ESRF news

March 2020

It's here

The EBS shines a new light for science

ESRF

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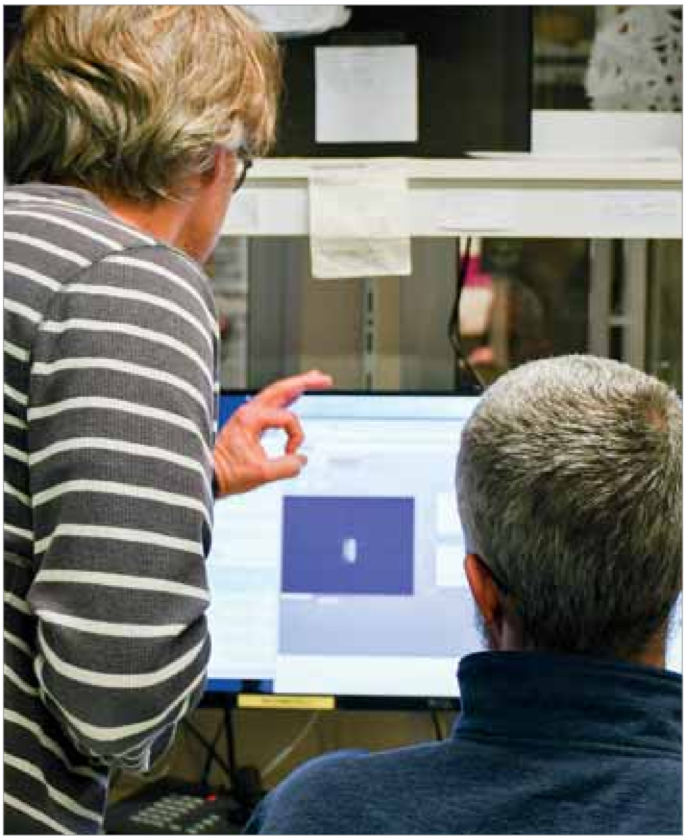

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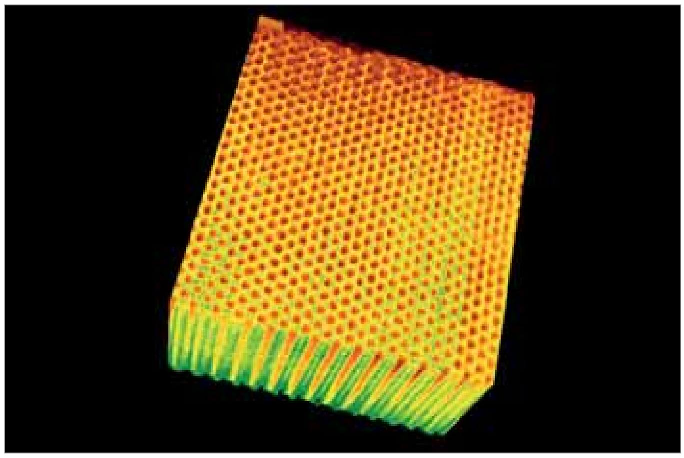

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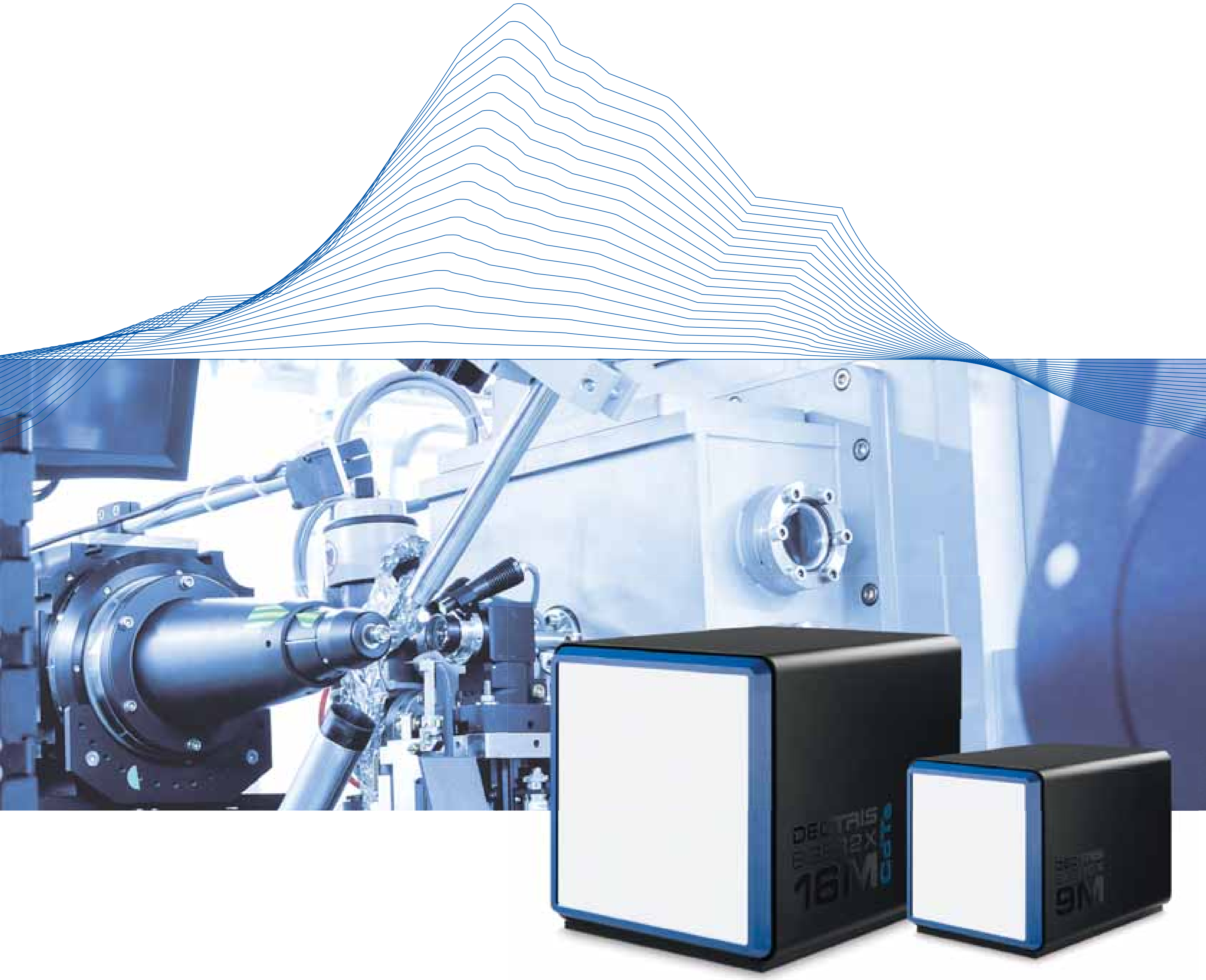

