

GETTING TOUGH Industry platform launches radiation-testing facility

FAIR'S FAIR Data policy implemented across all ESRF beamlines

LONG AND SLOW Early mammals behave more like reptiles

LIFE AT THE LIMIT Pantaleo Raimondi on the success of the EBS upgrade

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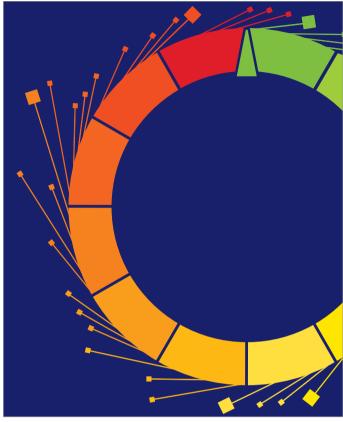
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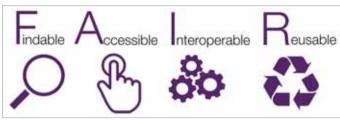
4Ch Focused Circular Through Hole Array with an Integrated OSA

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EDITORIAL

An Extremely Brilliant Source for society



Francesco Sette ESRF directorgeneral

On 25 August this year, the ESRF and its user community embarked on an adventure: the launch of user service mode (USM) with the new Extremely Brilliant Source (EBS). The ESRF is also welcoming an important cohort of young scientists, postdocs and PhD students, who have recently joined the ESRF–EBS adventure (p26). Training the next generation of scientists is at the core of the ESRF's mission and we are very proud to welcome them. Thanks to their enthusiasm, motivation and ideas, the ESRF can continue to provide a stimulating and inspiring environment of innovation and openness.

Of course, 2020 has been a very challenging year, which has led us to adapt our strategy and sometimes take rapid and tough decisions, both to maintain the USM restart date and to keep the ESRF safe for everybody on site. But the current COVID-19 pandemic highlights, more than ever, the importance of state-of-the-art research "The EBS is already providing a new vista for X-ray science"

infrastructure to enable the international scientific community to provide a rapid, collective response to the vital challenges facing our societies, such as health but also environment and energy.

As you can see in this issue, ESRF users have been making remarkable progress on these fronts, from understanding the longevity and safety of rechargeable batteries (p14) to developing new bioimaging techniques (p19). It is remarkable that, despite experiments being performed remotely, EBS performances are already providing a new vista for X-ray science.

Exciting times are ahead of us. On behalf of my fellow directors, the ESRF staff and myself, I would like to warmly thank all our partner countries for their constant support, which has been, and is, vital to keep science and innovation at the forefront for the benefit of the global scientific community and our society. I am looking forward to welcoming you on site soon.



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Alison Tovey Display advertisement manager Edward Jost Advertisement production Mark Trimnell Marketing and

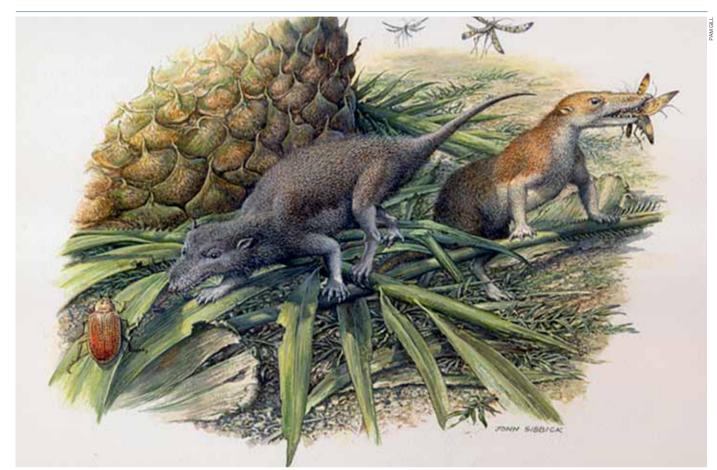
circulation Laura Gillham

ISSN 1011-9310

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December 2020 ESRFnews

NEWS



Early mammals lived long and slow

The earliest mammals led long but relatively inactive lives, much like reptiles, according to a joint study at the Swiss Light Source in Villigen and the ESRF. The study is the first time that palaeontologists have been able to study the physiologies of early fossilised mammals directly, via microand holo-tomography.

Much is known about the ecology, development and broad evolution of mammals in the Mesozoic era, 245 to 65 million years ago, when mammals first appeared. But very little is known about their physiologies, and in particular when their warmbloodedness developed. Warm blood allows animals to live highly active, if short, lives, and is thought to be key to mammals' evolutionary success.

Armed with some 200 teeth specimens taken from two Mesozoic mammals, *Morganucodon* and *Kuehneotherium*, researchers from the University of Bristol in the UK and the University of Helsinki in Finland Artist's rendering of *Morganucodon* (left) and *Kuehneotherium* (right) hunting in early Jurassic Wales, 200 million years ago. Below: 3D teeth rings from a *Morganucodon* specimen, as extracted by X-ray nanotomography.



came to three ESRF beamlines to learn how the mammals lived, using non-destructive imaging techniques addressing multiple scales. Like tree rings, teeth rings can reveal how long an animal has lived, and how active it has been during that time. At the former ID22 beamline, the researchers performed pilot scans via X-ray microtomography, before going to the ID19 beamline to retrieve more detail. Finally, holotomography at the ID16A beamline allowed the researchers to image key specimens with high energy, high contrast and at nanoscale resolution - a first for palaeontology.

The teeth rings showed that *Kuehneotherium* lived for up to nine years, and *Morganucodon* for up to 14. By contrast, their similarly sized furry successors, such as mice and shrews, tend to only survive a year or two in the wild.

"I was dumbfounded," says Elis Newham of the University of Bristol and lead author of the study. "It was thought that the key characteristics of mammals, including their warmbloodedness, evolved at around the same time. By contrast, our findings clearly show that - although they had bigger brains and more advanced behaviour – [the earliest mammals] didn't live fast and die young, but led a slower-paced, longer life akin to that of small reptiles, like lizards." Newham adds that Morganucodon and Kuehneotherium were otherwise typically mammal-like. "They had specialised chewing teeth, relatively large brains and probably had hair, but their long lifespan shows that they were living life at more of a reptilian pace than a mammalian one."

The Mesozoic animals did not laze about quite as much as reptiles, however. The size of the blood vessels in the thigh bones of *Morganucodon* indicated blood flow rates greater than lizards of the same size, albeit lower than modern mammals (*Nat. Commun.* **11** 5121).

ESRF supports COVID-19 actions

The ESRF has joined new initiatives to support COVID-19 research and to share data on the disease.

One, a joint initiative between the ESRF and the EMBL on the EPN campus, allows users to send a single proposal to remotely access both the EMBL's high-throughput crystallography lab and the ESRF's crystallography beamlines.

