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Adapting the European Synchrotron to Industry

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1. Introduction

In the 1990s, the European Synchrotron Radiation Facility (ESRF) became the first third generation synchrotron in the world, boasting new technologies and state-of-the-art beamline instrumentation. The ESRF welcomed its first commercial client in 1994, which used the new high brilliance beamline “BL4” at the time and now ID02 to collect structural biology data. Since then industry use of third-generation synchrotrons has grown at the ESRF and across similar facilities worldwide, demonstrating their relevance to supporting innovation and their impact in the ecosystem from commercial use supported sometimes with external funding in various ways [1–9]. As this use has grown, most synchrotrons have established industry liaison offices, staffed with industry contact officers who are often PhD qualified scientists experienced in synchrotron techniques and able to bridge synchrotron-to-industry, translating between the two worlds. This is complemented strongly by external bridges *via* research and technology organizations (RTOs) with their industry-heavy networks and collaborators, and by nimble specialized science service companies providing an entry point to research infrastructures.

In 2019, the ESRF started its long closure for the ESRF Extremely Brilliant Source (ESRF-EBS) upgrade [10, 11], and reopened in summer 2020 as the first high-energy fourth generation synchrotron with beam properties typically between 10 and 10,000 times enhanced depending on the X-ray technique. A game-changer for academic research, ESRF-EBS is also an unprecedented opportunity for industry, opening up entirely new vistas in the use of synchrotron X-rays for commercial research and innovation. The challenge is to harness the opportunities and adapt the synchrotron world to what industry needs, and not the other way around. This is not new, but with the increasing pressure from stakeholders for a more immediate return on their investments in large-scale research infrastructures, the need, interest, and motivation to engage constructively with industry now has a much different flavor than back in 1994.

In this framework, the ESRF Council, our highest governing body, instigated an annual commercial income target. Starting in 2016 at 1.6M€, the 2024 target is almost 2.8M€ and increases year-on-year by 5%, as set by the Council. This translates into a push to find new opportunities with industry, to reach out and engage proactively with

industry through various routes to our market, and to develop new services tailored to industry needs.

2. External support

Funding from stakeholders at European levels, the European Commission’s Framework Programmes, and at national levels, such as the French *Investissements d’Avenir* and France 2030, is increasingly directed by global challenges and societal needs—which industry can help to respond to and is indeed central to achieving translation of research into usable processes and products. With a market in continuous evolution and with novel challenges constantly arising, it is highly desirable that technical capabilities and offers at research infrastructures may also adapt rapidly to be in phase with industrial demand. This is essential to maintain strong and creative links with the industry and needs a further approach to business development. It is necessary to go beyond the simple promotion of existing services that may have been created without either industry input or industry interests at their core.

The Industrial Contact Offices (ICOs), Business Development Office at ESRF, at research infrastructures are the structures responsible for the actuation of the industrial programme of their respective facilities. The ICOs identify and anticipate future needs and are ready to develop novel services tailored for industry, with a particular emphasis on the unique opportunities offered by the next generation of high brilliance sources that will arise all around the world, following the successful upgrade of the ESRF. In this respect, a collaborative environment needs to be established where industry and ESRF can engage with each other. The creation of tighter collaborations is needed to: (a) identify coming needs; (b) develop, validate, and test novel pilot services and methods; (c) participate in harmonization and standardization working groups, to validate the use of light sources methods in approved industrial workflows and processes; (d) associate the light sources in the context of open innovation initiatives, innovation hubs, service platforms where selected institutions may join forces to provide a coordinated access to relevant characterization tools. A crucial role in this is played by the interaction of the ESRF with research and technology organizations (such as Fraunhofer, CEA, IIT, DTI, etc.) which work closely with industry and are trusted organizations by industry.

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This virtuous dynamic has been already acknowledged by public stakeholders. Clear indications were given in the ESFRI Roadmap and Sustainability Report [12]. As a consequence, a certain number of public funding instruments are already available to finance this kind of activity, mainly by the European Commission, *via* their Framework Programme, but also by national initiatives.