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A dream come true

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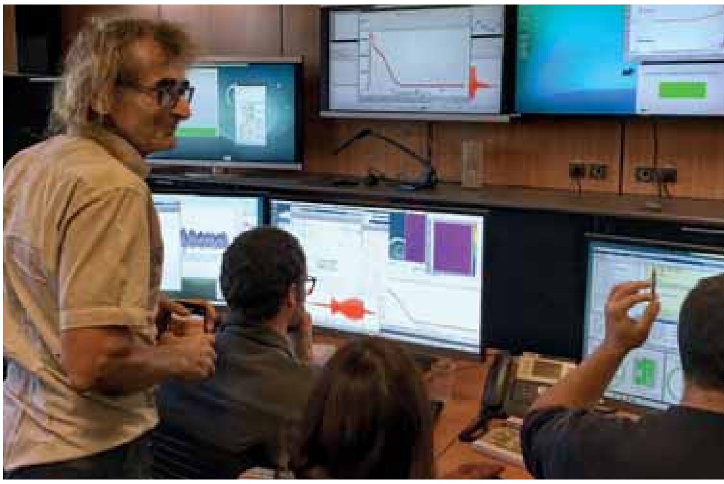
Pantaleo Raimondi
Accelerator and source director

In 2013, the Extremely Brilliant Source (EBS) was still a dream, a concept imagined on paper. Today, thanks to the expertise and enthusiasm of staff who have designed, developed, installed, tested and tuned around the clock, we are preparing to restart the beamline programme.

An intense commissioning phase has seen teams constantly and tirelessly pushing to achieve better and better machine parameters and increasing beam performances, from the injection of the first electrons at the beginning of December to the first stored electron beam just five days later.



"We have confronted immense challenges to implement a world-leading light source"

More recently, as you can see from the cover of this issue, an outstanding accomplishment and a key milestone in the EBS upgrade was achieved, as the beam parameters required to restart the beamlines were obtained – one month ahead of schedule – and X-rays were directed into the ESRF's Experimental Hall once again (see News, p6).

There have been great achievements and emotional moments, and it is with enormous pride that I take this opportunity to thank the teams that have worked so hard, with exceptional skill and dedication, to make this dream a reality. We have confronted immense challenges – technical, scientific and logistic – along the way to conceive and implement a world-leading source that will help to produce new science that tackles global and societal challenges, and improves people's lives.

Soon we will hand over this innovative new tool to the international community of synchrotron scientists and users. I am excited to see the science that will result.

ESRF news

Editor
Jon Cartwright
Tel +44 (0)117 2303080
E-mail jon.a.cartwright@icloud.com

Editorial committee
Nick Brookes
Delphine Chenevier
Andy Fitch
Michael Krisch
Gordon Leonard
Joanne McCarthy
Edward Mitchell
Pantaleo Raimondi
Harald Reichert
Francesco Sette
Jean Susini

Contributor
Anya Joly

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Head of media
Jo Allen
Production
Alison Gardiner
Technical illustrator
Alison Tovey
Display advertisement manager
Edward Jost

Advertisement production
Mark Trimnell
Marketing and circulation
Laura Gillham

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D CHENEVIER/ESRF

EBS beams hit their targets

The commissioning of the world's first next-generation, high-energy synchrotron light source has taken place at unprecedented speed, ready for the commissioning of beamlines and experimental testing by select ESRF users.

The first electrons were injected into the storage ring of the ESRF's new Extremely Brilliant Source (EBS) on 1 December last year. Just five days later, electrons were circulating continuously. Then on 16 December, EBS teams had managed to accumulate electrons in the ring with a horizontal emittance – a measure of how tightly packed the electrons are – of 308 pmrad, a world record. By the end of January, the electron beam was routinely circulating with a current in the region of 150 mA and an emittance in the region of 170 pmrad. At that point, the EBS teams opened up the front ends of the 27 insertion-device beamlines at 5 mA beam current: on all but one beamline, X-rays were found within a fraction of a millimetre of their positions before the start of the shutdown.

"The adventure has started"

"The ESRF–EBS meets our best expectations," says Francesco Sette, the ESRF director-general. "My thanks to the ESRF staff, whose outstanding expertise, dedication and involvement on the EBS conception and implementation since its launch in 2015 have made this new and revolutionary X-ray source a reality. The adventure has started."

The shutdown to install the EBS began in December 2018. Since then, EBS teams have had to remove the original 1 km-long machine, replace it in the confines of the same infrastructure and align a further 5 km of beamlines. "And it's working straight away – that's pretty amazing," says Harald Reichert, ESRF director of research for the physical sciences.

Indeed, as the ESRF accelerator and source director Pantaleo Raimondi pointed out to audience members at the ESRF User Meeting (see story opposite) on 4 February, the pace of commissioning means that the ESRF has not been without a beam in any year since its original launch in 1992. Raimondi – who is both the inventor of the EBS concept and the leader of the upgrade project – referred to the commissioning as "a once in a lifetime moment".