Meanwhile, the ESRF has endorsed the European Commission's "Manifesto for EU COVID-19 Research", which provides guiding principles to "maximise the accessibility of research results in the fight against COVID-19". These principles include making generated results public and accessible "without delay"; making scientific papers and "The European Commission's manifesto gives guiding principles to maximise the accessibility of COVID-19 research" research data available via open access following FAIR principles; and, where possible, granting limited-time, nonexclusive, royalty-free licences on intellectual property that has resulted from EU-funded research, in exchange for a licensee's commitment to rapidly and broadly distribute any resulting products and services that prevent, diagnose, treat or contain COVID-19.

The ESRF is already making data accessible according to FAIR principles under its new data policy (see Insight, p11). It will also adhere to the manifesto through the COVID-19 Data Portal, an online database run by the EMBL's European Bioinformatics Institute, with backing from the Commission and other partners. • www.covid19dataportal.org

ASD welcomes new director

Physicist Qing Qin of Peking University

"We're thrilled to have a new leader joining the ESRF directors' team who is taking responsibility for the core ESRF activity of producing brilliant and coherent X-rays," says Francesco Sette, the ESRF director-general. "This is a special time for the ESRF, in which we will consolidate the results of 10 years of upgrades, and enable a powerful scientific exploitation of the ESRF source by our users. With his experience, Qing will make a strong leader for ASD to get the best out of our new Extremely Brilliant Source. Qing – welcome on board!"

With a career spanning two decades, Qin has worked as a full-time professor at Peking University and at the Chinese Academy of Science (CAS) Institute of High Energy Physics (IHEP), as well as on CERN's machine advisory committee. He has contributed to the upgrade of the Beijing Electron-Positron Collider, its beam commissioning for collision modes and its use as a light source; he has also worked on the design of the future Circular Electron-Positron Collider, and the design and construction of the High Energy



Qing Qin has worked on the design of several major particle accelerators.

"With his experience, Qing will make a strong leader" Photon Source. Meanwhile, he has helped establish a strong collaboration with the ESRF for storage-ring lattice design and component conception and design for future synchrotron sources.

Qin, 53, began his new role with a five-year mandate on 1 November 2020. He succeeds Pantaleo Raimondi, the architect, engineer and scientist behind the new EBS storage-ring concept, construction and commissioning. "I take this opportunity to express the heartfelt gratitude of the ESRF to Pantaleo Raimondi for his dedication to keep the ESRF at the forefront of X-ray science with the EBS, and for leading the ASD for the last nine years," says Sette. "Thanks Pantaleo!"



User Meeting goes online

For the first time in its 32-year history, the ESRF User Meeting will be held remotely for users, to ensure the safety of them and ESRF staff in light of the COVID-19 pandemic. Freely accessible online between 8 and 10 February next year, the meeting will feature the same broad line up of learning and networking opportunities, all from the comfort of users' homes.

"Making the User Meeting an online event was a hard, if necessary, decision," says Michela Brunelli, the outgoing chair of the User Organisation Committee (see story below). "But we can assure users that there is just as much reason to attend – and perhaps more so, to learn about all the exciting opportunities ahead with the new Extremely Brilliant Source."

Keynotes at the meeting will be made by Pablo Beato of Haldor Topsøe in Denmark, Irene Margiolaki of the University of Patras in Greece, Letizia Monico of CNR-SCITEC in Italy, and Maximilien Ackermann of Mainz University and Danny Jonigk of Hannover Medical School in Germany. Meanwhile, microsymposia will be on the topics of structural studies of viral diseases, micro and nano X-ray analysis of biomineralisation, and the physics and chemistry of actinides probed by X-rays. The meeting will also include nine tutorials, the presentation of the Young Scientist Award, a facility report on the EBS and a poster session.

UOC welcomes new chair

Guillaume Morard has been named as the new chair of the ESRF User Organisation Committee (UOC). A planetary scientist at the University of Grenoble Alpes, Morard will lead the UOC in its promotion of ESRF research through discussions with the user community, in liaising between users and management, and in organising the annual User Meeting. He will take over from the existing chair, Michela Brunelli, in January (see User Corner, p12).



Gianluca lori (right) with ESRF scientist Alexander Rack at ID19.

BEATS scientist gets ESRF training

The beamline scientist overseeing the construction of a new tomography beamline at the SESAME synchrotron in Jordan has come to the ESRF for training. The programme for Gianluca lori is part of collaborative project, BEATS, to assist in the design, construction, assembly and commissioning of the tomography beamline.

Funded by the European Commission under Horizon 2020, BEATS (Beamline for Tomography at SESAME) is a consortium of nine institutes, led by the ESRF, which began last year and will last until the SESAME tomography beamline is ready for users in 2023.

A mechanical engineer and synchrotron user. lori has come to the ESRF for three weeks to meet scientists, engineers and staff, and to gain insights into the complexities and technicalities of building a beamline. One of his first roles as the BEATS beamline scientist was to contribute to the writing of the beamline's Technical Design Report, which was submitted to the European Commission in August. As well as co-ordinating the construction of the beamline. he will propose and co-ordinate an in-house scientific programme in close collaboration with the BEATS partners and the SESAME scientific director.

"It's a pleasure for the ESRF to welcome Gianluca," says Harald Reichert, ESRF director of research and member of the BEATS steering committee. "The ESRF is proud to play a role in co-ordinating the BEATS project and I can only stress how important it is for us, as a larger facility with almost 30 years' experience in synchrotron research, to help smaller facilities come into operation. As well as building scientific and cultural bridges between diverse societies, this project will benefit the scientific communities in the Middle East by providing them with the first tomography beamline in the region."



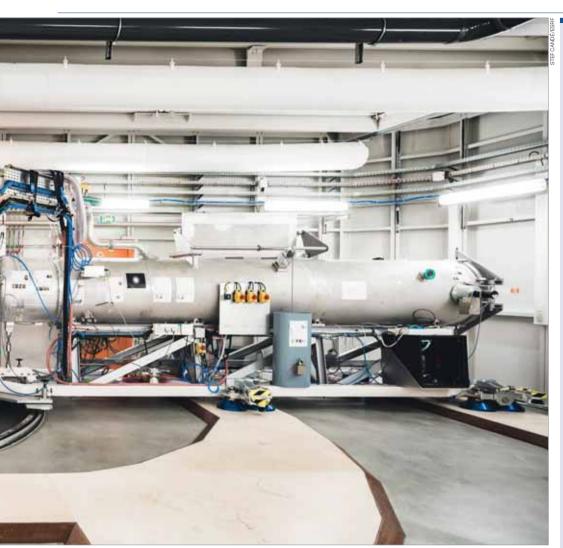
Heat-harvesting spintronic

An international collaboration of scientists has exploited unique instrumentation at the ESRF to discover how distortions within crystalline films create magnetic microstructures. The results are a first step towards creating a new, ecofriendly generation of "spintronic" devices that generate and process electronic charge from waste heat.

Spintronic devices are those that manipulate the spin, as well the charge, of electrons to process data. Their history starts in 1988 with the discovery of giant magnetoresistance (GMR), which exploits electron spin to detect tiny magnetic fields via huge changes in resistance. Back then, GMR allowed the capacities of harddisc drives to balloon from megabytes to terabytes, thus spurring the development of user-friendly desktops, laptops and vast data centres. Although today our smartphones and tablets are more likely to feature Bespoke instrumentation at ID01 (top) was crucial to obtain the results. electrically driven "flash" memory than a GMR hard disc, spintronics is still promising for the creation of smaller, more powerful and more energy-efficient processors and storage drives, based on the exploitation of waves of spins, known as magnons, in magnetic materials.