At the ESRF, leveraging the novel opportunities offered by the EBS, a business innovation initiative, was initiated to operate such a medium to long-term engagement action plan. This initiative is constituted by a team of business developers, charged with understanding the novel opportunities arising from the market, interacting with industry, defining potential business models, and identifying the most adequate strategies to exploit the prospects and run the preliminary promotional actions. Furthermore, this business development team coordinates with the relevant (and is in some cases dedicated) technical and scientific staff with the aim of bridging the potential technical gap between the industrial needs and the actual status of the technical infrastructure. A key point is that this team is wholly supported by external grant funding.

These grants have been built around a series of themes related to the main capabilities of the EBS: smaller beams, higher flux (operando/time resolved/high throughput experiments), higher performances at high energy (bulk characterization). The following grants have been mostly implicated in supporting the work: NFFA Pilot and NanoME-Commons in nanotechnology; IRT Nanoelec and RADNEXT on the characterization of nano-electronic components and on irradiation testing for the aerospace; TEESMAT and BIG-MAP on batteries; EASISTRESS on metallurgy and additive manufacturing; STREAMLINE on the development of routine high-throughput methods. Importantly, these proposals also support post-docs and engineers who can drive the technologies and methodologies for new industry services.

In particular, the ESRF, as a European Strategy Forum for Research Infrastructures (ESFRI) Landmark, secured a 5 M€ grant, “STREAMLINE” (streamline.esrf.fr; grant agreement no. 870313/Sustainable research at micro and nano X-ray beamlines), as part of the Horizon 2020 Framework Programme. As mentioned above, the ESRF-EBS upgrade has provided X-ray beams with significantly increased brilliance and coherence, and these enhanced X-ray beams permit breath-taking new science as well as significant gains in resolution or rapidity for existing experiments. Making these opportunities swiftly available to user communities, the STREAMLINE project aimed to adapt user operation procedures and to pilot new access modes and new services exploiting automation at the beamlines—particularly targeting industry for this latter—as well as supporting comprehensive outreach and training to user communities for the new ESRF-EBS enabled science.

STREAMLINE’s support has been instrumental in creating new, industry-oriented services. These services, detailed below, were conceived from the outset to address specific commercial needs. Such an approach is crucial when collaborating with industry, whether it involves conducting routine experiments, managing high-throughput measurements, or executing unique experiments that require complex

setups and sophisticated data analysis. By aligning closely with industry requirements from the beginning, common obstacles like mutual comprehension, cost, sample preparation, and data delivery are significantly reduced. This strategic alignment ensures a quicker adoption of the developed services.

3. Co-design and new service developments

As one can see, bringing the synchrotron to industry, and not the other way around, is critical in being attractive. Toward this, external funding has largely helped technology developments at the ESRF to co-design and create the instrumentation and workflows matching industry needs and expectations. Three examples are provided below.

3.1. High-throughput X-ray powder diffraction for materials development

X-ray powder diffraction (XRPD) is recognized as a critical technique in the structural characterization of materials, and it is especially vital in the development of advanced battery materials. With the advent of modern synchrotron sources, the acquisition times for XRPD measurements have been dramatically reduced to mere fractions of a second. To capitalize on this accelerated pace, the implementation of an automated, high-throughput sample treatment system has become necessary. This system is intended to match the rapidity of these experiments and maximize their throughput capabilities.