The achievement won praise at the User Meeting from outside the ESRF, too. "On behalf of the entire light-source community, I want to congratulate you all," said Caterina Biscari, director of the ALBA Synchrotron in Spain and the chair of the League of European Accelerator-based Photon Sources.

The new ESRF–EBS is set to begin full user operation on 25 August this year.

Users look to the future

Excitement pervaded the packed auditoria and meeting rooms of the ESRF's 30th User Meeting from 3 to 5 February – the last before the launch of the new Extremely Brilliant Source (EBS). In a typically varied programme, some 290 attendees discussed everything from the history of X-ray crystallography to the future of nano-engineering, but with a special emphasis on the unprecedented opportunities on offer at the upgraded and world-leading facility.

In a timely example of the relevance of synchrotron science to society at large, Stephen Cusack, the head of the European Molecular Biology Laboratory (EMBL) in Grenoble, kicked off the plenaries on Tuesday 4 February with an expert insight into the use of structural biology to tackle the threat of viruses. While the world's media focusses on the spread of the novel coronavirus that originated in Wuhan, China, at the turn of the new year, Cusack reminded listeners that the virus at the heart of his studies – influenza – still results in thousands of deaths every year in France alone and could result in a lot more when the next pandemic strain emerges. The long-standing partnership in structural biology between the EMBL and ESRF on their "world-leading campus" has, he noted, been integral to furthering our understanding of the mechanism of transcription by the influenza polymerase.

Cryo-EM clarity

Although not a coronavirus, influenza is, like coronavirus and most other human viruses, based on RNA rather than DNA. During transcription, the influenza polymerase uses its genomic RNA as a template to synthesise viral messenger RNA – which is translated into viral protein – before generating genome copies. These genome copies, and the viral proteins, become packaged into new influenza virions that can infect other cells and organisms. Via X-ray crystallography and single-particle cryo-electron microscopy (cryo-EM) – the latter of which, he said, "enables remarkably high-resolution structures to be obtained from even messy samples" – Cusack and his colleagues have been able to snapshot the influenza polymerase during active transcription. The work is enabling them to create a "molecular movie of the polymerase machine in action", which could help in the design of new anti-influenza drugs.

"It's a totally unique mechanism… You cannot believe how beautiful these structures are," he said. Cusack concluded his talk with reference to the greater threat of viruses to humanity – not just "known unknowns" like when the next influenza pandemic will be, but also "unknown unknowns" like HIV, SARS and the new coronavirus, which have emerged without warning. "That's why we need structural biology, to help figure out how they work and develop anti-viral drugs," he said. "The combination of cryo-EM and crystallography is incredibly powerful."

Later on the Tuesday, Kirsten Jensen, nano-materials scientist at


P.JAYET/ESRF


P.JAYET/ESRF

Top: Users listen intently to news about EBS commissioning. Above: Stephen Cusack, head of the Grenoble outstation of the European Molecular Biology Laboratory, discusses his work on influenza.

High demand for cultural heritage

More than 150 people registered for a workshop on cultural and natural heritage in January to explore the increasing use of X-rays to explore objects from the past – be they million-year-old fossils or fragments of impressionist paintings. The participation indicated an as-yet untapped demand to study the topic using ESRF instrumentation, with 60% of registrants being non-users and a particularly high turnout at an optional introductory talk about the main techniques and the best way to write experimental proposals.

The workshop was split into seven sessions: two on palaeontology; two on materials, processes and operating chains; one on conservation and alteration; one on inks and papyrus; and one on deep and machine learning. The talks gave recent examples of applications of synchrotron techniques, and described what will be possible with the ESRF–EBS upgrade, including the new source, new beamlines and new instrumentation. Of great interest to attendees was the future EBSL3 beamline (formerly BM18), the depth-resolved diffraction setup at the ID22 beamline, and access models and data policy.

● The discovery of *Australopithecus sediba* and other human ancestors has been named by *Smithsonian Magazine* as one of the "top 10 scientific discoveries of the decade" for its huge importance to natural heritage. Lee Berger, a paleoanthropologist at the University of the Witwatersrand in South Africa, and father of Matthew Berger, who found the first fossil of *A. Sediba*, has in the past made use of ESRF X-ray scanning facilities to characterise the species (below).

M CAPELLAS/ESRF

Young Scientist 2020 announced

The ESRF's Young Scientist of 2020 is Donal Finegan, a researcher specialising in lithium-ion battery degradation at the National Renewable Energy Laboratory in Colorado, US.

"The ESRF has played such a critical role in my research over the past seven years," he said upon receiving the award at the ESRF User Meeting in February. "The Young Scientist Award represents a tremendous honour for me, and is also testament to the outstanding beamline scientists with whom I've worked and who facilitated the cutting-edge techniques for my experiments."

Lithium-ion batteries, which are used in everything from consumer electronics to medical devices, electric vehicles and even satellites, are known to fail. Finegan, 29, attempts to understand their degradation mechanisms at different length scales, with an aim to improve their performance and safety. "At the moment, my job is split between studying the safety aspects of batteries, which I do at beamline ID19, and their performance limitations, which I do at ID15A," he said.

Shortly before receiving the award, Finegan published a paper highlighting the application of X-ray diffraction computed tomography to quantify how chemical defects within and between individual electrode particles affect a battery's performance (*Nat. Commun.* **11** 631). "This opens up new opportunities to understand the cause of battery degradation," he explained, "since many degradation mechanisms are related to sub-particle crystallographic phenomena."

