# ESRF Information Day

## The Use of Synchrotron Radiation in Science

## 6th of May, 2022

## Conference and Cultural Center
## University of Patras, Greece

live webcasting at:

Aristotle University of Thessaloniki-School of Physics, Thessaloniki
NCSR Demokritos - Institute of Nuclear and Particle Physics, Athens
National Hellenic Research Foundation, Athens
Foundation for Research and Technology-Hellas, Heraklion-Crete
University of Ioannina-School of Physics, Ioannina

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# Foreword

The Greek Synchrotron Users Network (GrSUN) is, by now, an informal network aspiring to organize, for the first time, the Greek community of Synchrotron Radiation Users and strengthen the interaction between its members. One of the major goals of GrSUN is to promote the participation of Greece in the European Synchrotron Radiation Facility (ESRF). In this framework, GrSUN, together with the ESRF and the University of Patras, are organizing "The ESRF Information Day on the use of Synchrotron Radiation in Science" in order to disseminate and communicate to the Greek scientific community the work and type of research conducted at the ESRF. The majority of the speakers belong to the directorate or the scientific staff of the ESRF, whereas some talks will be given by Greek synchrotron radiation users.

The program is organized in 6 sessions:

- ESRF overview by the ESRF directors
- Biomedical imaging and cultural heritage
- Life sciences
- Energy-catalysis-environment
- Nanoscale matter and quantum materials
- Industrial research and activities

The event will be hybrid with on-site and on-line presentations and live webcasting in a number of Institutes and Universities in Greece.

The Organizing Committee

# Program


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| | • Deputy Minister of Development & Investments, Research, Innovation and Technology<br>• Deputy Governor of Entrepreneurship Research & Innovation Region of Western Greece<br>• Rector, Vice Rector for Research and Development, Dean of the School of Natural Sciences (University of Patras)<br>• Dean of the School of Natural Sciences (Aristotle University of Thessaloniki)<br>• Representatives of the Greek Synchrotron Users Network (GrSUN) | |
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| $10^{20}$ - $10^{40}$ | **EBS: A new light for life sciences - first scientific highlights**<br>Annalisa Pastore, *The European Synchrotron Radiation Facility* | **8** |
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| $11^{50}$ - $12^{10}$ | **Hard X-ray bio-imaging at the nanoscale**<br>P. Cloetens, S. Bohic, M. Eckermann, D. Karpov, F. Monaco, A. Pacureanu, M. Salome, *The European Synchrotron Radiation Facility* | **11** |
| $12^{10}$ – $12^{30}$ | **Synchrotron Radiation in modern medical imaging**<br>Georgios Panayiotakis, *University of Patras* | **12** |
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| **$15^{00}$ -$16^{40}$** | **Session 4: Energy – Catalysis – Environment** | |
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| $19^{00}$ | End of meeting | |

# abstracts

# EBS: A new light for physical sciences - first scientific highlights

**Francesco Sette**, ESRF Director General
The European Synchrotron Radiation Facility, Grenoble, France

The ESRF, the European Synchrotron, a user facility supported today by 21 partner countries, started in August 2020 its user operation with a new and first-of-a-kind high-energy fourth-generation synchrotron light source, which provides synchrotron X-rays 100 times brighter and more coherent than ever before.

This new source, the ESRF Extremely Brilliant Source (EBS), is today unparalleled worldwide for its performances: it renews the pioneering role of the ESRF in synchrotron science, similarly to what had been the successful commissioning in 1992 of the first ESRF third-generation synchrotron light source. The ESRF-EBS provides today the driver for a new science reach and for the construction and upgrade of, respectively, new and existing synchrotron sources worldwide.

With these outstanding and unique performances, in 2022, year marking the celebration of the 75th anniversary of the first observation of accelerator-based synchrotron radiation, the ESRF strengthens its contribution in pursuing excellence in science for the benefit of our society, enabling new experimental possibilities and inspiring the new generations of scientists and engineers. Among others, the EBS hails a new era for X-ray science in the 3D exploration of matter from the molecular to the macroscopic scales, providing new reach to the understanding of life in its multitude of hierarchical organisations, as well as in the development of the materials of the future where energy and environmental sustainability are of key importance. Altogether, EBS is enabling scientists to address the major global challenges facing our society today, in areas such as health, energy, environmental and climate change, new materials, sustainable development and world's cultural heritage.

The ESRF is engaged to drive innovation by welcoming on its state-of-the-art facilities proprietary and non-proprietary, non-military industrial programmes, and by developing advanced instruments and new technologies, with the objective of serving society and bringing new opportunities to citizens life and economy.

I will provide an overview on the present status of the ESRF and of its user operation with the new EBS source, as well as examples of first EBS-based scientific results and new opportunities that the facility will provide to its user community in the coming years; themes that will be discussed in much more detail during the day.

# EBS: A new light for physical sciences - first scientific highlights

**Gema Martínez-Criado**, ESRF Director of Research for Physical Sciences
The European Synchrotron Radiation Facility, Grenoble, France

The ESRF is the first high-energy, fourth-generation synchrotron, which constitutes a landmark for fundamental and innovation-driven research. With the support of the ESRF's 21 international partner countries, a brand-new generation of high-energy synchrotron, the ESRF's Extremely Brilliant Source (EBS) was launched in 2020 with superior X-ray performances (up to a factor 100) in terms of brilliance, coherence and emittance. In the quest to push the boundaries of knowledge and technology for the benefit of society, EBS produces the most brilliant X-rays to unveil the structure of matter. Thus, EBS provides scientists from all over the world with new opportunities to pioneer new fields of investigation for fundamental research, also permitting unprecedented analysis and understanding of materials down to atomic resolution. Based on scientific excellence, research carried out with the EBS contributes to addressing the complex global challenges that our society faces, such as health, energy and the environment. Pushing the frontiers of science, ESB makes the invisible visible, unveiling the secrets of matter to advance fundamental knowledge and new applications, covering biomedical science, novel materials for energy, extreme conditions (planetary research and geoscience, cutting-edge materials), nanomaterials, etc. It also contributes to the development of new and clean technologies for industry and to preserving humanity's cultural heritage, lighting the way to a brighter, sustainable and peaceful future.