Within the framework of the STREAMLINE project, an automated sample-changing system has been developed in collaboration with an industrial partner, BASF Ludwigshafen in Germany, specifically for high-throughput XRPD measurements (Figure 1). Capable of processing several thousand samples in a single shift, this system is a product of combined efforts from sample environment specialists, data and workflow experts, and the beamline team. This innovation in high-throughput experimentation, achieved through a collaborative approach,

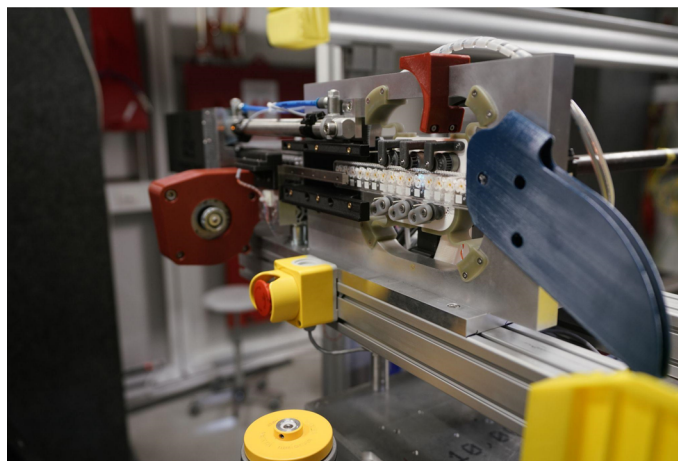


Figure 1: A photograph of the XRPD changer set-up as installed on ID31. X-rays come from right to left, with the sample holder and changer systems in the center of the photograph.

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is a significant advancement in material science research and its industrial applications.

To take full advantage of the ESRF-EBS and to satisfy the design demand from the industry, the sample changer was developed based on the following specifications: fast change from one sample to the next; an automatic identification labeling system for each sample and its results; fluidization of the powder; easy to charge sample racks and the capacity to run over 1,000 samples autonomously. The device was created, assembled, and installed at the ID31 high-energy beamline (which provides a very high flux of X-rays, a 2D detector, and a highly flexible experimental station with space for installing sample set-ups), dedicated to buried interface structure and materials processing studies [11].

A standardized, lightweight sample holder was designed and produced as a low-cost, injection-molded part. The holder can contain 16 samples in small, individual chambers, each with its own QR code. Each sample can be moved inside its chamber when a vibration is applied to fluidize the powder during data acquisition, thus providing excellent quality of the scattered signal. Injection-molded clips hold Kapton window foil seals, giving minimal background signal to the overall XRD pattern. It takes about 95 ms to move between samples in the rack and to read the QR code whilst the sample holder is vibrating. The sample holder is ejected into a tray after the 16 samples are measured, and the subsequent sample rack is then loaded. In this way, the system can fully automatically measure up to 66 sample holders (1,056 samples) within <1 h, depending on the chosen acquisition time. Attenuation of the beam to provide the highest data quality is done automatically, allowing samples with different scattering intensities to be measured in the same batches. Data are treated automatically online immediately after the measurement, generating an azimuthal integration with results stored in an hdf5 file with a name generated automatically from the sample QR code, allowing the user to download it remotely. Workflows make use of the ESRF workflow system (EWOKS, <https://ewoks.esrf.fr>).

The system speed makes synchrotron XRPD very cost-effective and scaling far beyond conventional laboratory techniques to enable screening, throughput, and quality which are not otherwise feasible.

Exploitation of the system is underway directly with the ESRF. In addition, and particularly exciting, is the creation of a start-up company, “Momentum Transfer” (<https://momentum-transfer.com>) which provides A-to-Z full service using the system. Momentum Transfer consults, prepares samples, supports logistics, and performs data analysis and analytics for clients, adding significant value to the X-ray technique as an expert science company and making the excellence of the ESRF more readily available.

3.2. High-throughput elemental analysis for the mining industry

The development of high-throughput X-ray fluorescence (XRF) was necessitated by the need for trace element characterization in commercial mineral reserves across a vast number of samples, numbering in the tens of thousands. Such a task was previously unachievable with existing characterization techniques. Given the large volume of sam-

ples, a key challenge in the project’s development was to enhance the efficiency of X-ray fluorescence measurement and to automate experimental processes. Promising initial tests conducted at BM23 led to the creation of specifications for a high-throughput characterization system, designed to provide quantitative data for precise contaminant detection. The introduction of the new ESRF-EBS light source has markedly improved data collection speed and detection limits.