"I'm very excited about the ESRF–EBS upgrade," he added. "The ESRF will be the leading facility worldwide for fast and high-resolution imaging."

the University of Copenhagen, and a "rising talent" in the eyes of the international L'Oréal-UNESCO programme for women in science, described the problem of properly characterising materials at the nanoscale, and how X-ray scattering in conjunction with pair distribution function analysis is helping to solve it. There were also keynote talks by Caterina Biscari, the director of the ALBA Synchrotron in Spain, about her chairing of the League of European Accelerator-based Photon Sources (LEAPS) initiative, and an introduction to imaging with coherent X-rays – which sees a major boost in the EBS (see *ESRFnews* March 2019, p19) – by Vincent Favre-Nicolin of the ESRF's X-ray nanoprobe group.

With the deadline for experimental proposal submissions on 2 March, many users were especially interested in Tuesday's facility reports, and what capabilities will be available in coming months. Initial reports by Francesco Sette, the ESRF director-general, and Pantaleo Raimondi, the ESRF accelerator and source director, on the advanced status of EBS commissioning received a very warm reception, and made way for communication on topics of more immediate relevance to users: beamline statuses, control systems and data. A particular challenge for users, according to the head of the ESRF's software group, Andy Götz, will be (if they have not done so already) learning Python, "the *lingua franca* of scientific programming" and the basis of the new BLISS control system. "It's a steady learning curve, and there's lots of tutorials out there," Götz reassured users.

"There's lots of tutorials out there"

Delivered after the announcement of the Young Scientist Award (see story left), the final long lecture of the Tuesday was by Elspeth Garman, an expert in radiation damage at the University of Oxford in the UK, on the belatedly recognised contribution of Rosalind Franklin to the discovery of the DNA double helix in the 1950s. "I am, of course, most ignorant about all things biological, but I imagine most X-ray people start that way," Franklin said at the beginning of her X-ray diffraction studies at King's College London in the UK – a sentiment shared by many newcomers to today's light-source community. She always followed her heart in matters of science, Garman noted, and was apt to directly confront senior male colleagues when cornered – traits that led to her unusual success for a female scientist of the day.

Activities plenty

The User Meeting was a valuable opportunity for users to become more informed about current trends in X-ray science, learn new skills and make new contacts. Besides Tuesday's plenary sessions, they could choose from eight practical and theoretical tutorials on Monday, and on Wednesday participate in three microsymposia: *in situ* and *operando* X-ray absorption spectroscopy for the study of catalysts and functional materials; the life-cycle of nanomaterials; and multi-crystal and serial data collection in structural biology.

Indeed, the first User Meeting for the world's first high-energy, fourth-generation synchrotron source was, as the chair of the ESRF User Organisation Michela Brunelli said, "an opportunity to make the science real".

P JAYET/ESRF

Pair distribution function analysis helps to characterise nano materials, explained Kirsten Jensen in her keynote talk.

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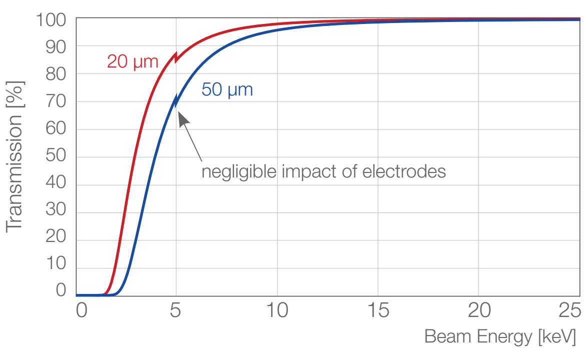

Figure 1: Transmission of the Diamond XBPM.

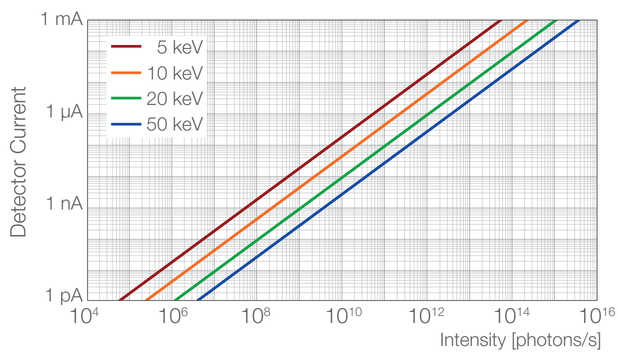

Figure 2: XBPM response as a function of the beam intensity.

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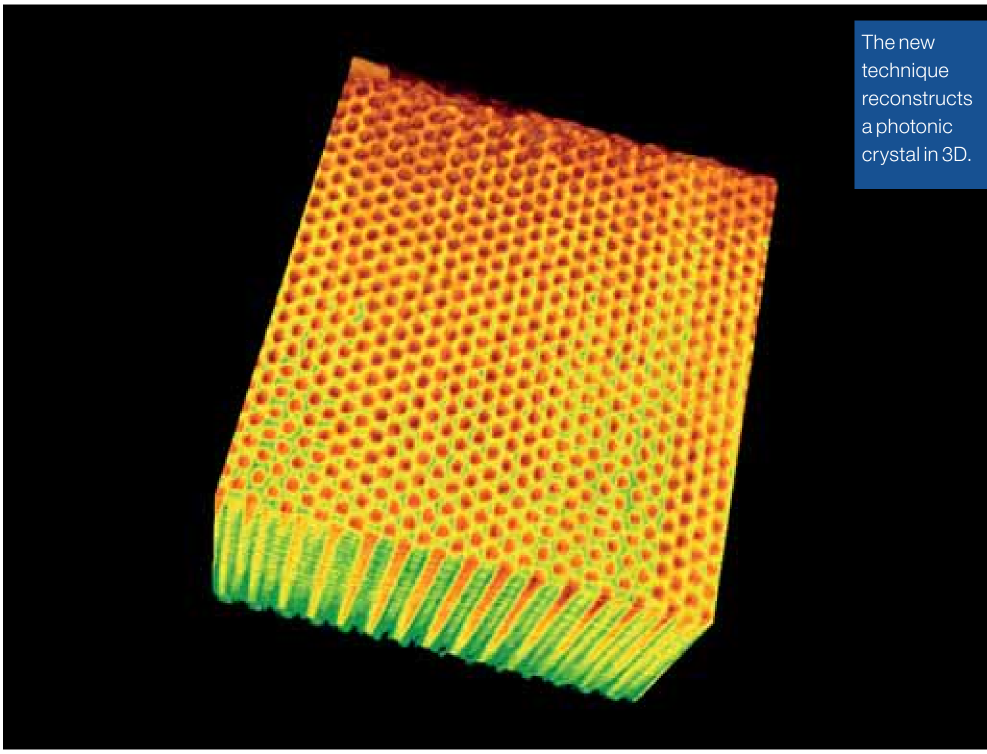
Facilities build pan-EU industry network

