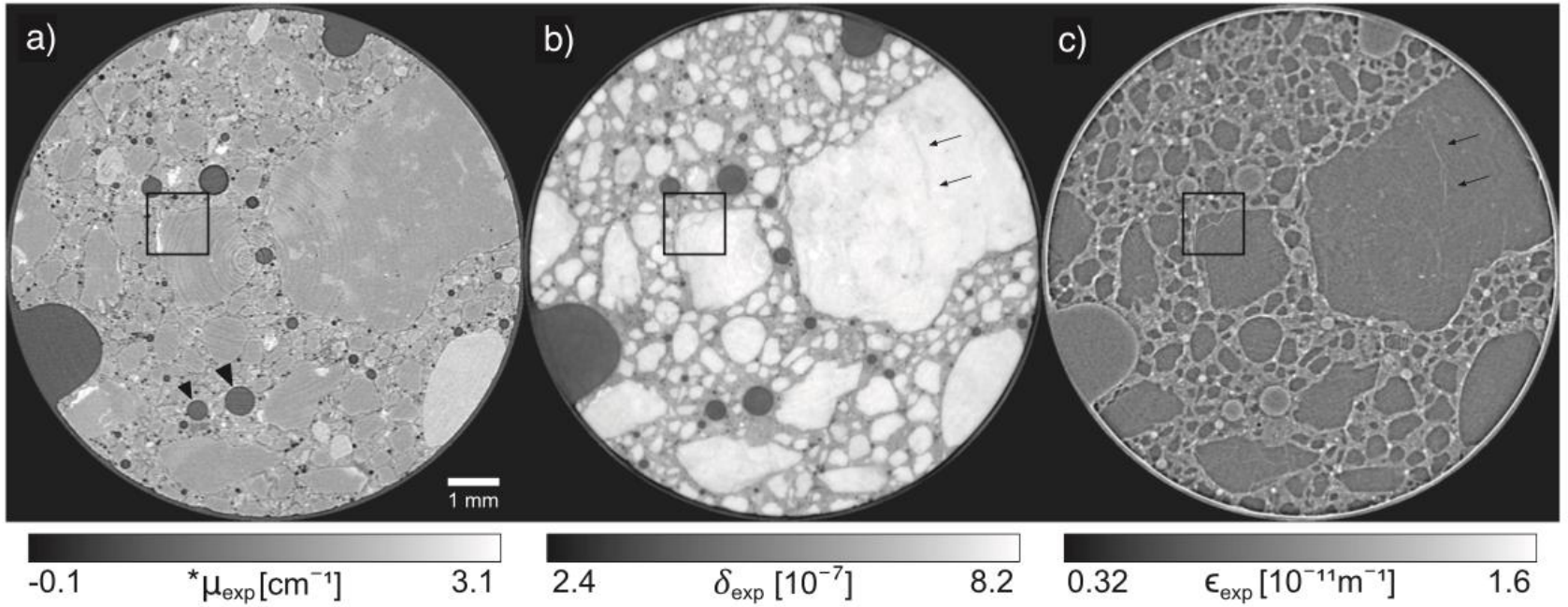
ESRF
The European Synchrotron

ESRF techniques available within ReMade

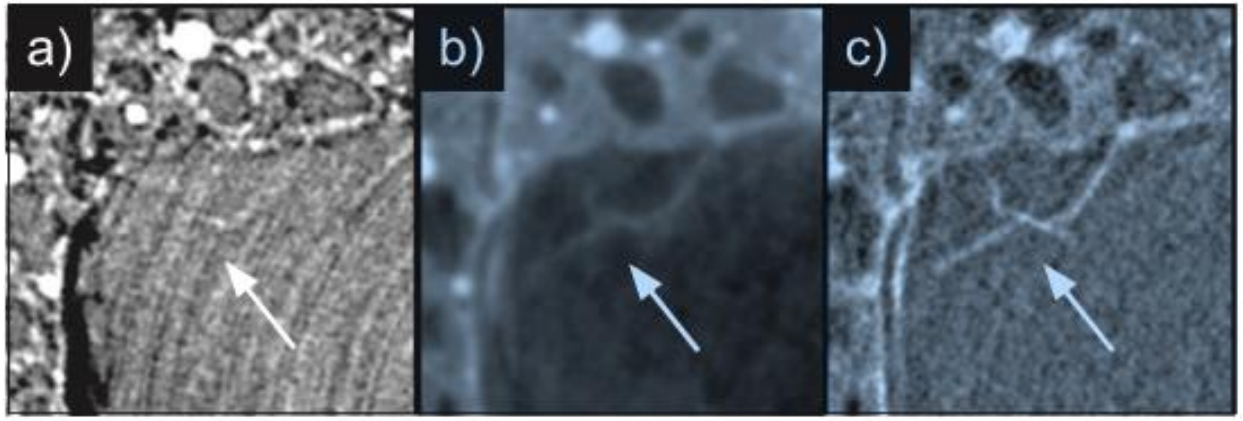
ESRF techniques available within ReMade: 1. Tomography

- 3D imaging of materials including *in situ* visualisation of processes; quantitative with exceptional contrast for both soft and hard materials using phase contrast imaging.
- Beamlines BM05, ID19
- Other beamlines are possible on request

Proof of concept of X-ray grating interferometry on a concrete sample



Attenuation-contrast, phase-contrast, and dark-field contrast axial slices from a tomographic reconstruction of a concrete sample. A. Sarapata, M. Ruiz-Yaniz, I. Zanette, A. Rack, F. Pfeiffer, and J. Herzen, Multi-contrast 3D X-ray imaging of porous and composite materials, Applied Physics Letters 106, 154102 (2015); https://doi.org/10.1063/1.4918617

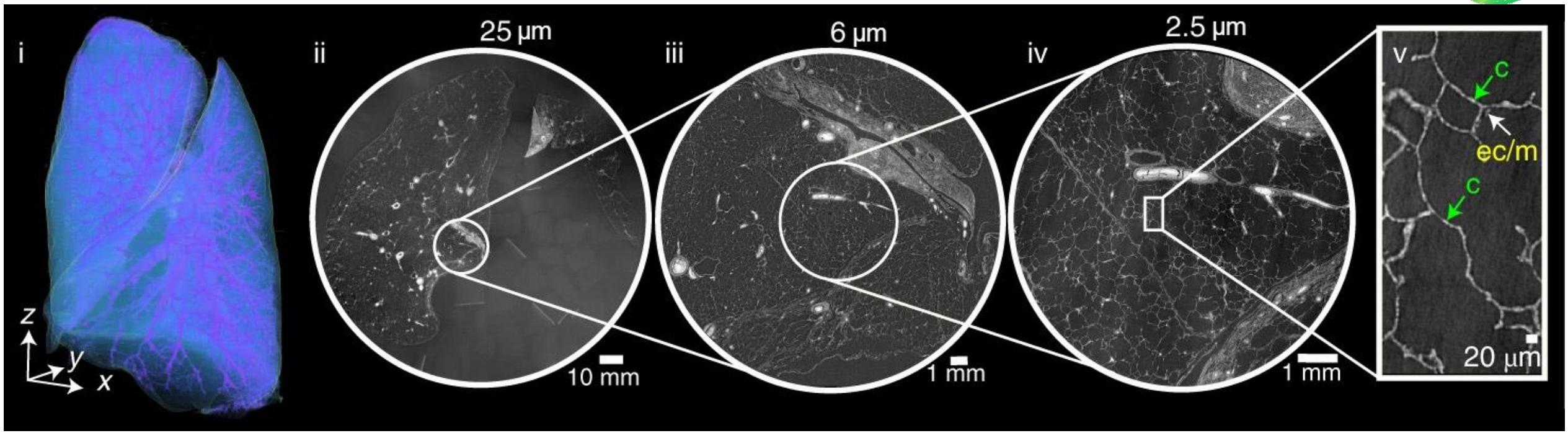


Beamline ID19

TABLE I. Contrast-to-noise ratios for attenuation- and phase-contrast reconstructions.