The inception of the STREAMLINE project marked the beginning of efforts to extend this solution’s accessibility to a wider client base. A focus group, including engineers from the ESRF and experts from the University of Montpellier, was formed to refine the existing procedures and develop an automated system for broader application. Key contributions came from ESRF experts specializing in sample environment and experiment/data workflow, leading to the development of a prototype sample handling system. This system, featuring a robotic arm and a laser system for meticulous sample positioning, requires precisely calibrated geometry to ensure accurate, reliable, and reproducible alignment between the beam, sample, and detector. A versatile and cost-effective sample holder has also been developed, providing both reusable and disposable options to mitigate cross-contamination.

The equipment, hardware, and data acquisition workflows are now operational (Figure 2). The University of Montpellier has the capability to transform raw data into client reports, with efforts underway to further enhance data analysis and data reduction techniques at the ESRF.

The advent of the new EBS synchrotron source, capable of reaching detection limits down to parts-per-million, heralds significant opportunities for both industrial and academic sectors. This advancement is particularly promising for fields, such as mining, chemistry, and the accelerated discovery of new materials.

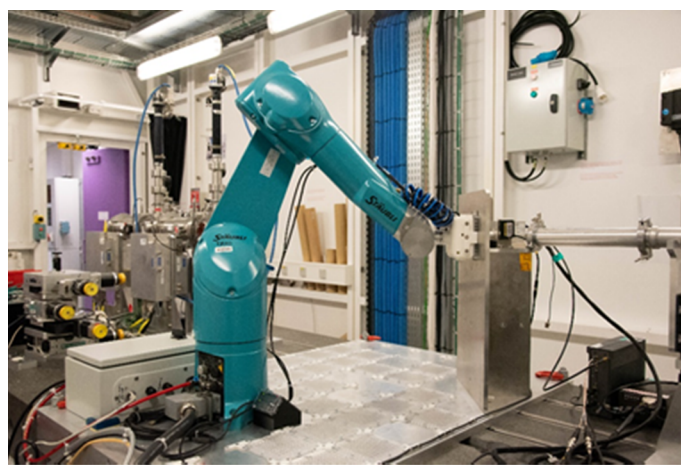


Figure 2: A photograph of the XRF set-up as installed on BM23. X-rays come from right to left, with the robot arm picking up the sample plates and holding it in the right position to complete the data acquisition. The full control on the position of the different elements of the setup enables the possibility of obtaining quantitative information.

3.3. New opportunities for aerospace radiation hardness testing

Traditionally, single event effects (SEE) qualification through heavy ion (HI) tests occurs at cyclotron facilities with energies around 10 MeV/nucleon. However, this method poses challenges. First, it requires demanding sample preparation to access the component's sensitive part, which may be impractical for emerging technologies like novel flip chip bonding or 3D packaging. Second, securing beam-time at these facilities is arduous due to overbooking, and third, the large beam sizes make pinpointing the exact location of the sensitive part challenging. Recent years have seen the validation of methodologies using photons (lasers) to generate electron-hole pairs, simulating the effects of HIs. However, this doesn't address the sample preparation challenge.

Pulsed synchrotron X-rays have proven effective in emulating HIs and testing electronic circuit sensitivity to ionizing radiation. High-energy photon X-ray beams ($1\text{ keV} < E < 30\text{ keV}$) can penetrate most metallization layers and possess a large penetration depth in the semiconductor, providing access to every embedded component within a 3D chip. Moreover, the X-ray beams can potentially be focused below one micron, offering unparalleled capabilities in debugging potential issues. Pioneering experiments conducted at the APS in Chicago (USA) over the last decade [13] have outlined the challenges, opportunities, and trends of using pulsed synchrotrons to emulate HIs and test electronic circuit sensitivity.