Research infrastructures across Europe have launched a new project to build a pan-European network of industrial liaison and contact officers (ILOs and ICOs) to boost partnerships with industry. Backed by the European Commission, the €1.5 m ENRIITC (European Network of Research Infrastructures & IndusTry for Collaboration) project launched in January. It is coordinated by the European Spallation Source in Sweden and includes 10 other participants, including the ESRF.

When it comes to innovation, industry plays an increasingly important role in research infrastructures. Collaborations have been fostered in the past with pan-EU initiatives such as the Innovation Union and the European Strategy Forum on Research Infrastructures, but these have not always fully utilised ILOs and ICOs. ENRIITC aims to plug the gap by inviting all interested parties to create a permanent ILO/ICO network. Over three years, the participants will establish a sustainable European network of ILOs and ICOs to enable mutual learning; map the potential for collaboration between research infrastructures and industry; develop and refine strategies and best practices to foster these collaborations; and raise awareness among industry for collaboration opportunities at research infrastructures. The project partners and their associates aim to balance the need for expertise from diverse scientific areas, combine it with practical insights that have been gained from relationships with industries in different sectors and places, and propagate it among their networks.

"The European research-infrastructure portfolio covers a wide range of 'flavours', from environment, health and food to physical sciences and social and cultural innovation," says Ed Mitchell, the ESRF's head of business development. "For the first time, ENRIITC will bring these together for a unique cocktail as technology buyers, drivers and suppliers, exploring better engagement with industry."



The new technique reconstructs a photonic crystal in 3D.

ACS NANO 2019

Traceless X-ray tomography demonstrated

A new synchrotron technique demonstrated at the ESRF allows scientists to explore the structure deep inside engineered nanomaterials without having to carve them up and destroy their functionality. Known as traceless X-ray tomography (TXT), the technique relies on higher-energy X-rays to penetrate samples with an equivalent silicon thickness of more than 1 mm, and could be used in future studies of photonics and electronic chips.

"In modern nanotechnology this is plenty sufficient to image through wafers," says lead author Diana Grishina of the University of Twente in the Netherlands. "TXT goes up to 20 times deeper than existing technologies. Indeed, all silicon devices during our study remained untouched and 'as is'."

"TXT goes up to 20 times deeper than existing technologies"

Today's engineered nanomaterials are complex three-dimensional structures, be they photonic for manipulating light, for example, or electronic chips consisting of multiple layers of silicon and interconnecting wires. To find out whether the fabricated structures are actually what the designers intended, they often have to be cut into thin slices and observed one at a time. It is a time-consuming process that also destroys the material's functionality.

Demonstrated on the ESRF's ID16A beamline, TXT involves rotating a sample to collect transmission images at different angles, with a beam focused to a spot of 23 by 37 nm. The separate images are then combined into a single 3D image, which can be enlarged if desired. Because of the higher energy of the X-rays, the photon attenuation length in the sample is much greater – 640 μm in silicon, or between 9 and 20 times greater than previously allowed.

Grishina and colleagues tested the technique on three photonic-bandgap crystals, seemingly identical on the surface according to electron microscopy. TXT revealed two to have inner flaws – in one, a large void, while in the other, a lack of 3D structure (*ACS Nano* **13** 13932).

Peter Cloetens, beamline responsible for ID16A, says the new technique will improve with the imminent Extremely Brilliant Source upgrade. "It will obviously increase the throughput for these kind of measurements, but also the resolving power and image quality by using even higher-energy X-ray beams with sufficient coherence."

Queensgate: collaborating to push the boundaries of measurement science

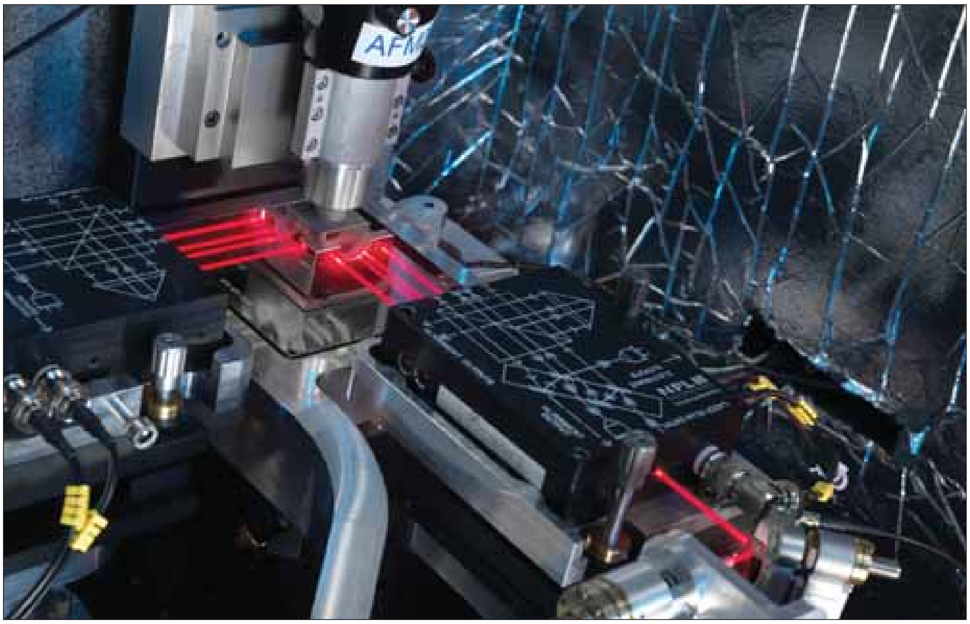
The National Physical Laboratory and Queensgate share an unrelenting passion for better precision at the cutting edge of measurement science.