A particularly sustainable form of spintronics is spin caloritronics, in which electricity is generated from a heat current via a magnon-induced spin current. In principle, devices employing this "spin Seebeck effect" (SSE) require no energy to run but that harvested from waste heat. However, the efficiency of that heatto-electricity conversion is dictated by how easily the electron spins can travel through a material.

To understand that behaviour, researchers at the European Spallation Source (ESS) in Lund, Sweden, the University of Grenoble Alpes, the University of Wisconsin–Madison,



devices come a step closer

US, the Bavarian Academy of Sciences and Humanities in Garching, Germany, and the MAX IV Laboratory in Lund used the ESRF to correlate magnetic domains with the presence of crystalline structures in a prototype SSE film 20 nm thick. Crucial to the success of the experiment was the co-development with the ESRF sample-environment group of a cryogenic fridge specifically designed for the ID01 beamline - to cool the film to just 4K, as well as the co-development with the XMaS beamline of a phase plate to generate circularly polarised X-rays. "The final experimental set-up was totally unique [and] would not have been possible without the dedication of the ESRF teams," says Danny Mannix of the ESS, the principal investigator of the team's ESRF proposal.

The results at ID01 pointed to the presence of nanoscale magnetic

"By combining our experimental set-up with EBS X-rays. we can image with far greater resolution"

textures that inhibit the conversion of heat into electricity (Sci. Adv. 6 DOI:eaba9351). "More efficient devices could be created by controlling these magnetic inhomogeneities. possibly by control of the crystal structure," explains Mannix. "Our results are therefore a first step towards highly efficient spintronics for wasteenergy recycling."

They are also a taste of the even higher-quality data that can now be generated by the ESRF's EBS upgrade. "By combining our experimental set-up with the coherent, high-intensity hard X-rays delivered by the EBS, we can use coherent X-ray diffraction imaging methods to investigate spin-caloritronic materials with far greater resolution than we have [so far] been able to obtain," says Mannix. "We're really excited to explore the new possibilities provided by this advanced synchrotron source."

ID16A data backs **3D-printed alloy**

Laser-based 3D printing can create high-strength alloys with finer microstructures than those created by traditional rapid solidification. That is the conclusion of an international team of researchers who have made use of the ESRF's ID16A beamline to study a titanium-iron alloy by ptychographic tomography.

With laser-based 3D printing becoming ever more widespread, scientists are keen to develop materials with properties tailored to the technique's metallurgical conditions. Eutectic alloys - mixtures of metals having melting points lower than that of any of their constituents - have already been shown to develop ultrafine structures with good mechanical performance in small samples, but have otherwise received little attention.

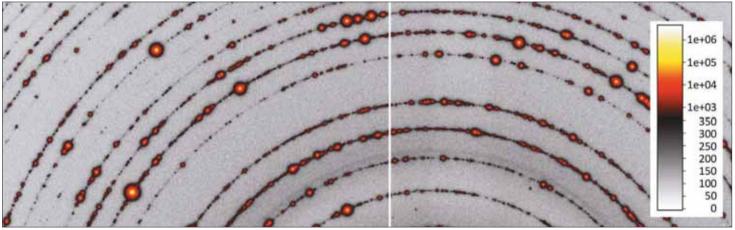
Guillermo Reguena of the German Aerospace Centre in Cologne, Germany, and colleagues have shed more light on the potential for eutectic allovs with a study of one such alloy, titaniumiron, produced by laser powder-bed fusion (LPBF). Near-field synchrotron ptychographic X-ray computed tomography with a resolution down to 39nm revealed the three-dimensional morphology of the internal structures, as well as their mass density (see image below), size and distribution. The researchers discovered that spacings between internal layers were as little as 30-50 nm - smaller than those obtained via traditional rapid-solidification of bulk materials (Appl. Mater. Today 20 100767).



 Meanwhile, in partnership with ESRF scientists, researchers at University College London and the University of Sheffield in the UK have employed ultra-fast X-ray imaging at the ID19 beamline to reveal the mechanisms of microstructure formation in 3D printing based on LPBF. The insights could improve the quality of LPBFmanufactured components (Appl. Mater. Today 20, 100650).

Advertising feature

Seven EIGER2 CdTe Detectors for the Extremely Brilliant Source



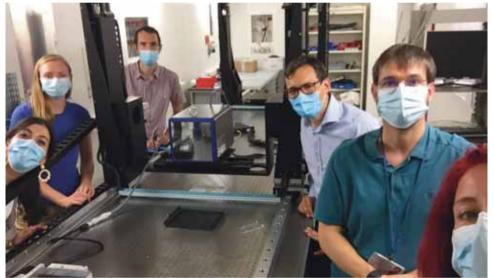
Powder data collected with an EIGER2 X CdTe system at the high-pressure beamline ID27, the sample containing large single crystallite species causing strong reflexes.

"The combination of the Extremely Brilliant Source and the EIGER2 X CdTe detectors' high dynamic range and high sensitivity will allow us to detect weak features in diffraction data, especially diffuse scattering and superstructure or impurity reflections", says Carlotta Giacobbe, beamline scientist at the ID11 beamline of the ESRF. "We are excited about the high frame rate of the EIGER2 X CdTe. Ultrafast three-dimensional mapping and ultra-fine slicing will now be accessible to monitoring systems as they evolve during *in situ* experiments".

The recent commissioning of an EIGER2 X CdTe 4M detector for the ID11 beamline is the first part of a continuing success story that will include the delivery of eight DECTRIS EIGER2 detectors in total, all of them ordered by the ESRF to exploit the power of the new Extremely Brilliant Source (EBS). Seven of these detectors will be large CdTe detectors with high sensitivity over the wide energy range that is available at many EBS beamlines.

The EIGER2 CdTe journey began in 2018, with initial tests of the detector systems at the ESRF. Beamline scientists were convinced by the features of the newly-developed detector family and its specifically designed readout chip, with frame rates up to 2 kHz and continuous (dead-time-free) readout; an enhanced photon count rate per area; and two thresholds per pixel, allowing users to suppress spurious fluorescence signals and higher harmonics; all packed into tiny 75 x 75 μ m² pixels.

From the variety of active-area geometries (500 K to 16 M) that are offered in this detector family, beamline scientists selected the optimal detector for their particular applications: coherent scattering, diffraction and imaging studies, biomedical imaging, high-resolution powder diffraction, macromolecular crystallography, high-



Happy faces in special times, during the commissioning of the new EIGER2 X CdTe 4M detector for ESRF beamline ID11.

pressure experiments, and small- and wideangle X-ray scattering. As the detectors start to arrive at the ESRF–EBS, beamline scientists, users, and DECTRIS are excited about the new experimental possibilities and the first data sets that have been produced.