After the implementation of the EBS, ESRF now offers unmet capabilities for this important industrial user community. In particular, the ID09 beamline delivers intense X-ray beams with complex time structures. Since 2018, supported by French national funds made available in the context of the IRT Nanoelec initiative, the ESRF has embarked on an ambitious collaboration project with a consortium of users, including representatives from the National French Centre for Space (CNES), STMicroelectronics, TRAD, Nucleotide (Ariane Group) and AIRBUS, along with the Technology Research Institute Saint Exupery. This initiative aims to demonstrate the methodology's validity and prove its unique advantages, particularly for the most advanced and complex packages of the latest generation of devices, where stacked circuits or flip-chip mounting gain momentum.

The ID09 setup (Figure 3) proved to be compliant with the results obtained with the current mainstream qualification methods on well-characterized samples [14]. Preliminary work conducted so far has shown promising results for logic analog systems based on different substrates, including Si, SiGe, AlGaIn/GaN HEMT, and GaAs. Initial investigations have also been dedicated to power devices based on SiC and GaN.

3.4. Standardized stress/strain measurements

Residual stresses arise from processes like plastic deformation or rapid cooling after heating. Within a body in thermal equilibrium, residual stresses are not due to external forces, but they are the consequence of internal constraints. They can manifest in terms of macrostresses (extended

areas of the sample), and microstresses (found at the level of single grains or grain boundaries).

Diffraction methods, based on the Bragg equation, can be used to evaluate residual stress. Laboratory diffractometers with X-ray tubes allow surface residual stress determination, with a penetration depth of around 10 μm . Synchrotron-based methods offer greater penetration depth. Neutron diffraction achieves even larger penetration depth, though they have lower flux density, requiring longer measurement times, and lower spatial resolution.

Residual stresses influence various properties of mechanical parts, such as strength, fatigue behavior, and corrosion resistance. Understanding residual stresses is crucial for assessing a component's strength and lifetime. During operation, residual stresses and stresses arising from externally applied loads linearly add up to account for the total stress distribution in a component. Premature workpiece failure can be the result. However, in some cases, residual stresses can be beneficial as it is the case for compressive residual stresses near the surface that can enhance a workpiece's lifetime. Industrial components, such as generator shafts or pressure vessels, may have specifications regarding maximum residual stress values and permissible distributions. Residual stress analysis, especially using diffraction methods, may play a crucial role in optimizing material performance and ensuring the longevity of components in various industrial applications [15].

The ESRF is a partner in EASI-STRESS. This project, funded by the European Commission, aims to enhance the industrial adoption of non-destructive synchrotron X-ray and neutron diffraction-based residual stress tools [16] and contribute to the definition of an *ad-hoc* standard. Validating against destructive methods and developing protocols in collaboration with industry, the project is improving the understanding of residual stress formation and progression. By incorporating measured data into modeling tools, it enhances the design and lifetime assessment of metallic components, yielding more reliable products with increased lifespan and reduced material usage. Currently, conservative safety factors, defined by EUROCODE, are used. Knowing actual internal stress levels may allow for a 15% estimated materials cost saving. The ambition of EASI-STRESS is to develop a service platform where non-destructive residual stress mapping measured with synchrotron X-rays and neutrons can be offered on a commercial basis (Figure 4).

4. Outreach and awareness

The external funding has not only supported technology developments—these need to be backed with exploitation approaches, marketing, awareness-raising, and training/education. For example, marketing the ESRF toward industry through targeted outreach and awareness raising has been a core activity of STREAMLINE which allowed us to initiate an “ESRF meets Industry” outreach programme. During COVID, the use of webinars was essential due to travel restrictions, but once travel was open again the face-to-face contact and discussion could restart and a healthy balance between electronic/remote engagement and in person was established. During the final phase of the STREAMLINE project, and working hand-in-hand with other European funded projects, such