	CNR	
	Attenuation	Phase
air–cement	1.7	4.6
air–aggregate	3.6	25.8
cement–aggregate	0.3	6.9

Imaging Covid-19 damage to lung

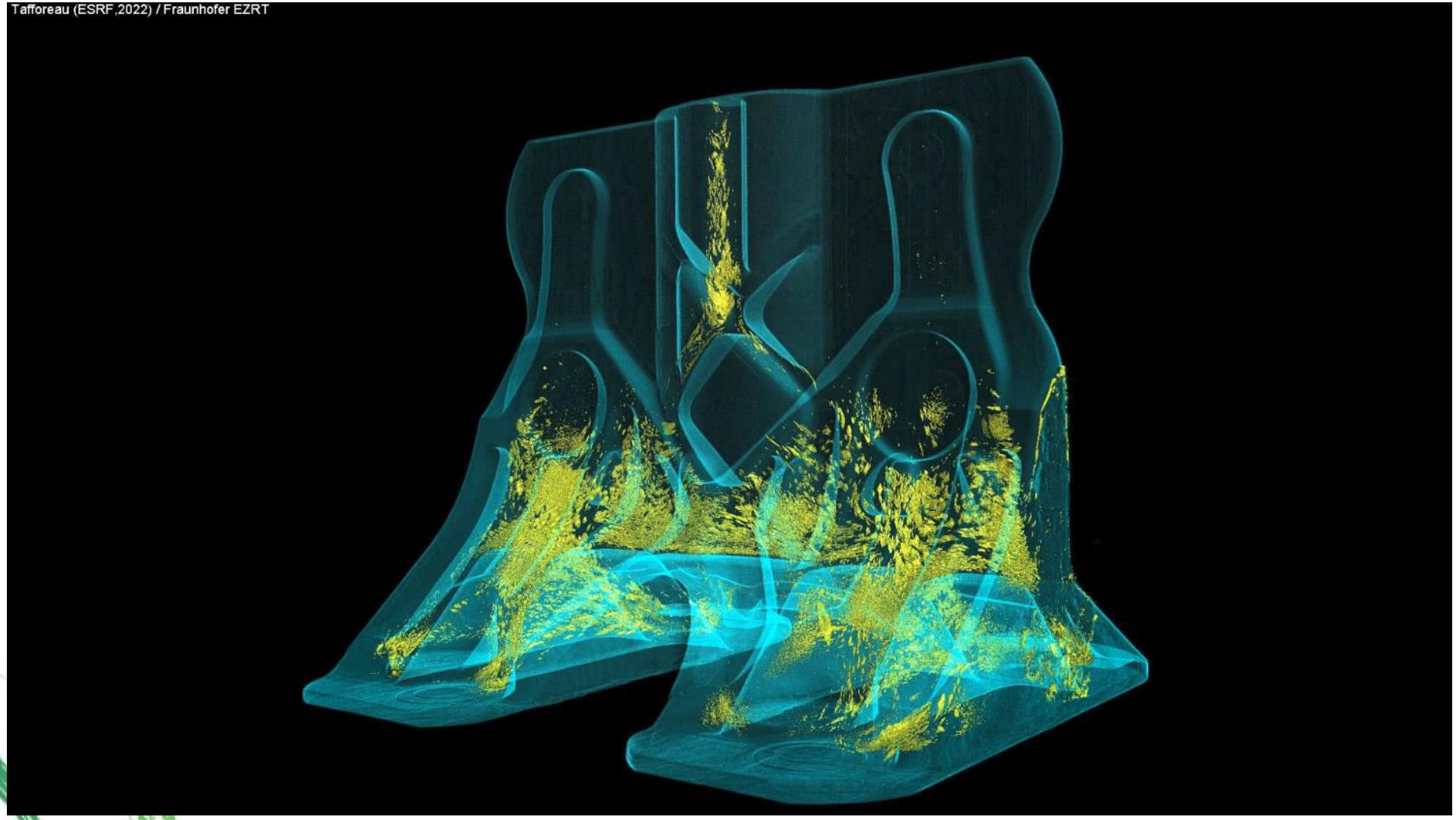


Anatomical to cellular: Hierarchical phase-contrast tomography (HiP-CT) of the lung (BM05), using 25 µm to 2.5 µm per voxel (c, blood capillary; ec/m, epithelial cell or macrophage).

Walsh, C.L., Tafforeau, P., Wagner, W.L. et al. Imaging intact human organs with local resolution of cellular structures using hierarchical phase-contrast tomography. Nat Methods (2021). https://doi.org/10.1038/s41592-021-01317-x

Beamline BM05

Tomography of a carbon fibre composite made by squeeze casting



Scanned at BM18 at resolutions from 42 µm down to 0.60 µm voxel pitch

Able to visualise pores in structure and carbon fibres of 6 µm diameter.

Credit: Paul Tafforeau (ESRF) and Simon Zabler (Fraunhofer EZRT); T-Bracket has been supplied by Fiber Institute Bremen (FIBRE)

Beamline BM18

Tomography of a carbon fibre composite made by squeeze casting (video)

Scanned at BM18 at resolutions from 42 µm down to 0.60 µm voxel pitch

Able to visualise pores in structure and carbon fibres of 6 µm diameter.

Credit: Paul Tafforeau (ESRF) and Simon Zabler (Fraunhofer EZRT); T-Bracket has been supplied by Fiber Institute Bremen (FIBRE)

Beamline BM18

ESRF techniques available within ReMade: 2. Diffraction based

- High resolution XRD, pair distribution function determination, high-throughput XRD and PDF, grain mapping, scanning microdiffraction, diffraction contrast tomography, combined SAXS-WAXS, and all possible *in situ* and *operando*.