As an innovator in synchrotron science, the ESRF is known for taking the leap into new technologies. As early as 2014, the ESRF ID15 and ID31 beamlines started using PILATUS3 CdTe detectors; this dramatically enhanced beamline capabilities for fast and scanning measurements and has transformed *in situ* PXRD, operando XRD-CT, and PDF techniques. Now, with the upgraded source and the advanced features of EIGER2 CdTe detectors, we expect another order-of-magnitude improvement.

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INSIGHT

New dawn, new data

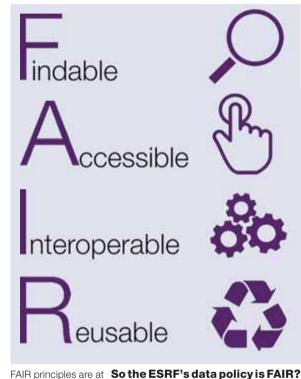
A new data policy is a big part of the return to user service mode at the upgraded ESRF–EBS, with big implications for users.

Why is there a data policy?

In short, because there is a lot of data out there. In the early 1990s, when user experiments began, the ESRF was generating tens of gigabytes of data a year; today, it generates some 30 terabytes every single day. Meanwhile, like the rest of the world, the ESRF has been caught up in a digital revolution, in which it has become possible to store, share and search vast quantities of data much more easily than ever before. Both these trends have given rise to the thinking that data can be analysed profitably not just by the experimenters who first record it. but also by other researchers, with faster and more rigorous scientific developments as a result. Indeed, many researchers and collaborations have already been sharing data in this way for some time: data policies such as the ESRF's are merely a way of doing so consistently and according to collectively agreed principles.

What are those principles?

Back in 2007, when large research infrastructures were first exploring the best ways to share data, the Organization for Economic Co-operation and Development named 13 underlying concepts, ranging from openness and transparency to security and sustainability. By 2016, however, more than 50 scientists had come together to distil these into just four "FAIR" principles: Findability, Accessibility, Interoperability, and Reusability. Very simply, "findable" means that data are accompanied by a set of universal metadata that both humans and computers can easily understand; "accessible" means that there must be well-defined terms dictating when, and for whom, a data repository is opened; "interoperable" means that data are written in a broadly understood language to make them reusable; and "reusable" itself means that data are presented in such a way as to help scientists apply them in different settings, or to replicate them.



FAIR principles are at the heart of the new ESRF data policy.

In fact, the formulation of the ESRF's data policy began back in 2008, when European photon and neutron facilities grouped to discuss the thorny issues at the heart of data sharing and management. In 2015, five years after that project was complete, the ESRF became the first light source in Europe to adopt a definite data policy based on principles today recognisable as FAIR – even though FAIR itself would come a year later. After the adoption, the ESRF software group spent a further two years identifying the technologies necessary to back up the policy - the use of ICAT as a database for metadata, for example - before steadily implementing them, beamline by beamline. Since last month, all the ESRF beamlines have been operating under the new data policy.

What does that mean in practice?

The biggest change is that the ESRF is now the official data custodian. In that role, it automatically generates

metadata, which it stores forever, and archives all raw data for 10 years. This is a huge undertaking, and one many believed impossible in early discussions, but has proved possible due to the fast-rising capacities of tape storage. For three years, the experimental group will have sole access to the data that it generates; beyond that embargo period, the data become freely available to anyone via the ICAT portal (online at https:// data.esrf.fr) by default - unless the experimental group submits a written request to extend the embargo to the ESRF directors of research.

How has the new data policy been received?

The FAIR principles have been embraced by much of the scientific community as part of a broader "open science" movement. Likewise, many ESRF users are enthusiastic about a FAIR-aligned ESRF data policy. Shared data could help to restore trust in science, making it easier for scientists to attempt to replicate one another's findings. The policy will also give funders more "bang for buck", pave the way for shared data processing and analysis services. and allow data once thought to be exhausted to be revisited in new contexts. Imagine, for instance, if past data of coronaviruses could have been revisited in light of the current COVID-19 pandemic.

Not everyone is on board. Despite the possibility of extending the embargo period, some researchers feel anxious that their hard-won data will automatically go public before they have published everything they ever intend to in journals. But for Andy Götz, head of the ESRF software group, the question at the heart of it all is one of duty. "Data are the ESRF's primary product," he says. "I've always thought something's missing if we don't curate data properly."

Jon Cartwright

USER CORNER

DATES FOR THE DIARY

- 15 January 2021 New
 31 January 2021 Ong
- 8–10 February 2021
- 20
- 1 March 2021
- 5 March 2021

New long-term proposal submission deadline Ongoing long-term progress reports deadline Online User Meeting (deadline for registration: 20 January 2021) Submission deadline for standard and BAG proposals for 2021/II Experiment report submission deadline for experiments from 2020/II

KEY CONTACTS

Users with questions, comments and ideas are welcome to contact the User Organisation Committee (UOC) at any time. Representatives of each scientific community and their contact details can be found at www.esrf.eu/UOC. ESRF contacts: www.esrf.eu/contacts.

Right: Guillaume Morard, chair of the UOC.



NEWS FROM THE BEAMLINES

NEW DETECTORS

Three post-EBS detectors have become operational: at **ID02**, an Eiger 2M; at **ID06-LVP**, a Pilatus 3 0.9M; and at **BM29**, an in-vacuum Pilatus 3 2M. Meanwhile, the commissioning of Eiger 2 4M detectors is underway at **ID10** and **ID11**, as is an Eiger 2 2M at **ID22**. Four more Eiger 2 detectors will be commissioned in coming months, at **ID17**, **ID15B**, **ID23-1** and **ID27**. The detectors will help users to better exploit the new X-ray source.

BM28 (XMaS)

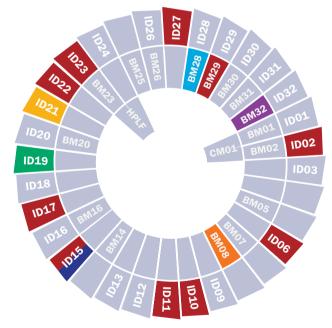
The LN2 double-crystal Si (111) monochromator has been retuned and recalibrated, the new IRELEC toroidal mirror system has been commissioned and the refurbished diffractometer has been installed. A focused beam at the sample position of less than 70 µm horizontally (<20 pixels FWHM) and 140 µm vertically (<38 pixels FWHM) at 8 keV is more than five times better than pre-EBS in both directions, boosting the flux density by at least a factor of 25. The beamline will return to full user operation in January.

ID21

A new X-ray nanoscope will be installed in the experimental hutch between now and April 2021, optimised for 2D nano-XRF and nano-XAS in the 2.1–11 keV range. The beamline will be closed this month until April 2021 for preliminary infrastructure work in the experimental hutch.

ID19

A new sample robot (see below) can now be used for remote experiments. The robot can either mount samples one by one, so that experimental parameters for each can be tuned prior to data collection, or it can be set to scan a series of samples of similar morphology automatically.