This talk will present the EBS benefits, its exploitation and the new experimental capabilities available to academic and industrial users in Physical Sciences. The first scientific highlights from the main flagship and refurbished beamlines will be briefly described. Like a super-microscope, the presentation will illustrate how the enhanced performance of the X-rays, combined with new stations and state-of-the-art instruments, will revolutionize biomedical phase-contrast imaging, and will make the study the structure of condensed matter possible at the nanometre scale under operando or extreme thermodynamical conditions with higher resolution, greater image quality and faster framerate. Finally, this presentation will give a summary of the status of the beamlines under construction, their design choices and strategic research, a snapshot of its present status and some considerations of their future perspectives.

## EBS: A new light for life sciences - first scientific highlights

**Annalisa Pastore**, ESRF Director of Research for Life Sciences
The European Synchrotron Radiation Facility, Grenoble, France

Life Science at ESRF mainly, but not exclusively, rotates around three groups: X-ray Nanoprobe, Complex Systems and Biomedical Sciences and Structural Biology. The X-ray Nanoprobe Group has developed new exciting areas of X-ray imaging, that cover fields as diverse as biology, cultural heritage and neuro-nanoimaging, in addition to more traditional physical, medical, materials science and engineering subjects. The Complex Systems and Biomedical Sciences group covers a variety of soft matter studies which address questions concerning the microstructure, dynamics, self-assembly, structural kinetics and rheology of complex and nanostructured materials (polymers, colloids, surfactants, liquid crystals, proteins, etc.) in bulk, at interfaces as well as in confined geometry. Among the beamlines in this group, there is the biomedical beamline ID17 which aims at coupling diagnostic and functional imaging with radiation therapy. This work relies on a strong net pf collaborations with local and European hospital teams. Finally, the Structural Biology group operates a world leading suite of synchrotron radiation beamlines dedicated to the study of biological macromolecules: Among the Structural Biology beamlines is a top class cryoEM facility which is operated as a beamline and jointly run by scientists form ESRF, EMBL, IBS and ILL. The Structural Biology group comprises also of the High Pressure Freezing Laboratory for Macromolecular Crystallography (HPMX) which allows users to run crystallographty without cryo-protectants or the introduction of gases in macromolecules.

In my talk, I will provide an overview of the impact that the recent refurbishment of the synchrotron and the development of the Extremely Brilliant Source have had on Life Science-related beamlines and discuss the commissioning of two new beamlines, ID18 and ID29. ID18, which has started construction only recently, will be devoted to the use of "coherent beams" to investigate the structure and the dynamics of complex systems. This dedicated beamline will take advantage of its length (sample at 200 m from source) to use an innovative optical layout that will allow unprecedented tunability in terms of beam size and degree of coherence. ID29 is intended for room temperature serial crystallography experiments over a wide energy range (10-25 keV and 35 keV) with an extremely high flux density that will allow room temperature and time resolved studies in crystal samples.

# ESRF Beamtime - Access, Proposals and Use

**Joanne McCarthy**, Head of ESRF User Office
The European Synchrotron Radiation Facility, Grenoble, France

The ESRF has built more than 30 specialised beamlines for use by the international scientific community. These beamlines are designed for research in areas as diverse as engineering, physics, chemistry, crystallography, earth science, biology and medicine, cultural heritage, environmental, surface, and materials science. In addition, several Collaborating Research Groups (CRGs), made up of institutes from countries participating in the ESRF, have built and operate 13 beamlines at the ESRF with independent funding. In 2022, a total of 48 independently functioning experimental stations are available for users to carry out their research. Of these, 35 stations are on ESRF public beamlines and 13 stations are on privately funded CRG beamlines. The privately funded CRG beamlines use two-thirds of the beam time on their beamlines for private use, while one-third is available for the ESRF public beamtime programme.

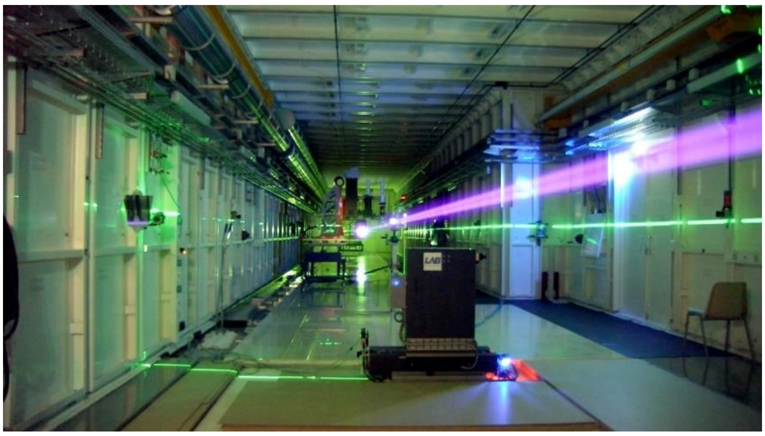
Access to the ESRF beamlines is open to scientists worldwide, and the ESRF welcomes proposals from both academic and industrial laboratories, proposing two main types of access channel for both: a public access channel which is peer reviewed and free of charge, and where users are under the obligation to publish their results in the public domain; and a proprietary research access channel where beam time is purchased and not subject to peer review, and where the results remain the property of the user. Access to the private beam time on the privately funded CRG beamlines is managed independently from the ESRF by the beamline owners, through their own peer-review procedures.

This presentation will describe the public access channel, giving the most important information for scientists interested in applying for public beamtime at the ESRF. The procedures for requesting access to public ESRF beam time and the different public proposal types available will be described. The talk will also cover the proposal review procedure and give some tips for writing good proposals. Finally, the support available for scientists with accepted proposals will be described (onsite Guesthouse and restaurant, travel organisation, financial assistance, etc), as well as the obligations of the user to report on the beamtime used and publish the results obtained.