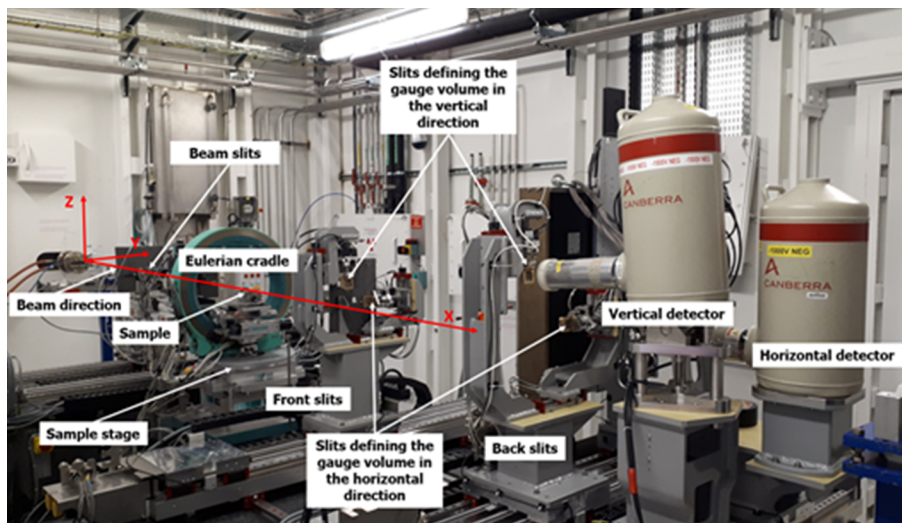


Figure 3: Typical single event latchup experimental setup on the beamline ID09 (courtesy of TRAD and CNES).

as LEAPS Innov (www.leaps-innov.eu) and ReMade@ARI (www.remade-project.eu), the ESRF Business Development Office team together with technical expert colleagues from the beamlines have been active in over ten industry or commercial events, be they workshops, conferences, or expos spread across diverse industry and technological sectors. This has created visibility and knowledge of the ESRF, of the capacities of synchrotron X-rays to support industry innovation, and particularly, of the EBS opportunities and new services. The direct impact of this outreach is difficult to measure—with much of the return being initially intangible, but delivering over time. Nonetheless, immediate returns as access demands, current clients stretching their interests, and new commercial clients are visible, and it is essential that outreach continues electronically, including social media, and in person. Selected success stories of ESRF working with industry can be found in Ref. [17].

From the standpoint of the ESRF, our commitment to fostering innovation extends robustly to small and medium enterprises (SMEs) through active participation in the TamaTA-INNOV initiative to support SMEs in using large-scale RIs. Recognizing the unique challenges faced by SMEs, including budget constraints and limited awareness of the transformative potential inherent in synchrotron facilities, ESRF positions itself as a facilitator and enabler. TamaTA-INNOV serves as a unique initiative, supported by the Horizon 2020 research and innovation programme, *via* projects CALIPSOplus and now LEAPS-INNOV, and aimed at empowering SMEs (see [18] for success stories from the initiative collected from across European light sources including ALBA-CELLS, DESY, ELETTRA, ESRF, KIT, MAXIV, and SLS). This includes leveraging ESRF expertise, infrastructure, and resources to de-risk SME access, ensuring that SMEs can harness the full capabilities of synchrotron facilities whilst keeping results confidential. The availability of this supported and subsidized access for SMEs has been seen to immediately lower barriers of cost and risk to SMEs and it will be important to understand how the programme may be continued in the future.

As part of our commitment to advancing innovation and supporting groundbreaking projects, ESRF has been selected to collaborate with the European Innovation Council (EIC) within the Ecosystem Partnerships and Co-Investment Support programme, offering specialised services to EIC beneficiaries. This partnership expands the EIC's business acceleration services, addressing key challenges in accessing sector-focused services and attracting private investors. ESRF, as an EIC Partner, can provide tailored synchrotron X-ray services for quick and remote materials and product characterization, workshops, consulting sessions, customized experimental solutions, and access to specific laboratory equipment. This collaboration allows ESRF to have a more direct route to deep tech companies, which would otherwise be challenging to reach out to. The pilot is just at the start, but is hoped that by being an EIC partner will enable a longer-term relationship with the EIC to be constructed and its beneficiaries.