BM32

The pink micro-beam is back to its best size (300 nm vertically x 200 nm horizontally at FWHM) at the Laue micro-diffraction station, thanks to a refurbishment of the elliptical KB focussing mirrors. The former millimetric cross of damaged-mirror scattering around the microbeam has disappeared. Anti-scattering slits upstream of the KB will soon be added to further clean the microbeam's surroundings. Better control of the cooling and X-ray induced charging of the mirror coating by a "mirror protection system" should improve the mirrors' life expectancy. Close monitoring of the long-term ageing under irradiation of the mirrors using 2D mapping is planned.



ID15A

The beamline control system has been upgraded from SPEC to BLISS. Meanwhile, a new technique, multi-resolution X-ray diffraction CT, has been implemented. Thanks to the EBS upgrade, flux on samples has increased by a factor of 30 to 40.

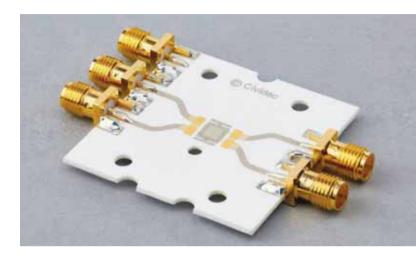
BM08 (LISA)

The beamline is open to users, following the successful completion of the realignment process and radio-protection tests in July. Although the integrated flux has remained the same, as expected from simulations, the EBS has provided a substantial improvement in the beam size at the focal point, which at $40 \times 70 \,\mu$ m is now half that given by the original ESRF source. This will have a considerable impact on all techniques needing a reduced beam size such as element mapping, measurements on small samples, data collection in grazing incidence for thin films and surface systems.



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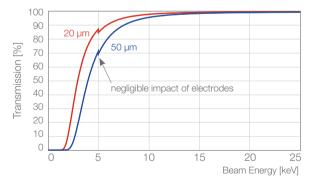


Figure 1: Transmission of the Diamond XBPM.

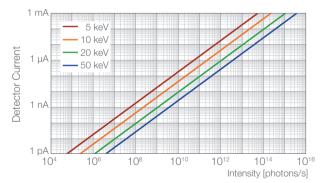


Figure 2: XBPM response as a function of the beam intensity.

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LUST FOR POWER

Newly boosted by the EBS upgrade, ESRF research is helping to satisfy demands for more advanced battery technology. ROM smartphones to personal health monitors, from renewable sources of energy to smart cities, from electric cars to drones – it can be hard to keep up with today's technological advances. Yet there is one technology whose performance underpins most of these others: the battery.

The importance of batteries to modern life is reflected in their growing market. Between 2010 and 2017, according to a report by the European Commission's Joint Research Centre, sales of the lithium-ion battery – by far the most dominant rechargeable battery technology – more than tripled, from €6.5 bn to €24 bn. They are set to climb much further in years to come. In parallel to this trend, however, are concerns about lithium ion's long-term sustainability. To meet the demands of the latest applications, including those helping shift us towards smaller carbon footprints, batteries need to charge faster and last longer, as well as be smaller, cheaper and safer.

These are issues that the ESRF's synchrotron techniques can help tackle. Instrumentation at the ID15A beamline enables scientists to image the insides of real batteries in working conditions, at high speed and in multiple dimensions. Meanwhile, the nano-probe beamlines allow scientists to study electrochemical reactions in extreme detail, again in operating batteries at ID16B, or in ultimate resolution at ID16A. Then there is the microtomography beamline ID19, which can show batteries failing catastrophically in super-slow motion, or the ID20 beamline, which provides a window into electronic and magnetic behaviour at record-breaking energy resolution (see "A battery of techniques", right). The EBS upgrade has given a turbo boost to much of this instrumentation. Fed with X-rays that are 100 times brighter and more coherent, the ID15A beamline, for example, is now home to diffraction imaging and tomography techniques of unprecedented time and space resolution. Indeed, as a measure of the importance of battery research to the EBS, one of the first images provided by the new EBS was a diffraction pattern of a battery taken at ID15A.

Donal Finegan, a chemical engineer at the National Renewable Energy Laboratory (NREL) in Colorado, US, uses ID15A to investigate ways of improving charging speed, which is widely considered one of the top three roadblocks—the others being charge capacity and cost—to the widespread adoption of electric vehicles. The first generation of electric vehicles in the 2000s had to be plugged in overnight to recharge; some of the latest designs, such as Tesla's Model S, are able to reach up to 60% capacity in half an hour. Now, however, industry is demanding 80% charge in just 10 minutes—about the time it takes to have a cup of coffee, and not too much longer than it takes to refuel a non-electric vehicle.

This is not an easy ask. Try to cram lithium ions into the negative, graphite electrode in which they are stored – that is, charge a lithium-ion cell too quickly – and they might not enter into the electrode at all, but mass on its surface. Lithium plating like this degrades the electrode, shortening the battery's lifespan. One solution, as employed in the Tesla Model S, is the use of special heaters to increase battery temperatures by 10 °C or so to improve the flow of lithium ions. The effect of this is limited, however, and it also wastes precious energy.

Finegan believes that the first step to improve charging speeds is to better understand how lithium plating occurs. That requires a technique able to study, at high speed, the lithiation, or charge, of a battery during a 10-minute recharge. "The ESRF is the leading synchrotron facility in the world," Finegan explains. "It has the highest flux in the world, which means the highest speed imaging, especially on ID15A."

Science at speed

Before the ESRF closure for the EBS upgrade, Finegan and his colleagues at the NREL, the University of Oslo in Norway, University College London in the UK and the Massachusetts Institute of Technology in Cambridge, US, used high-speed X-ray diffraction at ID15A to discover – at half-second intervals – how an entire graphite electrode's structure changed in response to fast charging. Published this year, the results suggested that tiny highways engineered into an electrode could encourage lithium ions to dive deep, rather than pile on the surface (*Energy Environ. Sci.* **13** 2570). "The same technique could help us test these potential solutions in the future," notes Finegan.

While practically important, however, the speed of charging is not always why lithium-ion batteries make the headlines. In recent years, explosions of lithium-ion batteries have been reported in various settings, including Samsung's Galaxy Note 7 smartphone, the Boeing 787 Dreamliner passenger aircraft, and several brands of electric scooters and e-cigarettes. In 2017, together with colleagues at NASA's Johnson Space Center in Texas, US, University College London and elsewhere, Finegan used the ESRF's ID19 beamline to characterise, at more than 20,000 frames per second, how six different designs of lithium-ion battery with the common "18650" form factor vent materials in the event of failure (see fig. 1, p16). The researchers found that the batteries explode partly due to the materials clogging in the battery corners, and that the simple inclusion of a bottom vent - in

A BATTERY OF TECHNIQUES

ESRF battery research extends to more beamlines than just ID15A, ID19 and ID16A (see main article). In the past year, for example:

• Victor Vanpeene at the Institut National de la Recherche Scientifique in Quebec, Canada, and colleagues have used *in situ* X-ray tomography at ID16B to understand why a new breed of silicon-based electrodes for lithium-ion batteries are prone to cracking (*Adv. Energy Mater.* **9** 1803947).