# From cultural heritage to biomedical imaging, development of synchrotron phase-contrast hierarchical imaging at the ESRF

**Paul Tafforeau**
The European Synchrotron Radiation Facility, Grenoble, France

The European Synchrotron Radiation Facility has a long history in X-ray full-field imaging, especially in propagation-based phase-contrast imaging. Among the many research topics involving this technique, palaeontology and archaeology are noticeable ones. During the last two decades, constant efforts have been made to investigate larger and larger fossils or archaeological remains, whilst being able to image regions of interest at high-resolution with local tomography. Imaging these larger specimens, especially those as dense as fossils, requires higher energies, longer propagation distances and higher beam coherence. In these respects, the technical limit with the 3rd generation synchrotron sources were basically reached. The project of the ESRF-EBS, the first high-energy 4th generation synchrotron source, is a game changer. The highest possible coherence of this new lattice is obtained on the short wiggler sources that replaced the original bending magnet sources of the previous machine. A specific project (BM18) aiming at fully exploiting this coherence up to 250 keV, began in 2017. In parallel, the existing beamline BM05 was refurbished to exploit this coherence up to 140 keV. In 2020, with the emergence of the COVID-19 pandemic, the ESRF decided to join an international research effort collaborating with Imaging scientists at University College London and Clinical scientists treating COVID-19 patients in Hannover, Mainz, Heidelberg and Grenoble, to help understand the disease. By adapting specific scanning protocols originally implemented to image fossils and archaeological remains, it was possible to develop a new approach on BM05 to image complete human organs, including those damaged by SARS-CoV-2 infection, with a level of precision unknown so far. The technique, termed Hierarchical Phase Contrast Tomography (HiP-CT), has already brought impressive results on BM05, but is now reaching much higher contrast, speed, resolution and sensitivity on much lar ger samples on BM18. This 220m long beamline with unique properties for phase-contrast imaging worldwide. It will be able to scan large samples entirely at 20 µm, and then to zoom-in on selected regions to reach micrometer resolution anywhere in the sample. BM18 is partially funded by a BMBF grant in collaboration with the Fraunhofer institute in order to develop industrial applications. The biomedical project for imaging of human organs is funded by the Chan-Zuckerberg Initiative. The main scientific topics of these beamline are material sciences, biomedical imaging, cultural and natural heritage and geology. BM18 will start academic user operations in autumn 2022, and is expected to reach its full capacity in 2023.



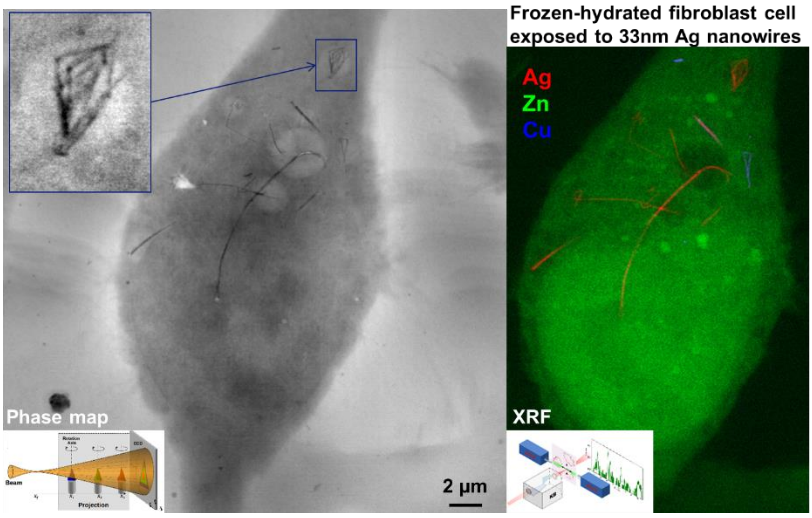
# Hard X-ray bio-imaging at the nanoscale

**P. Cloetens, S. Bohic, M. Eckermann, D. Karpov, F. Monaco, A. Pacureanu, M. Salome**

The European Synchrotron Radiation Facility, Grenoble, France

Coherent hard X-ray microscopy has seen remarkable progress in recent years and it is increasingly recognized as a disruptive technology for life science research [1-8]. With the advent of the fourth generation synchrotron sources, new opportunities arise to probe the 3D structure and elemental composition of biological specimens with unprecedented precision and speed. The penetration power of high energy X-rays enables imaging of thick biological samples, which can be preserved close to their natural state through vitrification and imaging in cryogenic conditions. The improved coherence of fourth generation sources fosters overcoming frontiers of resolving power.

This talk will cover current capabilities of X-ray phase contrast nano-tomography and X-ray fluorescence at the nano-imaging beamline ID16A [9] of ESRF for exploration of cells, tissues and organisms from our ecosystems. These studies reveal the intracellular targets of drug compounds or the effects of pathologies and novel therapies. We will discuss as well the challenges to address for improving spatial resolution and scalability, along with opportunities to integrate X-ray microscopy with modalities measuring functional and molecular properties of living organisms in order to advance our understanding of life.

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# Synchrotron radiation in modern medical imaging

**Georgios Panayiotakis**

University of Patras, School of Medicine

In the last decades, Synchrotron Radiation (SR), being the brightest artificial source of x-rays with a very promising geometry, has raised the scientific expectations that could be used for medical imaging with optimized results.

In particular, in a SR facility, photon beams of desirable energy can be produced (monochromaticity) while having a remarkably high brightness. Moreover, the high spatial coherence, beam collimation and polarization are important additional advantages that can lead to breast images with high diagnostic quality and lower patient dose in medical imaging. The monoenergetic nature of the SR beams is significantly better for medical imaging than broad spectra, as it is free of lower energy photons that only contribute to patient dose and of higher energy photons that degrade the image quality of the generated image.

Furthermore, the intensity and polarization of the SR beams have initiated an entirely new era that is yet to be explored in medical imaging. For example, phase imaging, mainly possible in SR facilities, is based on the photons' interactions as waves, contrary to the conventional absorption effects that focus on its interactions almost as a particle.

However, this "in situ" evaluation technique is complex due to the limited availability of SR facilities worldwide. The Department of Medical Physics of the University of Patras has developed and validated a Monte Carlo based simulation model for SR breast imaging, mimicking the SYRMEP beamline of the Elettra facility in Trieste, Italy. Results concerning the performance of SR as a tool for breast imaging have been derived and published.