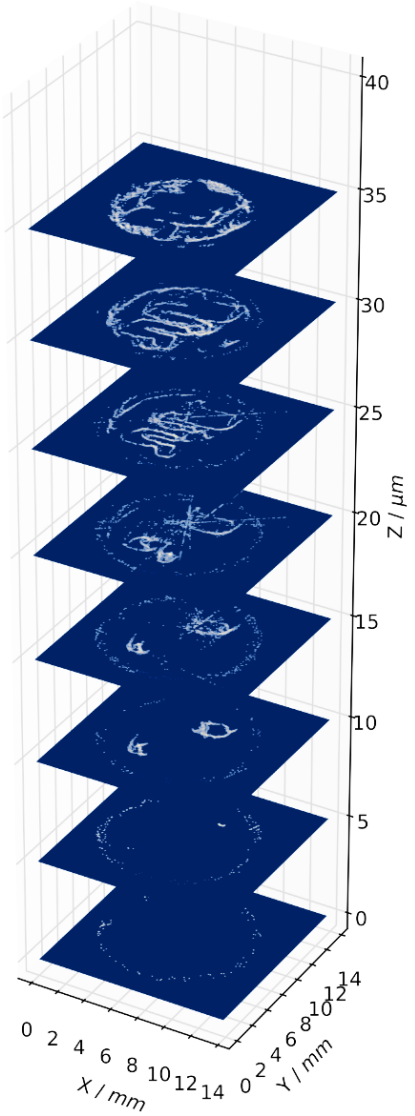
Beamlines:

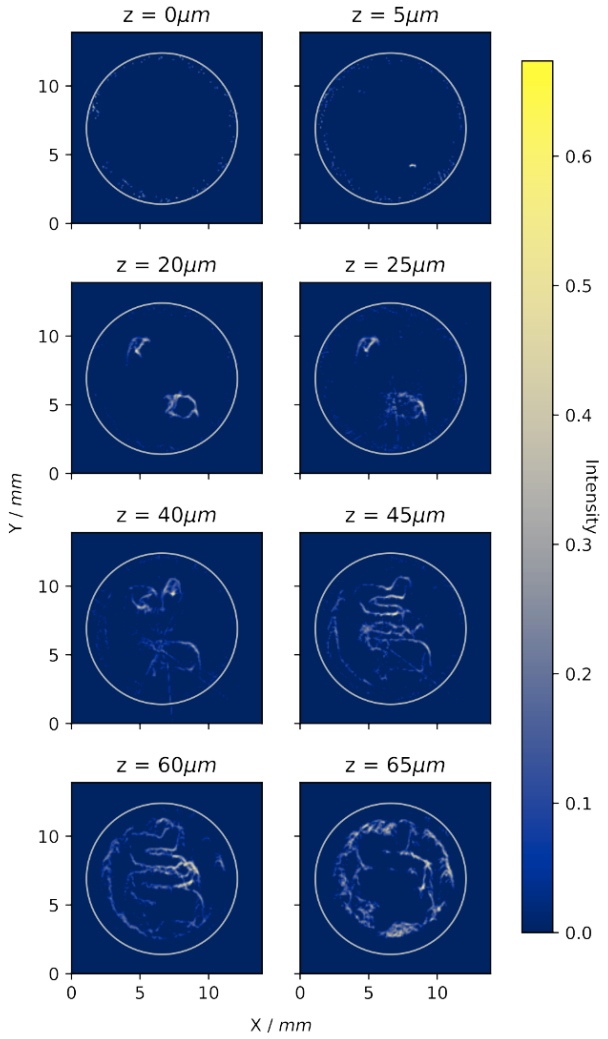
- ID11 (diffraction and imaging studies)
- ID15A (scattering (XRD) tomography)
- ID22 (powder diffraction)
- ID31 (High-energy XRD, SAXS/WAXS, *operando* studies)
- ID01 (reciprocal space mapping),
- ID13 (nano-XRD; SAXS)
- Other beamlines are possible on request

Operando diffraction tomography of an electrolyser

XRD-CT scan of a copper catalyst layer inside a reactor used for CO_2 recycling to artificial fuels.

“Coupling the WAXS/SAXS techniques with tomography reconstruction spans most of the length scales relevant for electrolyser operation in one measurement, allowing true *operando* experiments.”





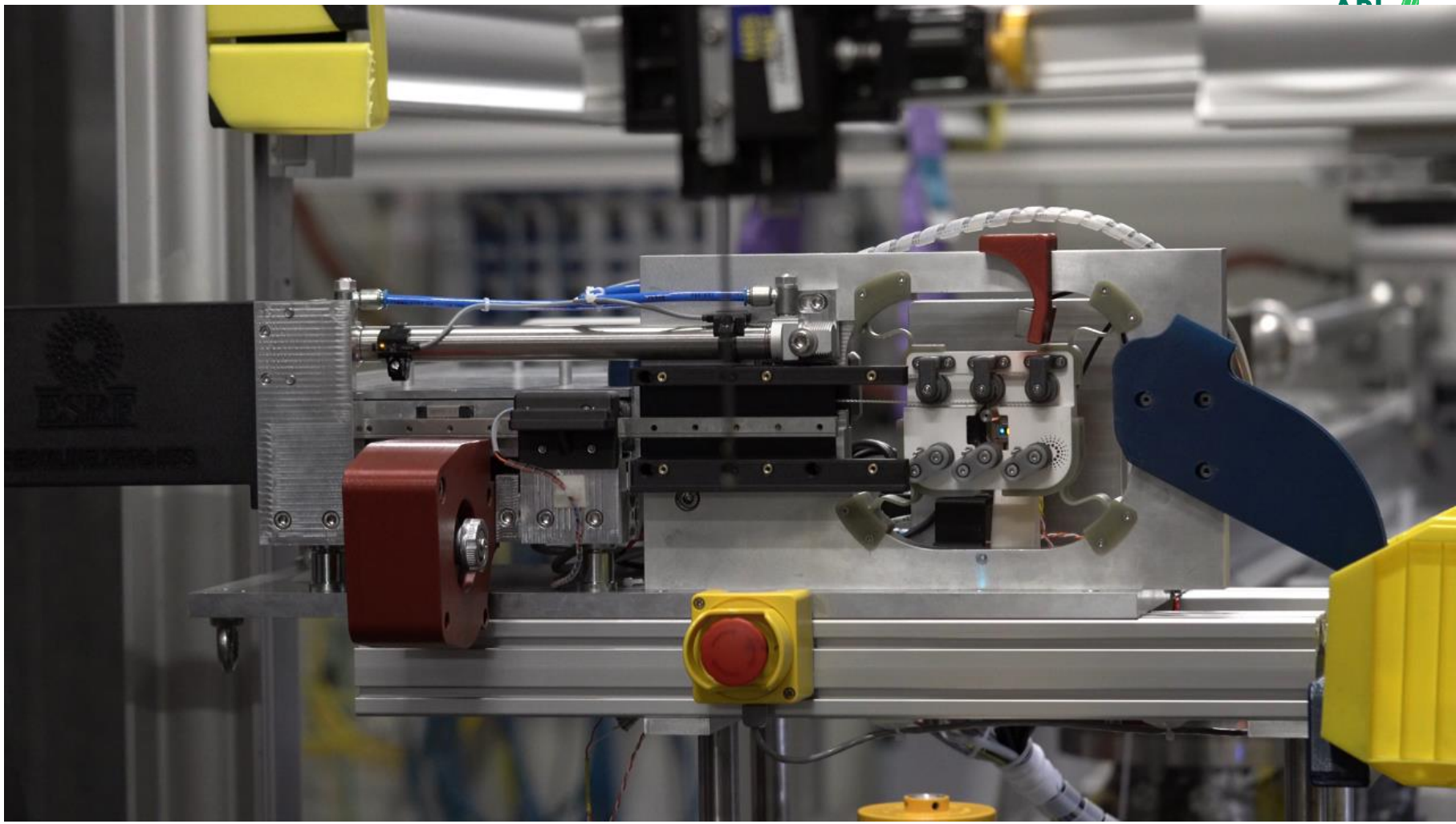
XRD-CT scan of the gas diffusion electrode’s copper catalyst layer before CO_2RR has taken place. The plotted intensity correspond to the surface oxide (Cu_2O) XRD peak (111), integrated in the range of q = 2.49 $Å^{-1}$ and q = 2.59 $Å^{-1}$. The dimensions of one voxel is 69.5 μm and the dimensions of one slice is 200 × 200 voxels (13.9 × 13.9 mm^2). Scan time: about 20 min/slice. Moss *et al.*, Journal of Power Sources, 2023, 562, 232754. DOI: 10.1016/j.jpowsour.2023.232754.