Queensgate – a brand of Prior Scientific Instruments – offers their customers maximum precision and accuracy along with high-speed solutions for the nanopositioning challenges they face. Customer applications range from hard disk testing for companies such as Seagate (see "Queensgate reaches the pinnacle of nanopositioning performance") to realizing units with national measurement institutes such as the National Physical Laboratory (NPL).

It is this reputation that has seen Queensgate provide high-precision nanopositioning stages for some of NPL's atomic force microscopes (AFMs), including NPL's extremely accurate metrological AFM.

The metrological AFM forms a key part of some of NPL's most important work. As the National Metrology Institute for the UK, it is NPL's responsibility to realize the international standards of measurement – the SI units – in the country. NPL uses this AFM to calibrate transfer standards that are then passed on to other AFM users in order for them to calibrate their AFMs.

Queensgate has been working with NPL in this way for many years. But more recently, NPL came to Queensgate with a more challenging task.



Queensgate XY stage in AFM at National Physical Laboratories.

Realizing the metre

The redefinition of the SI in 2019 now means all SI units are based on precise, unchanging and universal fundamental constants of nature. New definitions of the kelvin, ampere, mole and the headline-grabbing kilogram are now in effect. Perhaps less well known are the changes made to realizing the metre.

The metre was already defined by the speed of light back in 1983. Since then, metrologists have used optical interferometers to realize the metre and make length measurements – and for most measurements this technique is extremely precise. At the nanoscale, however, metrologists must subdivide the wavelength of light, which is several hundred nanometres, making the technique prone to errors.

"We needed a bottom up approach, at an atomic scale," explains Andrew Yacoot, principal research scientist at NPL and chair of the Working Group for Dimensional Nanometrology of the Consultative Committee for Length (one of ten Consultative Committees that oversee the SI units).

To solve this issue, Yacoot and colleagues from other metrology labs used a method directed towards measurement of the Avogadro constant in which the lattice spacing of silicon is measured very accurately using a technique called X-ray interferometry. "We were able to use this technique together with the known value of silicon lattice spacing to characterize and measure errors in optical interferometers or other displacement sensors, as a technique for making traceable length measurements at the nanoscale" he says.

Queensgate was tasked with designing a custom nanopositioning stage and digital controller for Yacoot's X-ray interferometry work. "The specification was challenging, because I wanted a long range of measurement (several hundred micrometres) together with picometre resolution and the stage's payload was almost 1 kg," he explains. "These are competing requirements for stage design."

To reduce the overall system noise, the Queensgate team used a digital interface to apply commands. This also allowed greater timing accuracy, as the field-programmable gate array (FPGA) could conduct all the data processing, including commanding the controller. "We provided an out of the box system that NPL can command and resolve down to 10–20 picometres, ten times smaller than the spacing between the atomic planes in a silicon crystal," says Queensgate's Principal Electronics Engineer. "That, I think, is impressive."

A bi-directional partnership

Dialogue was the key element of the partnership for Yacoot and his team: "We're very keen to have a deep understanding of how every component works and access to all the signals and control of the equipment, rather than having a black box," he says. "We want to [use equipment] produced by a company that's open to a dialogue with us and willing to share information – that's certainly been the case with Queensgate."

In fact, two-way dialogue and collaboration is the bedrock for the relationship between the two organizations. All Queensgate products are tested using interferometers and electronics, most of which are supplied by NPL. NPL acts as a third-party collaborator in verifying measurements on new products, validating their performance, which in turn has helped Queensgate secure substantial new business.

Collaboration, then, benefits both parties. NPL gets to use Queensgate's high-precision piezo systems and gains access to industry-level expertise in designing high-precision, flexure stages for their applications. Meanwhile, Queensgate gains trusted verification of the accuracy of its products. Moreover, by working in partnership with NPL's leading experts on applications that push the limits of performance, Queensgate is able to maintain its position as an expert in producing the highest precision stages. Perhaps best of all, the collaboration benefits us all, as knowledge gained from public–private collaborations like this one leads to more accurate measurements, which in turn leads to more efficient and effective technologies and products.

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Finding precious answers

Synchrotron techniques show long-time ESRF users Chimet SpA how to improve platinum catalysts for pharmaceuticals and other industries.

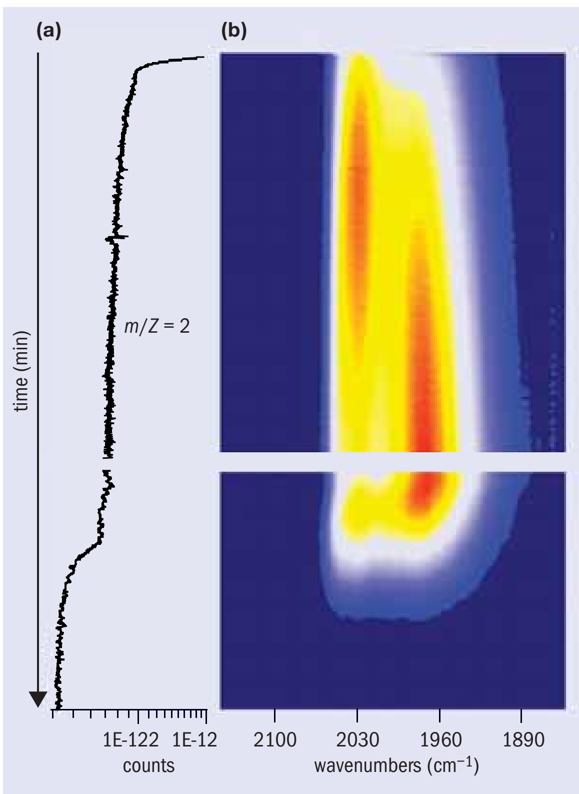
The scientists at Chimet SpA in Italy are experts in the manufacture of catalysts made of precious metals, but there are things that, even for them, are not completely understood. Precisely how hydrogen adsorbs onto nano-sized platinum particles, for instance. How many adsorption sites are there? What platinum-hydride species are produced? And under what operating conditions? All these questions determine the effectiveness of platinum catalysts, but despite intense research spanning decades, there are no definitive answers.