5. Conclusions

Worldwide synchrotrons have become an indispensable driver of research and innovation. Their advanced capabilities generate critical data and insights into materials and biological matter, which are vital for the development of new products and processes. These innovations are key to addressing the pressing global challenges facing humanity. Consequently, it is imperative for these facilities to be accessible to industry and adapt to its rapidly evolving needs, especially in an era marked by accelerated development cycles and heightened competition.

Partnering with industry is not yet an inherent strength of research infrastructures—it requires energy and drive—primarily because these collaborations were not initially considered in their financial planning. Consequently, internal budgets are often insufficient for the tailored developments these partnerships require. Additionally, industries seem reluctant to provide initial funding for development to de-

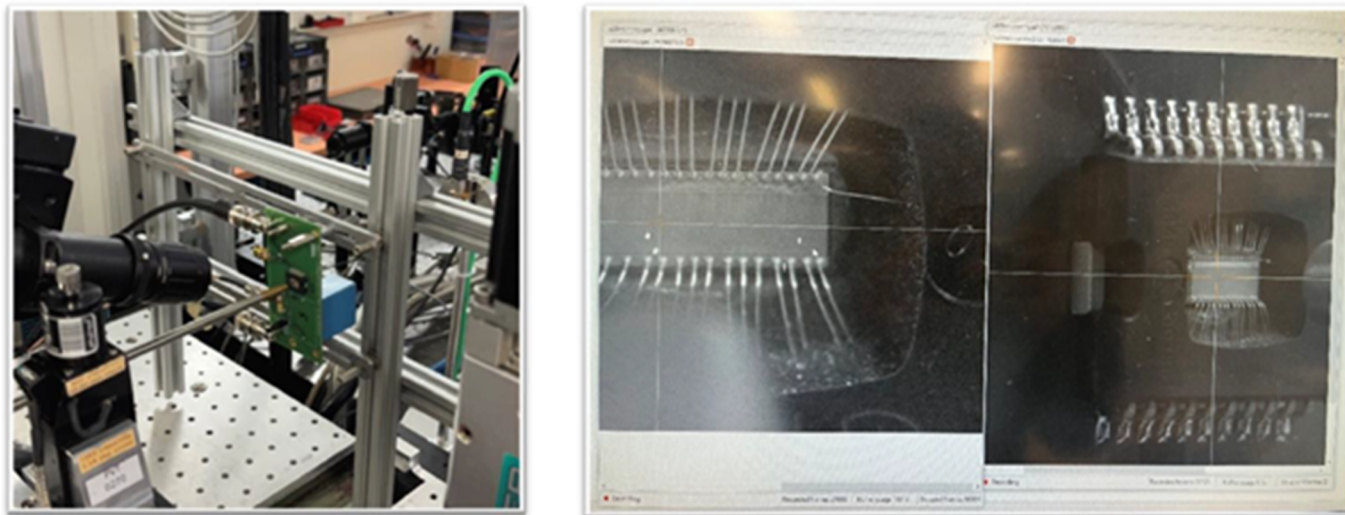


Figure 4: Typical energy dispersive X-ray diffraction experimental setup to measure residual strain on the beamline ID15. The residual strain maps obtained with this instrument can be transformed into residual stress measurements, using SOFT-AIS, an ad-hoc tool developed by EASI-STRESS.

liver the services they need being wary of the risks involved. To overcome these hurdles, external backing from funding agencies and industry-specific contributions are vital. These external resources, as demonstrated by our broad experience with funding from various European and national sources, can catalyze the potential of such partnerships. When strategically utilized, external support can magnify the impact and value of collaborations, far exceeding the initial investment.

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Disclosure statement

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