Beamline ID31

High throughput powder diffraction service for battery materials research and recycling

Developed in collaboration with BASF

- uses a commercial sample holder
- max. capacity 66 sample holders = 1056 samples
- service in place Jan. 2023



Commissioning test: **1056 samples: ~22 minutes**

- measuring time: 1s; time to change sample & read QR-code with sample descriptor: 0.095s

Beamline ID31

The ReMade@ARI Opportunity

The ReMade opportunity

Three access tracks:

1. ReMade-TNA: For academia and industry (applications open, next deadline 31/10/2023)
2. ReMade-SME: For small and medium enterprises (applications open, continuous)
3. ReMade-IND: For industry in collaboration with a knowledge provider, e.g. CEA, Fraunhofer, etc. (call opens in July, next deadline 30/11/2023)

Send us your idea
via a **Pre-Proposal**
(no deadline, open all year)

Submit your Proposal
via our **Portal**
(current deadline 30 April 2023)

ReMade - SME

Coordinated and **easy access** for SMEs to ARI facilities

To enhance the **circular economy** innovation process

ReMade - IND

Joint access for **Industry** and **knowledge providers**

For enhanced industry exploitation of ARI facilities to tackle more **complex challenges**

remade-project.eu

1. ReMade-TNA (Trans-national access)

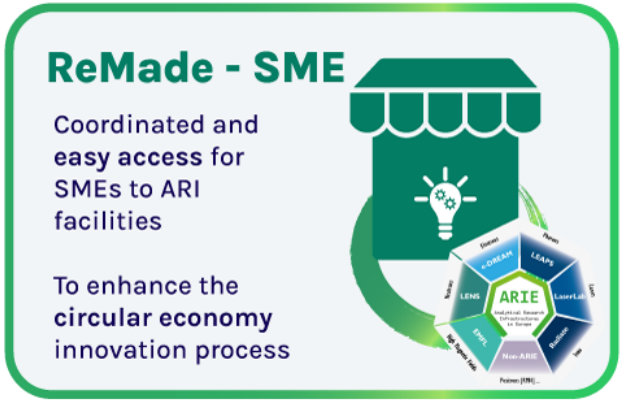
- Two calls for proposals per year; second call open, deadline 31/10/2023
- For academia and industry
- Up to three techniques can be requested by one proposal
- Access is free and travel expenses are reimbursed by ReMade
- Submit a **pre-proposal** for feasibility advice and guidance on the best techniques to use
- Submit a **proposal** if you already know about techniques
- Assistance by junior scientists during an experiment
- Data-analysis is by the user
- Training can be requested (e.g. before proposal; after data collection)
- This is EC funded TNA: obligation to request a facility in a different country; obligation to report / publish results (2-6 years embargo on data)
- Many experiments can be mail-in/remote access

Send us your idea
via a **Pre-Proposal**
(no deadline, open all year)

Submit your Proposal
via our **Portal**
(current deadline 30 April 2023)

2. ReMade-SME: for small and medium enterprises

- Rolling call for proposals began 24 April 2023
- Guidance from RIs industry contact offices
- Beamtime is free* (*accepted by most partners!)
- Assistance with both experiment and data analysis
- Priority for rapid experiments and materials characterisation by mail-in services
- Ideal test experiment before a more elaborate ReMade-IND project
- Confidential: no obligation to publish results
- You can work with your local facility
- => contact direct with industry contact offices



industry@remade-project.eu

3. ReMade-IND: for industry with support from a knowledge provider

- Two call for proposals: July to November 2023; second call in 2024
- Guidance from RIs industry contact offices
- ReMade can help to find a knowledge partner, if needed
- Funding (30 kEUR) to cover experiment planning; sample environment development; travel, data analysis by KP
- Free RI access or charged to industry (facility dependent)
- Standard RI support for experiment
- Can be confidential, no obligation to publish results if industry pays for beamtime; not TNA so use any facility
- Can be combined with ReMade-TNA (obligation to publish)
- => contact direct with industry contact offices

DTI is a project partner.
Other RTO's are also invited to participate.

industry@remade-project.eu

Industry contact offices

ALBA-CELLS	**FORTH**	**HZDR/pELBE**
https://www.cells.es/en/industry/services	https://www.forth.gr/en/home/	https://www.hzdr.de/db/Cms?pOid=25496&pNid=1
industrialoffice@cells.es	pissas@iesl.forth.gr	e.hirschmann@hzdr.de
CERIC-ERIC	**FZJ**	**ILL**
https://www.ceric-eric.eu/industry/services-	https://er-c.org	https://www.ill.eu/industry
angela.zennaro@ceric-eric.eu	er-c@fz-juelich.de	industry@ill.eu
CMAM-UAM	**HEREON**	**ISIS**
https://www.cmam.uam.es/	https://www.hereon.de/index.php.en	https://www.isis.stfc.ac.uk/Pages/home.aspx
gaston.garcia@uam.es	marc.thiry@hereon.de	graham.appleby@stfc.ac.uk
DTI	**HiLASE**	**MAXIV**
https://www.dti.dk/	https://www.hilase.cz/en	https://www.maxiv.lu.se/industry/
vla@teknologisk.dk	Bo-Anne.Rohlik@hilase.cz	magnus.larsson@maxiv.lu.se
ELETTRA	**Heyrovský**	**SOLEIL**
https://www.elettra.eu/technology/industry/e	https://www.jh-inst.cas.cz	https://www.synchrotron-soleil.fr/en/industry
ilo@elettra.eu	martin.kalbac@jh-inst.cas.cz	industrie@synchrotron-soleil.fr
ESRF		
https://www.esrf.fr/Industry		
remade@esrf.eu		