# Shedding new light on ancient technology: Synchrotron radiation-based micro-XANES and micro-XRF analysis of Egyptian blue from the Late Hellenistic workshop of Kos (Greece)

**Ariadne Kostomitsopoulou Marketou**
NF Norwegian School of Theology, Religion and Society, Oslo, Norway

Over the last years, synchrotron radiation (SR) methods have found application in the study of various materials of cultural interest. Questions regarding elemental and/or phase composition and complex degradation phenomena are increasingly approached through the analytical power of SR facilities. By extension, the high-resolution analytical data generated through SR-based techniques give insight into questions regarding authentication and dating, while at the same time, the contextual interpretation of experimental results can deepen our understanding of past manufacturing processes. This contribution aims to illustrate how SR can be exploited to answer research questions revolving around complex inorganic ancient materials based on my experience from working at the ID21–X-ray microscopy beamline of the European Synchrotron Radiation Facility (ESRF) to analyse Egyptian blue samples from the Late Hellenistic workshop of Kos. Egyptian blue is an artificial, multicomponent material that was produced through a demanding pyrotechnological process. Despite the widely documented use of Egyptian blue throughout different periods, very few production sites have so far been archaeologically identified. Therefore, the workshop of Kos, where evidence of Egyptian blue production has been found in the context of a multi-crafting workshop, works as an ideal case study to contextually approach the production process.

The principal crystalline phase of Egyptian blue is a copper calcium tetracilicate ($CuCaSi_4O_{10}$, the artificial equivalent of the mineral naturally occurring as cuprorivaite). Among the approximately one hundred Egyptian blue pellets found at the Koan workshop, the so-called 'unsuccessfully produced' pellets (i.e., finds that were characterised as Egyptian blue due to the archaeological context, their shape, and the presence of cuprorivaite, but did not demonstrate blue colour), were considered particularly intriguing as a medium to approach the technological process of production. A multi-analytical methodology was followed, combining 'conventional' laboratory techniques (i.e., optical microscopy, scanning electron microscopy coupled with energy dispersive X-ray spectroscopy and micro-Raman spectroscopy) with the SR micro-X-ray absorption near edge structure and micro-X-ray fluorescence spectroscopy and principal component analysis. The results suggest that the unsuccessful outcome was related to the use of inappropriate starting materials that introduced iron in the starting mixture, shedding therefore light on the local technological choices. Finally, moving beyond the discussion of the results, this contribution aims to discuss the challenges faced during the experimental design and data interpretation and to underline the importance of interdisciplinary and collaborative work when approaching complex archaeological questions.

# Facilities for structural biology at the European Synchrotron

**Christoph Mueller-Dieckmann**

The European Synchrotron Radiation Facility, Grenoble, France

The ESRF has a long-standing history and record in performing Structural Biology (SB) experiments. Since the beginning, a major focus has been put on automation of experimental procedures performed on and the ease-of-use of experimental end-stations along with the development of techniques either to extend further the offer made to users or to pave the way for new scientific procedures. Over the years, the offer made to the international SB community has constantly increased from covering 'classical' diffraction experiments in macromolecular crystallography (MX) to scattering experiments of biological samples in solutions (bioSAXS), singe particle reconstruction cryo-electron microscopy (SPR cryo-EM) and more recently time-resolved experiments using serial crystallography (TR-SSX) approaches. In addition to this, spectroscopic characterisations of macromolecular specimen either in crystals or in solution using UV-Vis, fluorescence and/or Raman have extended the spectrum offered, both online mounted on one of SB's diffraction end-stations and offline. Since 2013, high-pressure applications on macromolecular samples using various gases (e.g., He, Ar, Xe, Kr, $O_2$, $CO_2$) are also available which can be used for example to identify entry and/or exit routes of gases within biological macromolecules among other applications. Most of the experiments can be performed by users physically present at the ESRF with the support of SB staff. Beside this classical access mode, remote experiments (users taking control of an experimental end-station on the day of their scheduled experiment) or automatic and mail-in services (users sending samples to the ESRF with the experiments handled either automatically or by ESRF staff) are alternative possibilities.

The presentation will summarise various possibilities available to perform experiments in the field of Structural Biology at the ESRF including some scientific highlights of the last few years demonstrating specific applications.

# The Cryo-EM facility at the ESRF: an essential tool for Integrated structural biology

**Eaazhisai Kandiah**

The European Synchrotron Radiation Facility, Grenoble, France

Owing to the advent of several technological advances in the recent years, cryo-electron microscopy (cryo-EM) has become a routine technique to obtain near to or atomic resolution structures of (larger) macromolecular complexes. Stimulated by this ‘resolution revolution’ of the technique, many national and international centres for cryo-EM have been created since. At the ESRF, the cryo-EM facility (named CM01) focuses on single particle reconstruction of biological macromolecules as a complementary technique to existing macromolecular crystallography beamlines and facilities available at the ESRF. This facility is jointly operated by a consortium of all institutes (EMBL, IBS, ILL and ESRF) co-located on the European Photon and Neutron (EPN) Campus and is managed in a similar fashion to a synchrotron X-ray beamline. It is open to the ESRF’s international structural biology user community since November 2017 and has been exploited by users to reveal important insights of biological molecules involved in wide range of either diseases including neurodegenerative diseases or applications including nanomedicine. Since 2020, an additional service is being offered specially to those users with limited or no access to cryo-EM (screening and/or high-end microscopes) that starts from a purified sample solution or from pre-frozen grids. Additionally, bi-annual practical workshops on sample grid preparation are organised to train ESRF users relatively new to this technique. I will discuss the current set up of the facility and the user program including few scientific highlights.

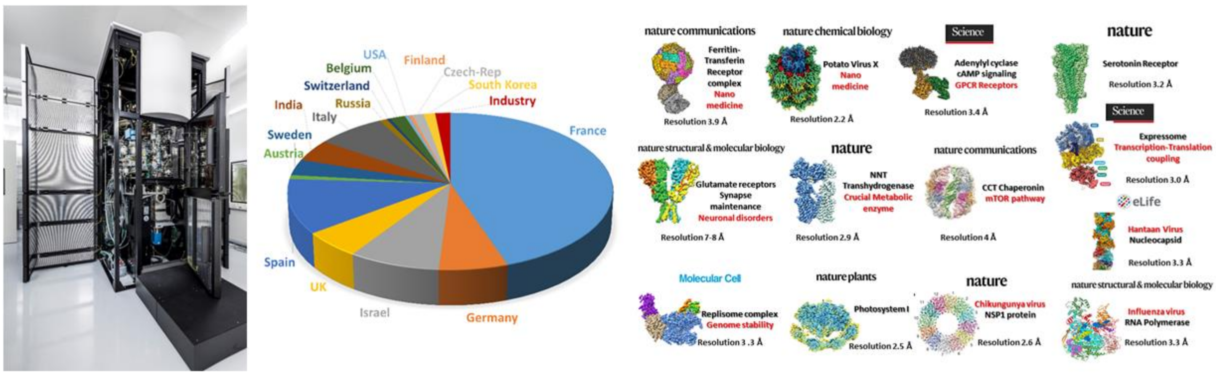


***Figure***: *Titan Krios G3 microscope installed in CM01 has been extensively used by the international user community (middle figure) to study structure and function of important of biological macromolecules.*