• Chunguang Chen at the Jülich Research Centre in Germany and colleagues have used full-field diffraction X-ray microscopy at ID01 to study the same phenomenon (*Nat. Commun.* **11**, 3283).

• Together with Marcus Fehse and colleagues of the Dutch-Belgian "DUBBLE" beamline, the ESRF's Christoph Sahle and colleagues have developed X-ray Raman scattering spectroscopy at ID20 as a straightforward way to measure how charge transfers within battery electrode materials (*J. Phys. Chem. C* **123**, 40, 24396).

"Before our work, virtually nothing was known about what happens inside batteries during failure. We're still the only ones doing these experiments"

addition to the single vent usually found on the top of a cell – can avoid the worst ruptures (*Adv. Sci.* **5** 1700369).

For one of Finegan's NASA co-workers, William Walker, linking exterior calorimetric measurements to a radiographic video of what is going on inside a failing battery provides "light-bulb moments" for his team. "The videos are helping us characterise the velocity of internal propagation as the cell materials fail, and how this may influence the heat we measure on the outside," he says. "We had never seen this before. It gives us more confidence to say why one failure event produces more heat than another."

Indeed, according to Finegan, some of the major manufacturers of 18650 cells now employ dual vents. "Virtually nothing was known about what happens inside batteries during failure before our work," he says. "Logistically, these high-speed imaging experiments are challenging, and we're still the only ones conducting them."

Building on abrupt failure, the gradual degradation of lithium-ion batteries is studied at the ESRF by Yang Yang and Peter Cloetens of the ID16A beamline, in collaboration with the SLAC National Accelerator Laboratory in California, US, Virginia Tech in the US, and other institutions. This year, building on data collected at SLAC, the researchers used hard X-ray phase-contrast nano-tomography to observe how the breakaway of nickel-manganese-cobalt particles from

the carbon matrix of an electrode leads to a loss of capacity over time – a problem every owner of a laptop or smartphone is familiar with. Unlike the only other technique able to observe electrode particles within their carbon binders – electron microscopy – the phase-contrast nano-tomography at ID16A gave a complete view of the degradation, without having to cut up any samples. The results cast doubt on a widespread assumption that smaller batteries are likely to have longer lives than bigger ones (*Nat. Commun.* **11** 2310).

Studies like these will help manufacturers develop the next generation of lithium-ion batteries. Equally, it may well prove in a few years to come that alternative battery technologies will need to be considered to meet the demands of future applications (see "Beyond lithium", opposite). With the mass of data and competing interests out there, knowing which route to take will be difficult -but perhaps artificial intelligence (AI) can help. That is the thinking behind a new project under the banner of the European Commission-supported BATTERY 2030+ initiative. Known as the Battery Interface Genome Materials Acceleration Platform (BIG-MAP), the project is a collaboration between more than 30 universities, research facilities and companies that aims to turbocharge battery innovation through the comprehensive, AI-led analysis of research outcomes. With €450 k of the total €20 m in

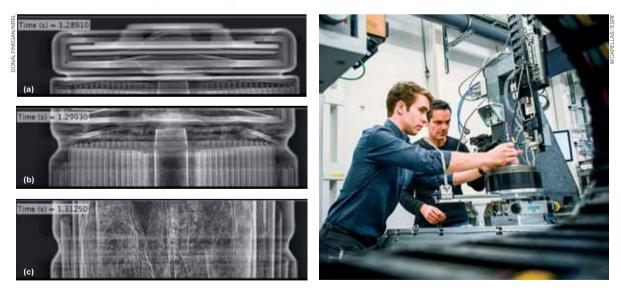


Figure 1 Above left: The 18650 is one of the most common form factors of lithium-ion battery, but it is not always reliable. High-speed radiography at the ESRF's ID19 beamline shows, in super slow-motion, how one sample fails catastrophically. (a) Before failure, the top of the battery consists of a number of components that make up a vent, which is designed to release gaseous material in the event of excess internal pressure. (b) About 1/100th second later, the failure is such that the internal electrode assembly has shifted upwards to clog the vent. (c) Another split-second later, the assembly and other contents have been been ejected violently. Above right: Finegan at work at ID15A on another experiment with Marco di Michiel, scientist in charge of the beamline.

BEYOND LITHIUM



Despite its recent success, lithium-ion battery technology faces some hurdles for much wider-spread adoption that are arguably insurmountable – not least the finite supply of lithium in the Earth's crust, which is expected to run out in just a few decades if projections of electric-vehicle uptake are correct. For that reason, many scientists are exploring other possible technologies. One of these is the magnesium-sulphur battery, which has abundant raw materials and potentially 10 times the energy density of lithium. In 2017, at the ESRF ID26 beamline, Ana Robba of the National Institute of Chemistry in Ljubljana, Slovenia, and colleagues studied the bulk electrochemical reactions within a working magnesiumsulphur battery cathode for the first time with resonant inelastic X-ray scattering and X-ray absorption spectroscopy, to shed light on why the emerging technology loses capacity fairly quickly (*Chem. Mater.* **29** 9555).

Another challenge with alternative battery materials is the development of electrolytes that are reliable and environmentally safe. Room-temperature ionic liquids – salts that remain molten well below $100 \,^{\circ}\text{C}$ – are a promising solution, because they are stable,

conduct ions well, do not easily set on fire and hardly evaporate. However, their suitability for batteries depends on the structures that they form at their interface with air. In 2018, Julia Haddat at Barllan University in Israel and colleagues employed synchrotron X-ray reflectivity at the ESRF, DESY in Hamburg, Germany, and the APS in Illinois, US, to determine the liquid-air interface structure at submolecular resolution of a broad series of room-temperature ionic liquids for the first time (*PNAS* **115** E1100).

Solid-state electrolytes are another route towards safety in conjunction with higher-charge capacity. Studies of these can now benefit from a totally new technique at the ESRF: dark-field X-ray microscopy. Offered for the first time in a general user programme at the ID06 beamline, dark-field X-ray microscopy allows scientists to produce a detailed map of the strains inside a solid-state battery during rapid charging. "By mapping the strains during rapid charging, we can get the fundamental understanding that we need to mitigate failures in the next generations of this promising battery technology," says Hugh Simons, a materials scientist and ESRF user at the Technical University of Denmark.

funding, and supervised by Harald Reichert, the ESRF director of physical sciences, scientists at the ESRF will feed the AI system with systematic studies of the structural, morphological and physio-chemical properties of battery materials.

AI is not a total stranger to scientific research. It is already employed to predict protein structures from genomic data, to predict the effects of climate change in specific localities and to discover new extrasolar planets in noisy astronomical data, to name but three examples. But according to Ennio Capria, the ESRF deputy head of business development, the use of AI to rank material systems, with all their unique attributes, is unprecedented. "The whole point is that it will be chemistry-neutral," he says. "The AI platform will be capable of analysing an enormous quantity of data provided by different sources to suggest new solutions. That will accelerate the discovery of novel systems for the battery of the future."