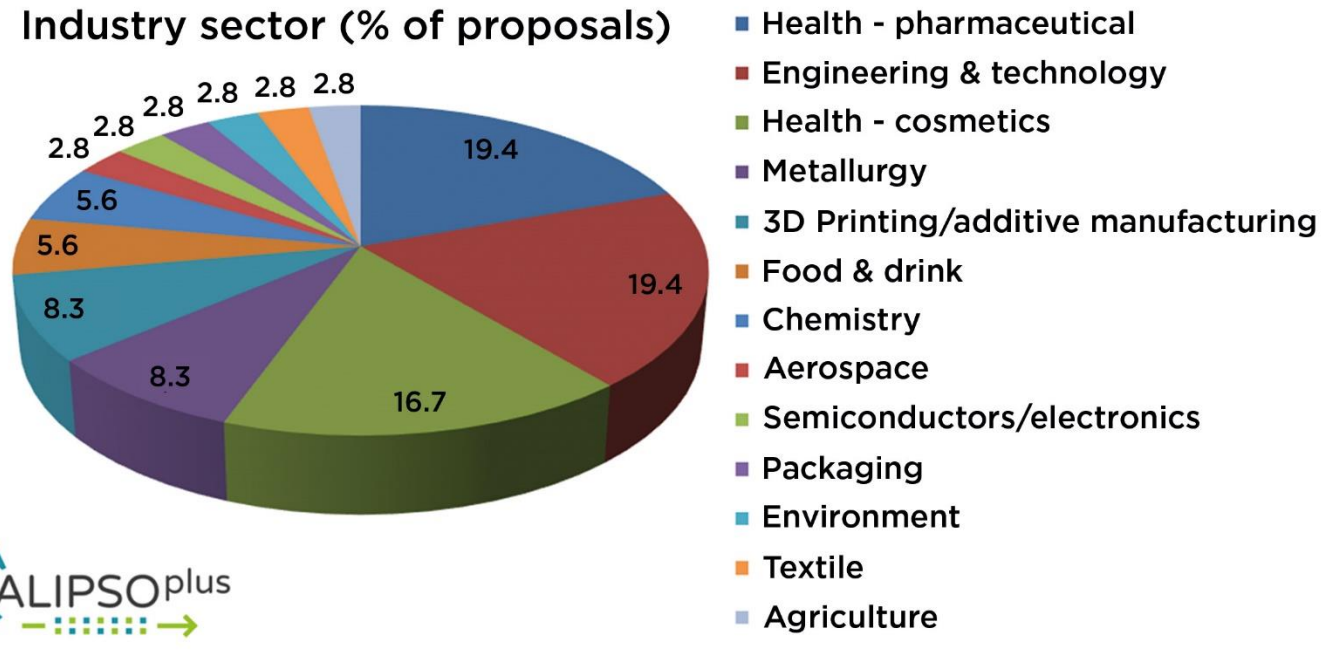
industry@remade-project.eu

SME success story: TamaTA programme

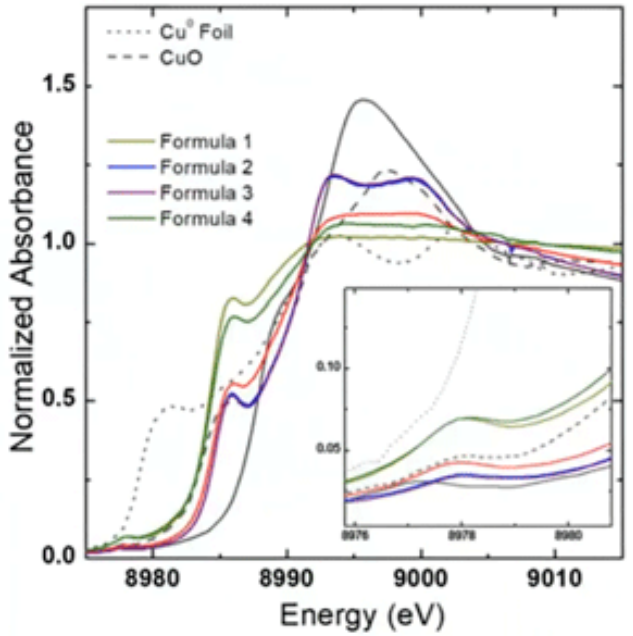
SME success story: TamaTA programme

Within the EU project CALIPSOplus, RI facilities boosted European SMEs innovation by gathering micro-scale information about their products.

- Synchrotron SOLEIL and a Swedish SME worked together to improve methodologies for surface analysis
- ESRF and an Italian SME perfected its detector for food safety
- ALBA-CELLS and a Spanish SME worked together to improve food packaging systems
- ALBA-CELLS helped a Spanish SME for the formulation of an antimicrobial for agriculture



SME example: Enhancing agrochemicals through synchrotron light



X-ray absorption spectra of various formulations collected at ALBA synchrotron.

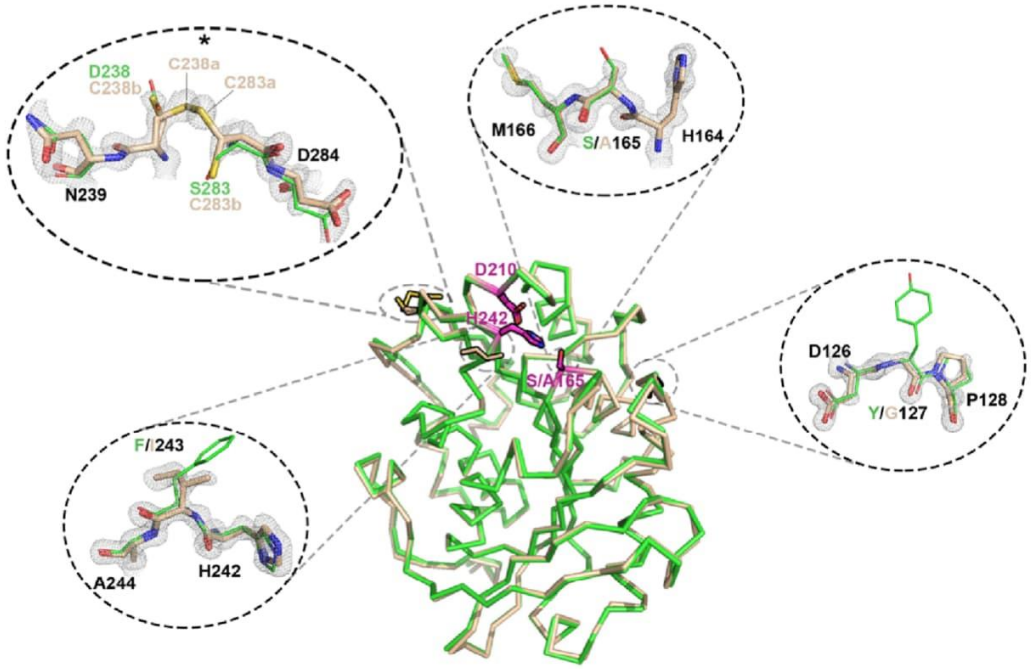
- A complexed copper solution has been developed to improve the pruning process.
- Higher complex concentration is linked to a greater effectiveness of the solution.
- A synchrotron light technique was used to characterise the copper contents in four different formulations and allowed the verification of the product, a vital part of the project, crucial to register the product under the ECHA (European Chemicals Agency)
- https://www.cells.es/en/media/news/enhancing-agrochemicals-with-synchrotron-light

These measurements were funded by the TamaTA programme, under the European CALIPSOplus project (grant 730872)

Examples of circular economy research at ESRF

Example: Structure determination of a plastic degrading enzyme

Carbios, in collaboration with the Toulouse Biotechnology Institute, engineered an efficient PET depolymerase starting from an enzyme with a known structure and depolymerase activity, however, insufficient for industrial use. The new enzyme had higher thermal stability and activity permitting a 100-fold productivity improvement. MX data collection at ESRF beamline ID30B provided a crystal structure of the new PET depolymerase and confirmed modifications to the structure.