# Synchrotron Radiation as a catalyst for the evolution of Structural Biology in Greece

**E.D. Chrysina,** Coordinator of Inspired-RIs- The National RIs on Integrated Structural Biology, Drug Screening Efforts & Drug Target Functional Characterization, Institute of Chemical Biology, National Hellenic Research Foundation, Athens, Greece

The Greek structural biology community has been active for more than 40 years. The activity was initiated in the late 1970s – early 1980s when only a few Greek crystallographers, in the National Hellenic Research Foundation (NHRF), the National Centre for Scientific Research (N.C.R.D)-"Demokritos" and the University of Crete", transferred the knowledge and the expertise they had acquired in laboratories in Europe and the USA and paved the way for structural biology in Greece. X-ray data collection was possible only in synchrotron radiation sources and access was granted initially through scientific cooperation built with large research groups some of which were either based at synchrotrons or were regular users at the time and then through competitive grant applications.

It was only in 1997 that the first in house X-ray facilities for macromolecular crystallography were installed at the N.C.S.R.-"Demokritos" and the Foundation for Research and Technology (FORTH) in Crete. During those years, the research output of the community was on the increase, and it was access to SRS that made this possible. The community of X-ray protein crystallographers was growing fast and in 2001 the Hellenic Crystallographic Association (HeCrA, www.hecra.gr) was established to meet the needs of the community.

The power of SRS has been determinant for the expansion of the community. Tight bonds with the pioneering groups based at SRS made a tremendous impact giving the opportunity to Greek users to access the European facilities through a number of transnational access schemes available at the time. Furthermore, a significant number of young scientists were trained through the frequent access for X-ray data collection, and got a flavor of the team spirit and the excitement of performing cutting edge research at large scale facilities. This atmosphere acted as a source of inspiration for the structural biology community that unlocked the regional potential by attracting European funds for establishing more X-ray protein crystallography facilities for example at NHRF in 2010.

The structure-based drug design approach has become one of the core activities of the community, which had already started to expand including a broad range of expertise for protein sample preparation, biophysical characterization, structural analyses with X-ray crystallography and powder diffraction, SAXS and NMR. In the meantime, at European level, Instruct was one for the ESFRI projects that was entering the preparatory phase and Greek structural biologists had already created the critical mass to join this initiative. In 2017 Greece joins Instruct-ERIC (www.instruct-eric.org) as an observer and in 2018, structural biology gets recognized as a national priority though Inspired-RIs (www.inspired-ris.gr).

Today, the national RI is unique in South-East Europe, the Balkans and the Mediterranean area, including Cyprus. Our aim is to preserve the momentum by becoming full members of Instruct-ERIC and by joining forces with large European facilities like ESRF that will allow nurturing the next generation of structural biologists preventing the brain drain.

# Towards holistic understanding of electrochemical energy conversion and storage systems using high energy X-rays

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Complete physico-chemical operando characterization of electrochemical devices for energy conversion and storage is necessary to guide the development and to improve the performance. High brilliance synchrotron X-ray sources play a crucial role in this respect as they act as a probe with relatively high penetration power and low damage potential. Synchrotron sources will undergo major upgrades in next decade and will provide even higher brilliance and, more importantly, coherence. These upgrades will be particularly advantageous for beamlines providing high energy X-rays as they will allow use of advanced scattering techniques with highly penetrating probe. Therefore, the techniques typically used for ex-situ measurements or at lower X-ray energies could be used on materials in liquid half-cells and operating electrochemical devices. In this contribution the new possibilities of using high energy, high intensity, coherent X-rays to probe model systems and whole electrochemical devices will be presented.

To study fuel cells or batteries as a whole, elastic scattering techniques such as wide angle and small angle scattering are typically employed, as they can provide important complementary information to more standard X-ray imaging and tomography. The advantage is that the chemical contrast and sensitivity at atomic and nm scales is superior. Coupling these technique with the tomographic reconstruction (XRD-CT and SAXS-CT) is much less common as it requires bright synchrotron sources, fast 2D detectors and advanced instrumentation [1]. However, such combination allows spatial reconstruction of materials important atomic parameters in operando conditions. This will be demonstrated on imaging of standard 5 $cm^2$ PEM fuel cell during operation and Li-ion battery during charge and discharge.

Furthermore, local atomic and mesoscale structure, together with defect content, can also be determined by using Rietveld fitting, Pair Distribution Function (PDF) analysis and advanced SAXS theory. This in principle allows holistic investigations of interfaces at the device level and understanding the interplay between different phases during operation. These are critical questions needed to be answered in order to incorporate novel materials into the electrochemical devices. Examples will be given on studies of the ORR catalysts, amorphous Li-ion anodes and battery electrolytes.

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# Shining synchrotron light in agriculture

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Metals are persistent and accumulative in the environment, in agricultural soils they originate from natural and anthropic sources such as fertilizers, pesticides, manures, sewage sludges, or from atmospheric deposition from point sources (e.g., mining activities). Metals can accumulate in the food chain, and may enter the human body through diet or via inhalation of (re)suspended soil particles. In the last decades, engineered nanomaterials have been emerging as sources of metals in agro-systems. Metal based nanomaterials reach soils as biosolids components or as agrochemicals (nano-pesticides or nano-fertilizers), where metals are often the active ingredient and increase the risk of occupational exposure (through inhalation) as well as dietary exposure to metals (potentially in nanoforms). Since total concentrations rarely correlate to plant available concentrations, speciation is of high relevance, the metal pools available for plants depend on speciation as well as the translocation to edible tissues. Metal biodistribution and speciation in plants is an active research field, whether to understand metal biological roles, to improve food quality or to understand and prevent the accumulation of toxic metals (ionic and NPs) in the tissues (e.g. Cd in wheat grains and globe amaranth leaf; Fig. 1).