The problem is that, during a reaction, the electronic and atomic structure of platinum nanoparticles change in a highly complex manner. But here, synchrotron techniques can help. For nearly 15 years, Chimet has made use of the ESRF in partnership with a research group currently led by Elena Groppo at the University of Torino in Italy. "The experiences that I've had at the ESRF, either with experiments I've attended directly or through our collaborators, have been always positive and successful," says Riccardo Pellegrini, the manager of research and development in Chimet's catalyst division. "We enjoy not only the instruments that are available, but also the professionalism of the people working there."

SHUTTERSTOCK.COM/ ALEKSANDRA GIGOWSKA

P-aminophenol, an intermediary in the production of paracetamol, is one of several chemicals in the pharmaceutical industry whose production involves the use of platinum catalysts.

Last year, together with co-workers at the University of Torino and the Institut Laue-Langevin on the EPN campus, Pellegrini made use of a combination of diffuse reflectance infrared Fourier transform (DRIFT) spectroscopy, X-ray absorption spectroscopy and mass spectroscopy at the ESRF's BM23 beamline to study a catalyst consisting of platinum nanoparticles supported on a bed of powdered alumina (fig. 1). The catalyst is used in the pharmaceutical and fine-chemical industries in reactions such as the hydrogenation of aromatic rings, ketones and nitro-groups.



M CAROSSO *ET AL ACS CATAL.* **9** 7124

Figure 1 During a dehydrogenation reaction on a platinum catalyst at the BM23 beamline, (a) a mass spectrometer located at the outlet of the cell charts the reduction in hydrogen. Simultaneously, (b) DRIFT spectroscopy records how strongly (with weaker depicted as blue, stronger red) photons of different wavenumbers are absorbed on the surface of the catalyst. The strength of absorption indicates the species of platinum-hydrides present.

The results, which agreed with predictions in the literature, told Pellegrini and his co-workers exactly what platinum-hydride species are involved in the hydrogenation process. They also told the researchers what species are not involved directly, but still help to keep the platinum nanoparticles electronically and morphologically stable during the reaction, and thereby stave off deactivation (*ACS Catal.* **9** 7124). "This will allow us to improve the reactivity and stability of the catalyst in the industrial hydrogenation process where it is involved," explains Pellegrini. ■

Jon Cartwright

D CHENEVIER/ESRF

Moment of joy

At 12.30 p.m. on 6 December 2019, in front of a bank of monitors in the ESRF control room, director-general Francesco Sette (centre) witnesses the first storage of electrons in the ESRF's new Extremely Brilliant Source (EBS). Coming just five days after the first electrons are injected into the new storage ring, the historic milestone sets a fast pace for commissioning, which culminates in the delivery of the EBS beam to the experimental hall one month ahead of schedule (see News, p6).

The EBS teams are used to working quickly: it took just one year for them to dismantle the original storage ring and install the new one within the constraints of the existing infrastructure. As this issue went to press, they were continuing to optimise the beam parameters so that the experimental programme could recommence in March.

The ESRF is set to reopen to scientific users in August. ■

Anya Joly

Tales from the tunnel

A new blog celebrates the staff behind the EBS upgrade.

The Extremely Brilliant Source (EBS) is a product not only of diverse expertise, but of diverse personalities, too. Now, to celebrate those faces that have been hidden behind the upgrade, the ESRF has launched a new series, "Humans of ESRF: In the tunnel".

Following the success of a 2018 blog, "Humans of the ESRF", which paid tribute to ESRF staff from across the facility, the new series of portraits will highlight the vibrant range of experts and fresh recruits who have worked together, sharing knowledge and skills, to make the EBS dream a reality. From assembly and dismantling, to installation and commissioning, ESRF staff from countless different fields have worked towards the same goal: to shine a brighter light on the world we live in. A new portrait will be released each Wednesday, over 10 weeks. Visit humans.esrf.fr, or check out the ESRF's social-media sites with the hashtags #inthetunnel and #humansofESRF. ■

STEF CANDÉ/ESRF

STEF CANDÉ/ESRF

Clockwise from top left: Bernard Ogier, mechnical engineer; Razihan Karakas, control-room operator; Jean-Claude Biasci, EBS assembly and shutdown coordinator; Cristina González-Torres, survey and alignment engineer.