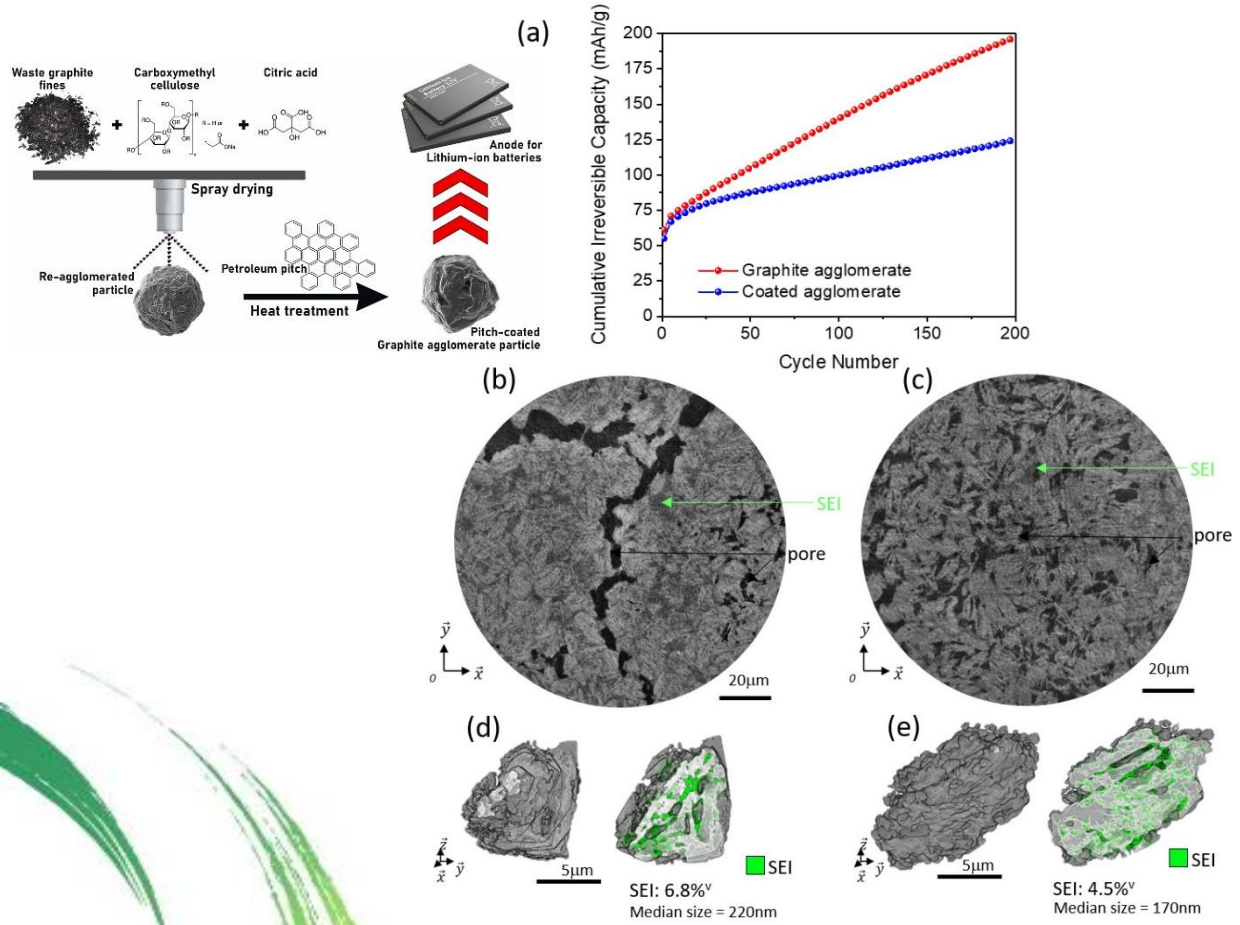
This research was concluded by a successful pilot-scale depolymerisation of PET waste reaching 90% conversion to terephthalic acid in 10 hours, which, following purification (99.8% pure), was demonstrated as suitable raw material for the fabrication of new PET bottles.

An engineered PET depolymerase to break down and recycle plastic bottles, V. Tournier et al., Nature 580, 216–219 (2020); doi: 10.1038/s41586-020-2149-4

An inactivated variant (S165A) of the engineered PET depolymerase is compared to the wild type (green). Close-ups show the introduced mutations and their surrounding residues. Credit: V. Tournier *et al.*

Example: Recycling graphite waste into battery anode material

Scientists from Institut National de la Recherche Scientifique (INRS), Canada, have developed a process to recycle waste graphite fines into a new electrode material by agglomeration and pitch coating. X-ray nano-tomography was used to visualise and compare the material at various stages of production and after battery cycling.



The CT volumes allowed quantification of the pore network on calendaring, which was reduced from 57% to 32%, electrode thicknesses of 105 µm and 41 µm.
CT was also used to visualise degradation of the materials after cycling.

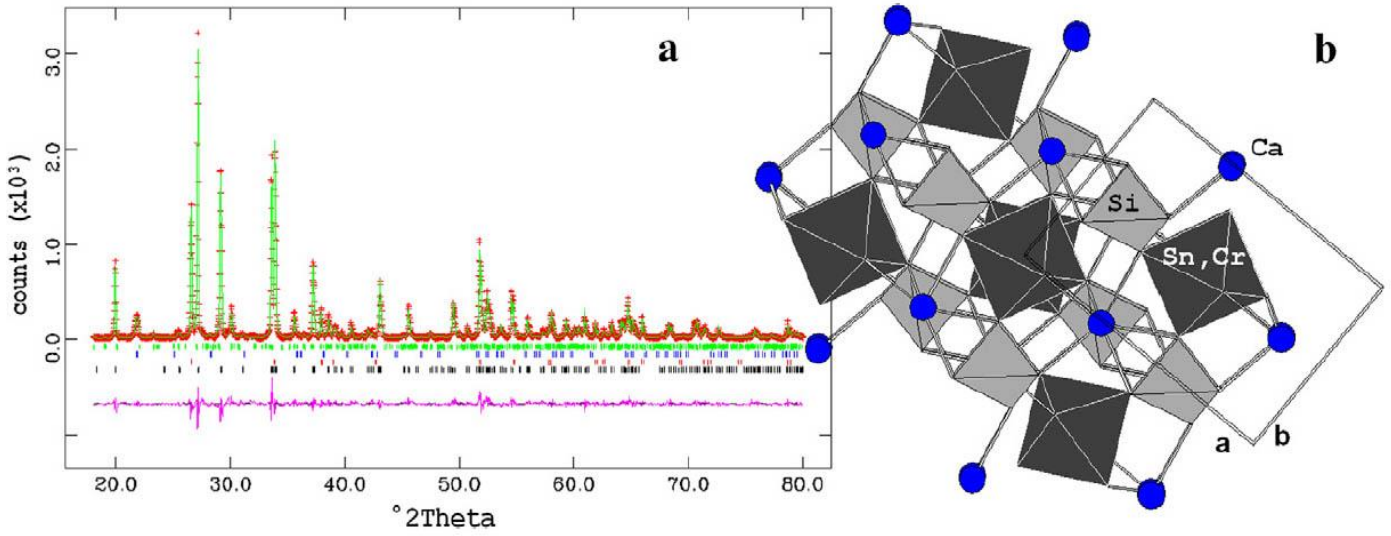
From waste graphite fines to revalorized anode material for Li-ion batteries, J.C. Abrego-Martinez, Y. Wang, V. Vanpeene, L. Roué, *Carbon* 209, 118004 (2023); https://doi.org/10.1016/j.carbon.2023.118004.