The ID21 beamline at the ESRF is dedicated to micro X-ray fluorescence (µXRF) and micro X-ray absorption spectroscopy (µXAS) in the tender X-ray range (2-10 keV). It has an important scientific activity in environmental sciences, in particular for studying the fate of metals in the environment. This includes, the study of pollutants and nutrients in crop plants. This presentation will highlight experiments performed at ID21 taking full advantage of the beamline capabilities to investigate the distribution and speciation of pollutants (Cd, Cr), nutrients (Fe, S, P) and engineered nanomaterials (Ti, Ag, Ce, Cu) in plant samples.

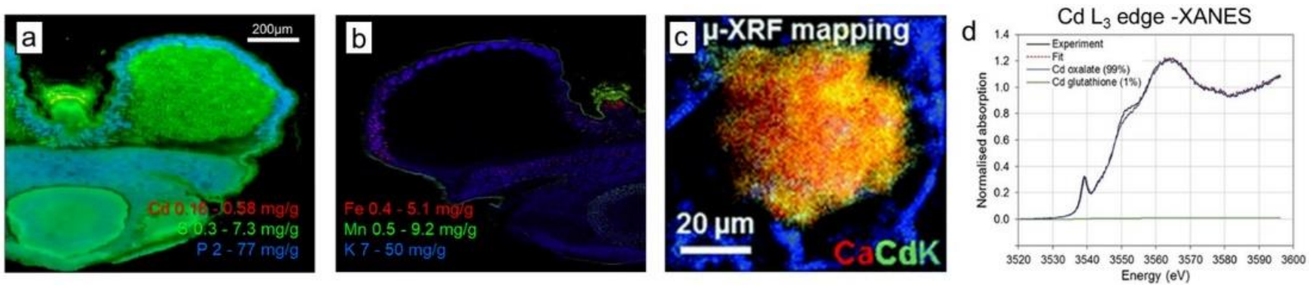


***Figure 1:*** *(a) Cd, Mg, and P biodistribution in wheat grain, (b) Fe, Mn, K biodistribution in wheat grain (Yan et al. 2020), (c) A calcium oxalate loaded with Cd in globe amaranth leaf, (d) Cd speciation in Cd loaded Ca-oxalate crystal by µXANES [1].*

**Reference**

*[1] P. Pongrac, T. S. Serra, H. Castillo-Michel, K. Vogel-Mikuš, I. Arčon, M. Kelemen, B. Jenčič, A. Kavčič, M. T. Villafort Carvalho, and M. G. M. Aarts, Metallomics **10**, 1576 (2018).*

# X-ray absorption spectroscopy for industrial and environmental catalysis

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Hard X-ray absorption spectroscopy (XAS) frequently proves to be very useful in catalytic studies because of its capability to deliver element-selective structural and electronic information about the active sites of complex materials, often inaccessible by other methods. Moreover, high penetrative power of hard X-rays offers a possibility to perform experiments in operando regime on the actual working catalysts. BM23 and ID24 beamlines of the ESRF are powerful and versatile XAS instruments, with important focus on catalytic studies. The beamlines combine XAS with various complementary techniques, such as X-ray emission, XRD, UV-Vis, FTIR and mass-spectrometry. Time-resolution available for chemical studies at BM23 and ID24 spans from microseconds to minutes per XAS spectrum. The beamlines are fully equipped for handling reactive gases and possess a wide choice of experimental cells for different types of samples and conditions. The talk will highlight the capabilities of BM23 and ID24 beamlines for chemical applications, featuring several recent case-studies in the fields of de$NO_x$ catalysis, characterization of novel metal-organic frameworks, homogeneous catalysis and others.

# X-Ray Absorption Fine Structure Spectroscopy as a tool to elaborate on the immobilization of pollutants in soil and groundwater

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Environmental pollutants have various adverse health effects linked to increased risk of morbidity and mortality. Toxic metals and highly soluble elements like Cr(VI), As(III) and As(V), Pb(II), Hg(0) and Se(IV), originating mainly from human activities are extremely hazardous and various stabilization or removal methods are discussed in the literature. Two representative cases will be addressed: stabilization of lead contaminated residues of oil industry and removal of contaminants from drinking water. Both cases are discussed in light of information derived from a combination of X-Ray Absorption Fine Structure (XAFS) and X-ray fluorescence (XRF) spectroscopies.

The Pb-contaminated sludge which accumulates in the storage tanks of oil industry is often disposed in burial sites posing a detrimental environmental impact. A cost effective and simple stabilization method is the incineration of the toxic sludge at elevated temperatures and the use of the resulting fly ash as a component in the production of industrial glass. It is established by XAFS that the vitrified samples immobilize effectively Pb which is successfully trapped into the vitreous matrix. Yet, incorporating high waste concentrations leads to the formation of micro-crystalline regions with varying stability and capacity to effectively immobilize Pb on the long run or under adverse environmental conditions. The use of a focused X-Ray micro beam (5 μm) permits the performance of μ-X-Ray Fluorescence (XRF) imaging of the two-dimensional distribution of the metals under question and the recording of μ-XAFS spectra from selected sample spots. The identified microcrystallites belong to either Fe- or mixed Fe/Pb-oxides embedded in the vitreous network; nevertheless, their presence has a detrimental effect on the stability of the end product, since Pb can easily escape from the Fe-depleted glassy matrix.

Conversely, the contamination of drinking water with several toxic elements is another important issue concerning public health. Iron (oxyhyd)oxides are studied as candidate filters in public water supply systems. The exact adsorption mechanism of the contaminants to the materials surface is addressed using XAFS spectroscopy. It is disclosed that chemisorption is the dominant adsorption scheme. Depending on the ppm concentration of the toxic element, the inner sphere complexes share edges and/or corners with the Fe-centered octahedra of the material's surface. Nevertheless, it is demonstrated that the ability to efficiently remove the toxic elements depends strongly on the type of complexes formed: the higher the number of oxygen atoms shared between the contaminant and the surface, the stronger the uptake mechanism.