Lasting just three years, the project is not long enough for the full AI system to be built, but Capria hopes that he and others in the collaboration will be able to demonstrate the concept in a small-scale pilot, based on data collected during the project combined with that already in existence. The work will complement another European project, TEESMAT, in which the ESRF and other facilities are pooling their resources to help manufacturers characterise different battery technologies.

There is a lot in store for battery research for the upgraded ESRF. "All this dynamic is the result of an ambitious design," says Capria. "And that design is the EBS."

Jon Cartwright





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Table of Elements



Brain storm

A developing technique at the ID16A beamline promises to blow away microscopy limits with super-fast imaging of neural circuits.

T took 10 years, and tens of millions of dollars, but by 2018 it was complete: a three-dimensional image of an entire brain at nanoscale resolution. Led by a team of researchers at the Janelia Research Campus of the the Howard Hughes Medical Institute in Virginia, US, the result was hailed as a tour de force for electron microscopy – a breakthrough in scientists' efforts to visualise neural circuits.

This was no human brain that was imaged, however. In fact it belonged to a specimen of the common fruit fly, *Drosophila melanogaster*. Had the researchers wanted an equivalent image of a *Homo sapiens* brain, they might have been waiting not 10 years, but 10 million years. Such

NEURAL IMAGING

is the drawback of volume electron microscopy: time. The technique involves dividing a sample into slivers about 500 times thinner than a cell, taking hundreds of snapshots of each slice, then painstakingly stitching together the tens of millions of two-dimensional images that result into a three-dimensional composite. Even for a fruit fly, the Janelia group leader Davi Bock told the journal *Lab Animal*, the goal presented "a huge problem".

What if the same imagery could be obtained in days, or hours, at a fraction of the cost? According to ESRF scientist Alexandra Pacureanu, this is not wishful thinking but a definite possibility with the latest synchrotron imaging technology. With backing since March from the European Research Council (ERC), she has been developing a technique to three-dimensionally image large biological samples at nanoscale resolution - fast. Known as X-ray holographic nano-tomography (XNH), the technique promises to elevate studies of neural circuits from one to multiple specimens, and from insects to mammals, far sooner than anyone had anticipated. This would help scientists to learn how collections of neurons in the human brain give rise to thoughts, cognition and behaviour, and how their malfunction leads to neurodegenerative diseases, such as Alzheimer's, Parkinson's and multiple sclerosis.

"For over half a century, the dense mapping of neuronal wiring and the resolving of synaptic connectivity has been the sole domain of electron microscopy," says Wei-Chung Allen Lee, a neuroscientist and a collaborator of Pacureanu's at Harvard Medical School in Massachusetts, US. "XNH provides a new avenue."

"XNH could enable scientists to learn how collections of neurons give rise to thoughts, cognition and behaviour"

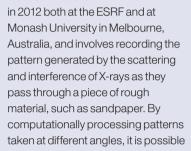
For those scientists coming to the ESRF for nanoscale three-dimensional imagery, the usual destination is one of the X-ray nanoprobe beamlines. Even before the synchrotron's upgrade to the Extremely Brilliant Source (EBS), the ID16A beamline was the world-leading instrument in hard X-ray nano-imaging, able to resolve features in three dimensions as small as 30 nm at energies greater than 20 keV. Thanks to the exceptional sensitivity of X-ray phase-contrast, even subtle variations in electron density, such as those occurring within soft tissue, can be resolved. And thanks to the highly penetrative nature of hard X-rays, there is no need to slice up samples.

The problem for neuroscience has been the size of its images. Meaningful neural circuits are millimetres in size or bigger, while the synapses connecting neurons become clear only at resolutions of 30 nm. To capture a single neural circuit, therefore, researchers need to create a huge image made up of 30 to 300 trillion voxels, which is why Pacureanu is currently working with ESRF scientists and engineers to upgrade the imaging detector and related

A ROUGH TOOL FOR FINE SCIENCE

Advances in

physiological imaging often require novel instrumentation (see main text), but one new technique can be performed with an optical element most people have in their toolbox: sandpaper. Known as specklebased imaging (SBI), the technique was first demonstrated



to reconstruct the three-dimensional density distribution of a soft-tissue sample in high detail. Now, at the ID19 beamline, Marie-Christine Zdora of the University of Southampton in the UK and colleagues have shown just how powerful SBI can be, by obtaining the microstructure of a

hydrated mouse kidney with a density resolution of more than 2 mg/cm³ and a spatial resolution better than 8 µm (image above). "We expect that SBI virtual histology will find widespread application in biomedicine and will open up new possibilities for research and histopathology," the researchers say (*Optica* **7** 1221).

instrumentation at ID16A. Meanwhile, a major challenge of the ERC project is to improve XNH's resolving power, which is determined equally by aspects of instrumentation – such as advanced X-ray focusing optics and precise, active mechanical stabilisation systems – and by strategies for data collection and image reconstruction.

This year, together with neuroscientists at Harvard Medical School, the University of Washington in Seattle, US, and Janelia, as well as Peter Cloetens, the scientist in charge of ID16A, Pacureanu used XNH to image both mammalian and insect nervous tissue, including part of a fruit fly, to expose the neural wiring. (A single-scan volume rendering of the insect's brain is shown on p19.) Although the resolution was less than the 2018 Janelia study, it covered a larger volume (*Nat. Neurosci.* DOI:10.1038/s41593-020-0704-9). More importantly, data collection took just two days.

In fact, the data was taken just before the ESRF shutdown in December 2018. According to Pacureanu, the greater coherence and flux of the synchrotron's EBS upgrade opens the door to XNH data collection with better resolution and more than an order of magnitude greater speed. That raises the possibility of being able to image, for example, an entire mouse brain at synapseresolution – a goal shared by a US government-backed project, at a cost upwards of \$100 m (€85 m).

There is also the tantalising prospect of combining insights at ID16A with those at larger scales at other ESRF–EBS beamlines. "Until now, it has been impossible to link this range of scales," says Pacureanu. "The ESRF–EBS has the potential to revolutionise neuroimaging."

Jon Cartwright

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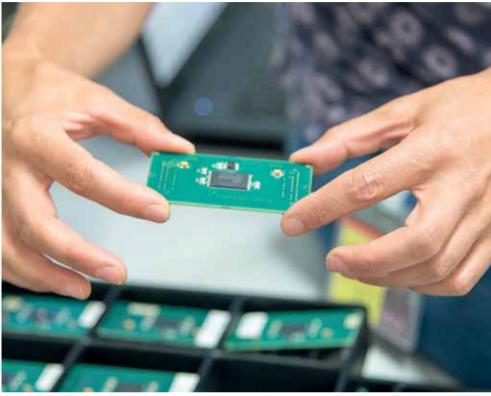
Making electronics hard

The ESRF leads the expansion of a platform to test radiation-hard electronic circuits.

It comes from the ground and from outer space; it is in the food that we eat and the air we breathe. Fortunately, humans suffer little directly from this natural background of ionising radiation. But what happens when an ionising particle zaps an electronic device? And what happens when that device is one in a high-risk setting – an autonomous car on a busy road, for instance, or a server that is handling a financial transaction?