Evolution during cycling of (a) cumulative irreversible capacity and electrode/particle microstructures for (b,d) uncoated and (c,e) coated graphite agglomerates after 200 cycles. Credit: J.C. Abrego-Martinez *et al.*

Beamline ID16B

Example: Recycling asbestos

Zetadi s.r.l. + Univ. Modena have developed a thermal process (>1200°C) to transform waste asbestos into an inert Mg-rich silicate clinker ("KRY-AS"), for use as a low CO_2 secondary raw material for use in clay bricks, glass or ceramics. Methods such as SEM, XRPD and XRF were used in the development of this process, and high-resolution characterisation of products was made by powder diffraction (XRPD) at the ESRF.



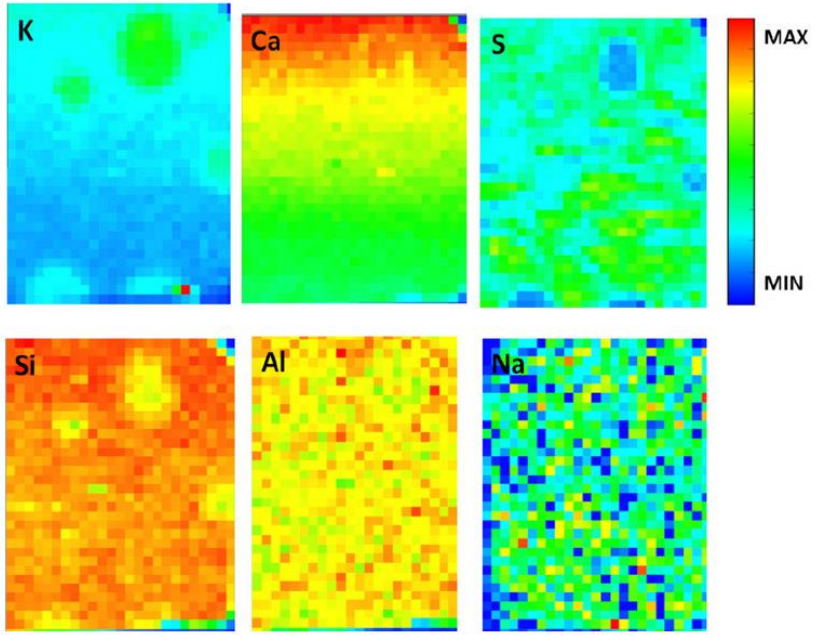
Recycling of the product of thermal inertization of cement–asbestos for various industrial applications, A.F. Gualtieri et al., *Waste Management* **31**, 91-100 (2011); Doi: 10.1016/j.wasman.2010.07.006.

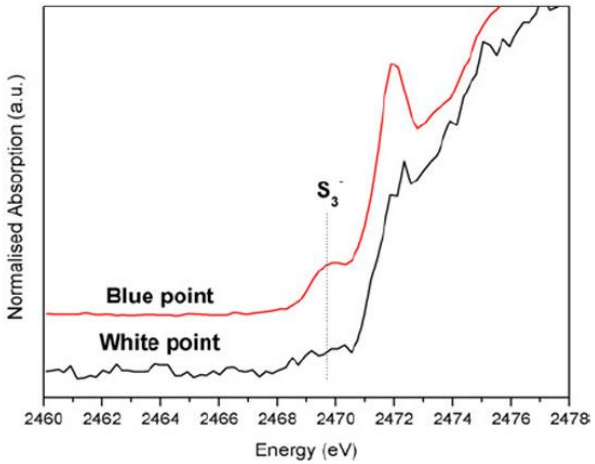
XRPD characterisation of a ceramic pigment made from KRY-AS.
Credit: A.F. Gualtieri *et al.*

Beamline ID22

Example: Concrete from recycled ground granulated blast-furnace slag

ECOCHEM's slag rich concrete is a low CO2 product. Researchers wanted to identify the chemical source of its blue-green colour, which can remain in the final product. They used X-ray fluorescence imaging and then K-edge X-ray absorption near-edge structure (XANES) to follow the evolution of the speciation of sulphur as the slag was hydrated.