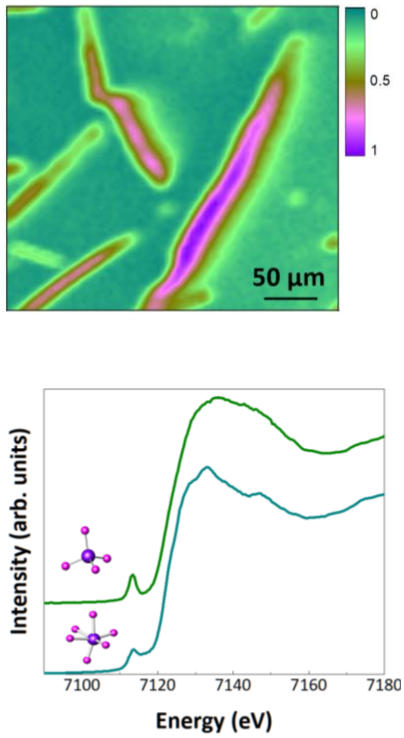


***Figure:*** *The distribution of Fe in a vitroceramic product with 60 wt% Pb-rich waste, as determined by μ-XRF mapping (top). Fe-K-μ-XANES spectra recorded from the Fe-rich micro-crystalline (violet) and Fe-poor glassy (dark green) regions (bottom).*

# Solid-State Chemistry through the prism of synchrotron radiation: Case studies in the field of high-performance halide perovskite semiconductors

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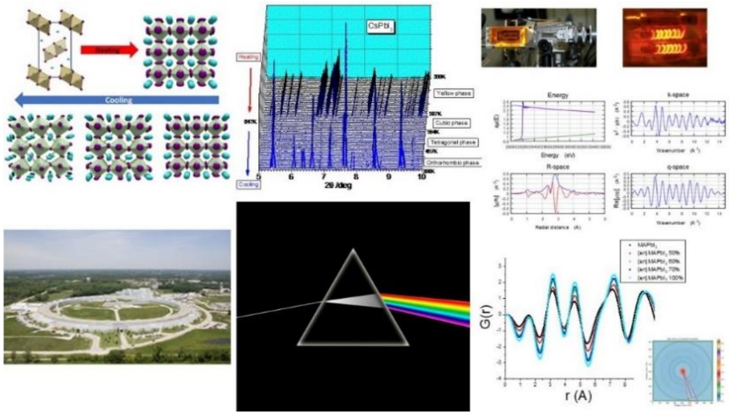
Synchrotron radiation and the plethora of experimental capabilities hosted in synchrotron facilities have played an instrumental role in the revolutionary developments of solid-state chemistry in recent years [1]. Among the multitude of materials that have been studied in this context, halide perovskites - the latest iteration of high-performance semiconductor materials- have been proven to be ideal specimens for synchrotron studies because of their diverse crystal chemistry and unusual spectroscopic properties [2]. Universally, halide perovskites are characterized by a variety of structural phase transitions at relatively narrow temperature and pressure ranges which can be studied using exotic methods that are readily available in synchrotron facilities. The combination of structural characterization and spectroscopic analysis at non-ambient conditions provide a powerful tool to extract structure-property relationships that are invaluable for designing new materials with desired properties [3].

In this talk, I will highlight notable experiments performed in synchrotron facilities (mainly in Advanced Photon Source, U.S.A.) on halide perovskites. I will discuss how synchrotron facilities provide the perfect platform in the study of the materials, enabling for "trivial" characterization methods, such as high-resolution X-ray diffraction, and advanced studies related to total scattering techniques. Whereas the former can provide a reaction map to navigate through the synthesis and crystal growth during a solid-state reaction [4], the latter can reveal hidden features of halide perovskites that arise from their dynamically disordered state [5]. Complementary to the structural analysis, synchrotron enabled X-ray absorption spectroscopy has also played a key role in understanding the electronic structure and the chemical bonding in selected perovskites, thus obtaining a complete picture of their chemistry and reactivity [6]. The technologically relevant thin-film techniques, such as GIWAXS imaging, can provide crucial information related to the morphology and the orientation of the perovskite in photovoltaic devices, thus providing an important tool towards boosting the devices' performance. *(7)* I will conclude by stressing out the importance of synchrotron accessibility as a means for promoting fundamental research and enabling technological breakthroughs towards industrial applications.



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# Nano-imaging of functional nanomaterials by spatially resolved X-ray diffraction

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We present spatially resolving X-ray diffraction-based tools than are of relevance for most emerging nanomaterials. There resolving power has proven to reach down to length scales relevant for catalysis and basic solid state chemistry as encountered in most electrochemical systems. While developments over the recent years were mainly affected by the rather slow evolution of basic X-ray equipment like optics and detectors, their level of maturity now leads to a limit set by the X-ray source itself. With worldwide efforts in the upgrading or new conception of synchrotron sources, improvements of several orders of magnitude in the throughput of these methods are expected in the next years. Our first results show the relevance of such new tools in complex experimental environments as encountered in nanotechnology and chemistry.

Although many basic principles and processes in what is in a wider sense called nanotechnology are known and exploited since more than a century, their development and improvement is still very much based on a trial and error approach. Many common characterization techniques cannot assess functional nanomaterials on relevant time and length scales. This maybe the case for electrochemical systems under reaction or for ferroelectric oxides during switching processes. We have developed several synchrotron x-ray diffraction and fluorescence based microscopes that have proven their usefulness in microelectronic samples where high strain resolution needs to be combined with large fields of view and sub micrometer resolution[1-3]. For typical nanostructures as present in catalytic reactions or batteries, coherent X-ray diffraction imaging tools allow to zoom into single nanocrystallites with the potential of 3D nanometric imaging. While most published examples of the recent past aimed at high spatial resolution in static samples, current efforts are aiming at the operando assessment at a time resolution of relevance in chemical engineering, while preserving a 3D spatial resolution well below 10 nm [1]. These tools that are today mainly limited to a small expert community are prone to quickly develop into one of the most powerful operando assessment methods; We will present the current state of the art of X-ray diffraction based microscopy with examples for imaging of individual nanostructures.