This is a new focus of the Technological Research Institute (IRT) Nanoelec, an initiative of the French government to promote innovation in the micro- and nano-electronics sector through public-private partnerships. The ESRF has just taken on co-ordinating the materials characterisation side of the initiative, and will oversee the expansion of the Platform for Advanced Characterisation Grenoble (PAC-G) for the testing of radiation damage, using the combined X-ray and neutron facilities of the ESRF. the Institut Laue-Langevin and the Laboratory of Subatomic Physics and Cosmology. "In some applications we cannot afford any failure," says Ennio Capria, the ESRF deputy head of business development and the director of the characterisation programme. "But as we rely more and more on integrated electronics, that will become harder to achieve - unless we can make radiation proofing routinely affordable."

IRT Nanoelec began in 2012, and until now has focussed on four areas of electronics expected to becoming increasingly important: three-dimensional integrated circuits, silicon photonics, gallium-nitride power components and cyber-security. According to Capria, there are at least



"In some applications we cannot afford any failure. We need to make radiation proofing affordable"

two reasons for adding radiation-hard circuits to that list. The first is that, faced with the steep cost and limited range of dedicated radiation-hard components, satellite companies have increasingly been trying their luck with conventional off-the-shelf components in the hope that the resultant savings will exceed the cost of any failures. Here, radiation testing is needed to show in advance which components are likely to survive the harsh environment of space, and which will not. The second reason is the lowering of the acceptable failure rate in terrestrial applications due to ionising radiation be that radiation naturally occurring, or deliberately generated by cyber

A state-of-the-art Airbus electronic circuit was the first device to be radiation tested on the ESRF's ID09 beamline, as part of the PAC-G centre.

criminals to create faults in hardware and thereby extract secured codes.

Today, the PAC-G centre collaborates with four industrial partners with bases in Grenoble: STMicroelectronics, Soitec, Schneider Electric and SERMA Technologies. It has also collaborated with companies farther afield, such as Airbus, which in 2018 was the first company to use the ESRF's ID09 beamline to run an irradiation test on a state-of-the-art three-dimensional device. "The pulsed hard X-rays available at the ESRF deeply penetrate materials and can induce 'single event effects' (SEEs) in a whole stack of semiconductor layers," says Cécile Weulersse, the Airbus researcher who performed the study. "The method is a promising and convenient alternative for future SEE tests."

Jon Cartwright

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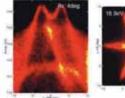
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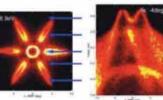
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PORTRAIT

Life at the limit

Pantaleo Raimondi, inventor of the HMBA lattice, reflects on the success of the ESRF upgrade.

Throughout his life, Pantaleo Raimondi has loved extreme sports - rock climbing, surfing, skiing, even paragliding. "You name it, I've done it," he says. Not that he has had much time to spend outdoors lately. For the past nine years, as the director of the ESRF accelerator and source division, he has spent every waking hour overseeing an entirely different extreme venture: the creation of the world's first high-energy fourth-generation synchrotron light source. "There is a parallel," he says. "In sport, you are always looking upwards. to the next possibility. That is what we have done with the ESRF-EBS."

Nine years may seem a long time to spend on a single project. In fact, for Pantaleo the EBS upgrade of the ESRF has been more like an entire career in the making, starting in the mid-1980s with his postdoctoral work – and his entry into accelerator physics - at the Italian National Agency for New Technologies, Energy and Sustainable Economic Development (ENEA). From there he jumped to and fro across the Atlantic, including stints at the Stanford Linear Accelerator Center (SLAC) in California, CERN's Large Electron-Positron (LEP) collider on the Franco-Swiss border, and Italy's national institute for nuclear and particle-physics research in Frascati, the INFN-LNF, where he headed up the SuperB B-meson factory project. During all this time he developed various novel ways to manipulate particle beams, for example the "crab waist" technique, which exploits a simple geometry of sextupole magnets to focus electrons in tight spaces.

He finally settled at the ESRF in 2012, where he designed the hybrid multi-bend achromat (HMBA) – the accelerator lattice at the heart of the EBS. "That was the product of all my previous experience," he says. A step-change in synchrotron design, the HMBA lattice is based on seven bending magnets per cell, unlike

the original ESRF's two, all of which bend electrons of different energies in the same way. The result is a 30-fold reduction in horizontal emittance, and a 100-fold improvement in X-ray brightness and coherence. A complex and revolutionary design, the HMBA has inspired the construction of other big light sources around the world - such as the APS-U at Argonne National Laboratory and the ALS-U at Berkeley Lab in the US, Spring8-2 at the Japan Synchrotron Radiation Research Institute, and the Shanghai Synchrotron Radiation Facility in China - and won Raimondi a prestigious Gersch Budker Prize from the European Physical Society Accelerator Group in 2017. It "shows Raimondi's ability to foster new ideas, his deep understanding of accelerator physics and [his] mastering of technological aspects", the prize jury said at the time.

Two months after the successful restart of user service mode at the new ESRF-EBS in August this year, Pantaleo's time at the light source came to an end: he hands the baton to accelerator physicist Oing Qin of Peking University in China (see news, p7). Now he can enjoy a little well-earned time for sport and cooking – he remains a southern Italian at heart - as well as seeing the first fruits of his design emerge. "Everybody has been astonished by the preliminary experiments," he says. While he insists that he loves to witness developments in all areas of science equally, he admits that it will be particularly gratifying to see the results from experiments that exploit the EBS's uniquely small beams and high coherence. Of course, no-one can predict what those results might be. "It is a dream machine, but it is also a machine that allows you to dream," he says.

Jon Cartwright



BORN: 1961, Bisceglie, Italy

EDUCATION: PhD theoretical physics, the ALEPH detector at the Large Electron Positron (LEP) collider, CERN (1986).

CAREER: Physicist, ENEA-Frascati, Italy, (1987–1991); research associate, SLAC (1991–1993); physicist, ENEA-Frascati (1993–1994); physicist, SLAC (1994–2002); head, accelerator division, INFN-LNF, Frascati (2002–2005); faculty professor, SLAC (2006–2007); director, accelerator division, INFN-LNF (2007–2012); director, accelerator and source division, ESRF (2012–2020).

"It's a dream machine, but it's also a machine that allows you to dream"

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Clockwise from top: Kudakwashe Jakata from South Africa explores materials science at BM05; Peng Li from China works on conventional and Bragg ptychography at ID13; Eleanor Lawrence Bright from the UK delves into uranium materials at ID11.





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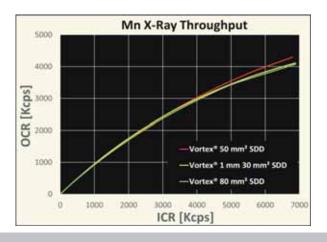
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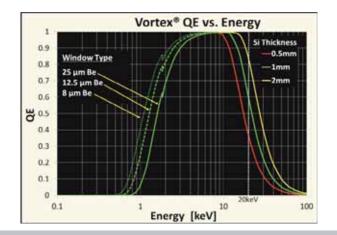
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