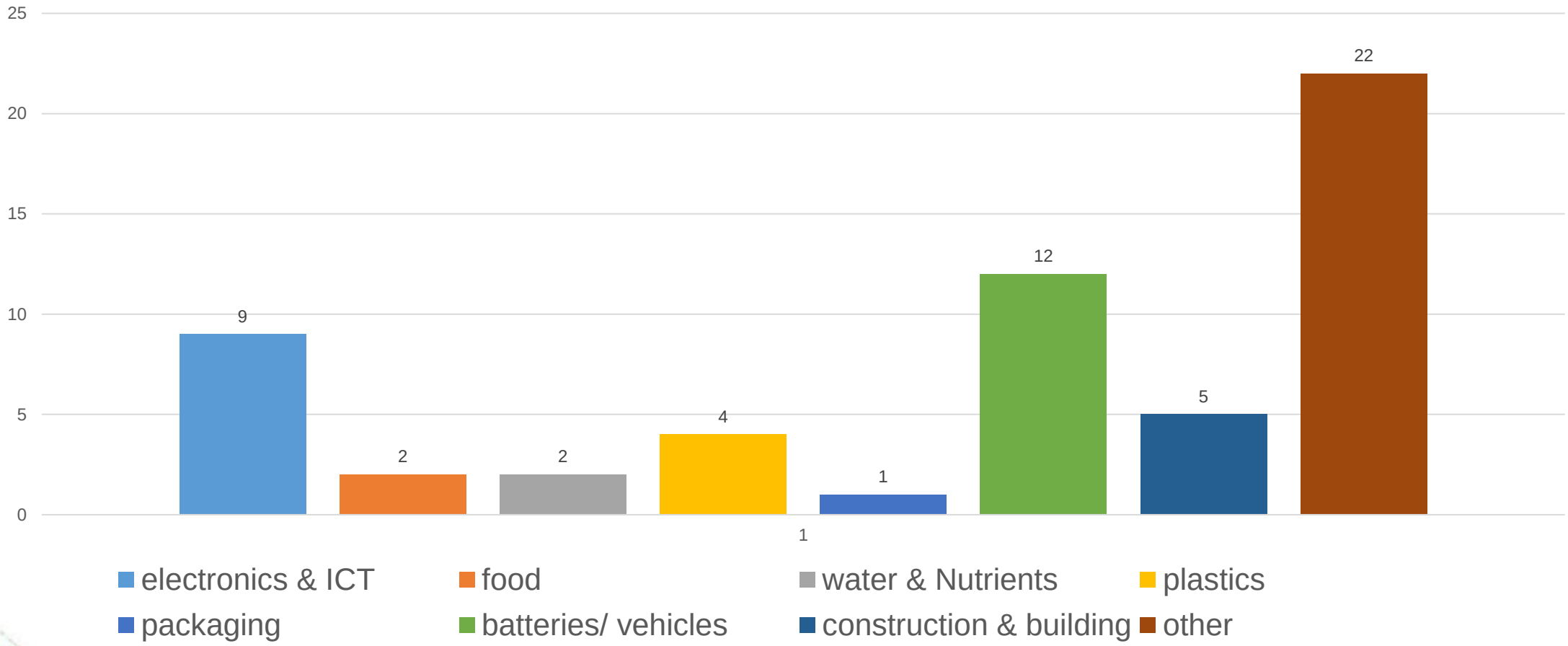
A cafe built with ECOCEM cement. Credits: ECOCEM.

Elemental distribution obtained by μ-XRF of ground-granulated blast-furnace slag activated by 4% sodium silicate solution; XANES spectra of blue and white regions. Credit: Chaouche M. *et al., J Am Ceram Soc.* 2017; 1-10. doi: 10.1111/jace.14670.

Beamline ID21

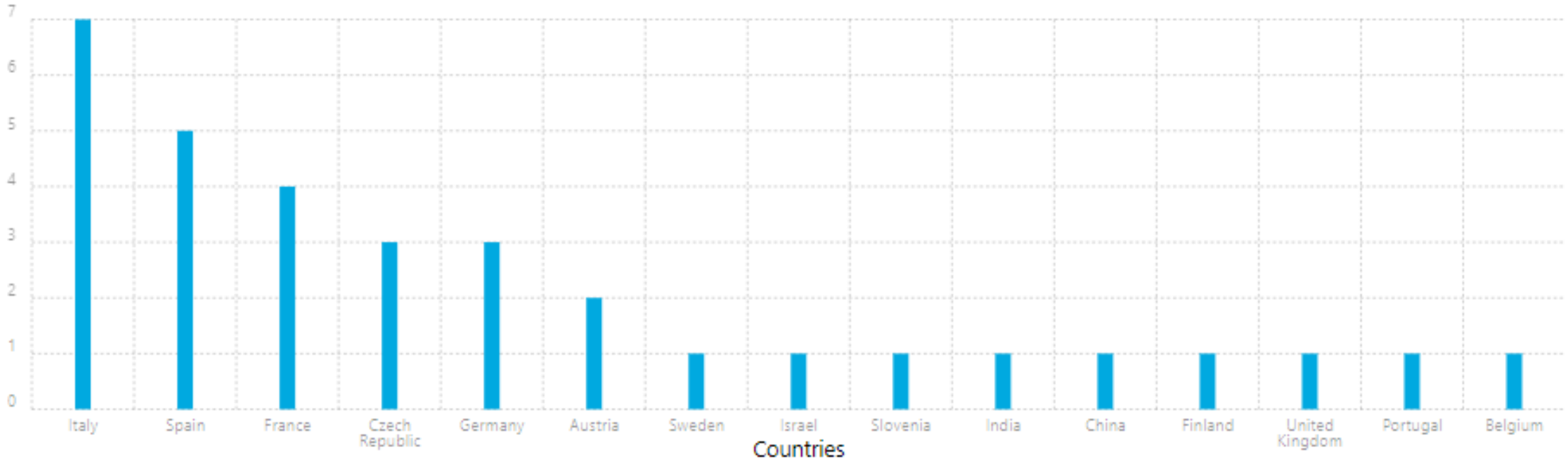
Results of first call for ReMade-TNA (30 April 2023):

Results of first call for ReMade-TNA: 34 project proposals received



Results of first call for ReMade-TNA (30 April 2023)

Proposal owners by country



Results of first call for ReMade-TNA (30 April 2023)

Top six requested techniques

Facility	Service/Technology	Visits
ReMade: MAX IV Laboratory (MAX IV)	? Please assign my facility	8
ReMade: Ion Beam Center (IBC), Helmholtz-Zentrum Dresden-Rossendorf (HZDR)	Ion beams materials modification at HZDR-IBC, Dresden, Germany	6
ReMade: ALBA Synchrotron	SAXS-WAXS at ALBA Synchrotron, Barcelona, Spain	4
ReMade: The Swiss Lightsource (SLS)	X-ray spectroscopy at SLS, Villigen, Switzerland	4
ReMade: Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons (ER-C)	Electron microscopy at ER-C, Jülich, Germany	3
ReMade: ESRF Experiments Division	X-ray tomography at ESRF Experiments Division, Grenoble, France	3

ReMade Webinar Series

ReMade @ARI

31st March 2023, 14.00 CET

Stefan FACSKO
HZDR, Germany

ReMade@ARI - A hub for material research towards a European circular economy

21st April 2023, 14.00 CET

Jakub DRNEC
ESRF, France

X-ray diffraction used for operando studies of batteries and fuel cells

19th May 2023, 14.00 CET

Sarah ROGERS
ISIS, United Kingdom

Neutrons and Nanometers

30th June 2023, 14.00 CET

Johan MEERSSCHAUT
IMEC, Belgium

Introduction to ion beam analysis

28th July 2023, 14.00 CET

Demetrios ANGLOS
FORTH & UoC

Mobile and remote analysis of materials using laser spectroscopy

26th January 2024, 14.00 CET

Peter FOUQUET
ILL, France

Neutron spectroscopy studies of hydrogen and oxygen diffusion in energy materials

24th November 2023, 14.00 CET

Iva BOGDANOVIĆ RADOVIĆ
RBI, Croatia

Ions - a powerful tool for analyzing and modifying materials

27th October 2023, 14.00 CET

Belén BALLESTEROS
ICN2, Spain

Electron microscopy techniques and application examples

29th September 2023, 14.00 CET

Ralph GILLES
TUM, Germany

Neutron techniques for in-situ/operando studies of batteries and gas turbine components

1st Sept. 2023, 14.00 CET

Eric HIRSCHMANN
HZDR, Germany

Non-destructive material Analysis using positron annihilation spectroscopy (PALS) - an overview

https://remade-project.eu/index.php/events-list/

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Many thanks for your attention

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Find us here!

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