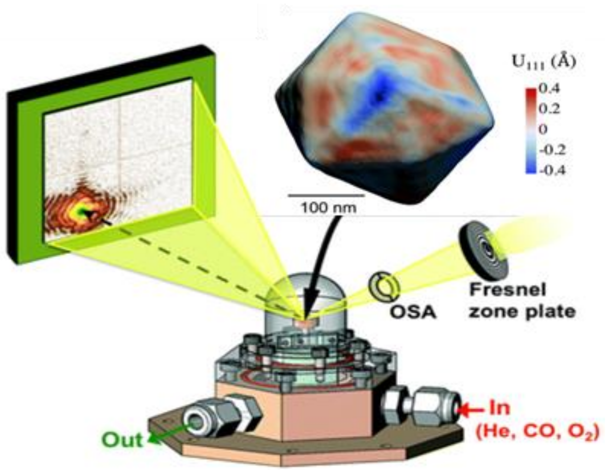


***Figure 1***: *Example for coherent X-ray diffraction and reconstruction of a catalytic nanoparticle during reaction (from ref. [3])*

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## Studies of Quantum Materials at the ESRF

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The ESRF has many capabilities for studying Quantum Materials and in particular magnetism, superconductivity and strongly correlated materials. A variety of x-ray techniques including x-ray absorption, x-ray diffraction and energy loss spectroscopies are available. With sample environments such as high-pressure, low temperature cryostats and superconducting high field magnets, the experimental possibilities are very wide.

Topics that have been covered in the past years include 2D materials, multiferroics, quasi-2D gases, topological materials, and Kondo phenomena.

A brief overview of the possibilities offered by the ESRF beamlines will be given. Some examples will be presented, in particular using the ID32 Resonant Inelastic X-ray Scattering (RIXS) spectrometer (see figure).

***Figure 1:*** *The 13 m Soft X-ray Resonant Inelastic X-ray Scattering (RIXS) spectrometer at ID32.*

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# Total scattering – a probe for all length scales that matter

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Quantum materials harbor exotic behaviors, including metal-insulator transitions, colossal magnetoresistive effects, topological insulators, frustrated magnetism, high-temperature superconductivity to name a few. Their remarkable functions, thanks to the emergence of collective behaviours that arise from strong couplings among the electron degrees of freedom (spin-charge-orbital) and the crystal lattice, appear crucial for developing electronic or photonic devices with great technological potential. In that respect there is great demand to selectively perturb microscopic interaction parameters in order to transform the materials into a desired state [1]. This may involve avenues ranging from conventional chemical substitutions (e.g., doping) to intense electromagnetic stimuli and even nanostructuring or interface engineering. So, the question is over what volume or length scale that a property averages the structure matters?

Local structural distortions matter in many low-dimensional materials but are rather difficult to detect with conventional low-energy powder diffraction techniques. Buried underneath the Bragg peaks, diffuse scattering signals due to disorder (cf. occupational or displacive) unveil information on how atoms bond and offer a view of how electronic states correlate [2]. Here is where we resort in utilizing total scattering methods, based on high-power, state-of-the-art synchrotron X-ray sources to detect subtle but crucial structural responses associated with microscopic quantum phenomena [3]. To demonstrate this powerful tool, we discuss case studies by applying a quantitative local structure probe, namely, the atomic pair distribution function analysis (PDF) technique. As a high-throughput approach, it can be implemented remarkably well in diverse materials systems such as ferrite nanocrystals that crystallize to a limited degree [4] and over to the time-resolved in-situ visualization of phase formation in intercalated superconductors [5], not easily observed by conventional analytical tools. It is evident that structure-property relations are much better understood with advanced synchrotron-based structural probes on materials when a local description of the electronic states is included [6].

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# The ESRF: a knowledge hub for industry

**Ed Mitchell**

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Since the start of user operations in 1994, the European Synchrotron Radiation Facility (ESRF) has actively worked and partnered with industry. Since then, the commercial programme has grown, and now encompasses:

- routine and tailored access for enterprises performing confidential research and innovation
- IPR valorisation through technology transfer and licencing of advanced instrumentation designs
- provision of off the shelf components for instruments
- provision of complex one-off instruments.

Synchrotron light, and particularly that of the ESRF-EBS could arguably be described as the means of our age for the ultimate characterisation of materials: such facilities provide the ability to visualise the atomic, nano-, and macro-structure of a huge range of complex materials, often under processing or end-use conditions and in real time. This capability lends itself to an equally wide range of industrial R&D problems which, in particular, have been adopted by the healthcare industry.

As the use of synchrotrons grows rapidly and beyond traditional academic user communities, new business models are springing to life, with more partnerships, more services, and nimble small start-ups bridging the gap between the light source research infrastructures and the commercially driven industry world. European funding has also woken up to this opportunity, and industry is now knitted throughout the Commission's funding programmes for access to facilities like the ESRF, but also in developing technologies to support advanced instrumentation.

This presentation will present and discuss the overall strategies being deployed in building engagement and impact in working with industry at the European Synchrotron Radiation Facility (ESRF), based in Grenoble France. The talk will look to both the current developments and future possibilities of business, as well as review several examples of partnerships between research and industry.

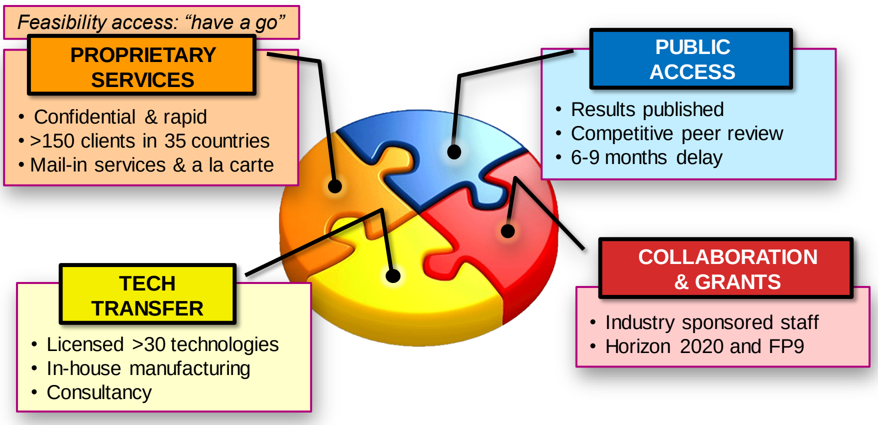


***Figure:*** *ESRF engagement with industry as a user of facilities and technology.*

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