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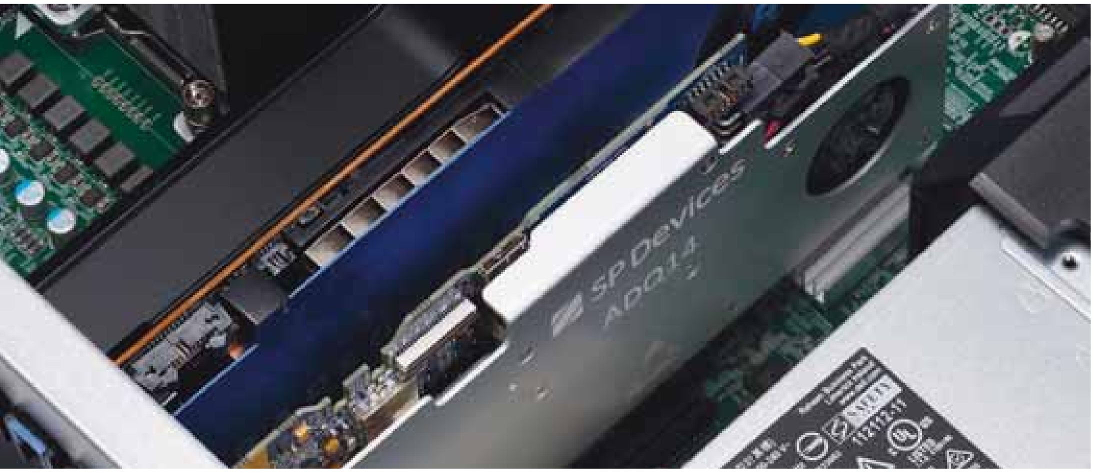

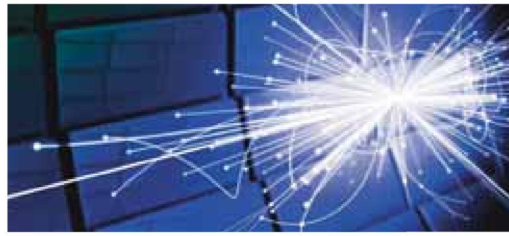

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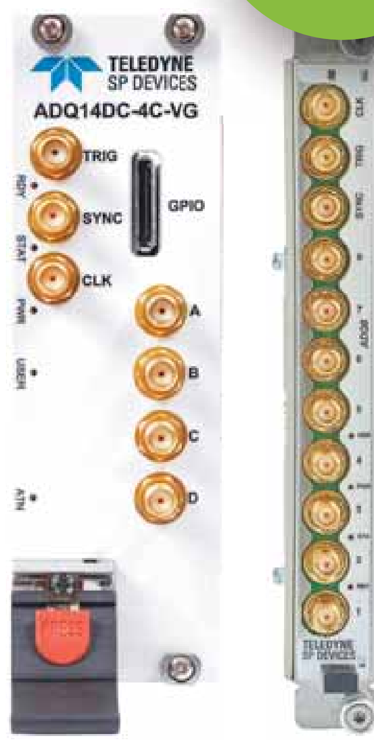

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The unsung heroes

Without the ESRF's survey and alignment group, the EBS would not work at all.

What is alignment, and why is it necessary?
When electrons enter a storage ring, they need to be guided by magnets along a smooth path, without hindrance, and that means aligning the centres of the magnets relative to one another. If any part of the ring is misaligned, the electrons will simply hit the walls of the vacuum tube through which they travel within the magnets, and there will be no more X-rays. Alignment is important to the operation of any storage ring; for the Extremely Brilliant Source (EBS), with its ultra-fine tolerances, it is absolutely crucial. But the process receives little public credit. As Harald Reichert, the ESRF director of research for the physical sciences, has remarked: "The ESRF survey and alignment (SA) group are the unsung heroes of the upgrade project."

Is it just a case of making the storage ring a perfect circle?
No. When the original ESRF was built in 1992, its engineers did their best to create a circular storage ring on the EPN campus with the technology of the time. Sandwiched between the Isére and Drac rivers, however, the campus is in a relatively active geological area – albeit very slowly, it is actually slipping down the valley towards Voreppe. As a result, the ESRF infrastructure today is slightly tilted and slightly elliptical, rather than a perfect circle. That does not in itself matter, as the goal is for the path of the electrons to be smooth, whatever their overall trajectory. Still, the SA group has to monitor the evolution of the 3D shape of the ESRF infrastructure, because it provides the backdrop to which all internal measurements refer.

How to they determine that 3D shape?
There are references at various points surrounding the ESRF – elsewhere on the EPN campus, for example, and especially in the ESRF tunnels – to which the SA group can measure precise distances and angles with laser trackers. These observations can then be entered into a computer algorithm to construct an accurate and continually evolving 3D model of the ESRF infrastructure.

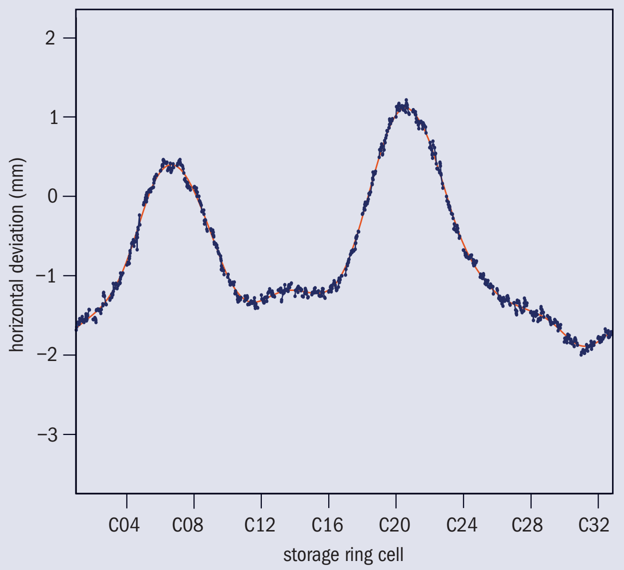


Figure 1 The horizontal deviation of the magnet cells from the position they would have if the EBS were a perfect circle – which would be a horizontal line at zero on the vertical axis – after final alignment. The two main bumps on the wobbly line indicate that the ESRF has, over nearly three decades, become slightly elliptical due to geological shift. Importantly, however, the deviations of individual cells from the wobbly line itself after alignment are almost all within 60 µm, indicating that the overall shape of the EBS is very smooth, and therefore conducive to incredibly bright X-rays.

"The fact that electrons can now continuously circulate the EBS storage ring is proof of the SA group's success"

Is it that model that tells the team where to place each magnet relative to the actual infrastructure, to create a smooth ring?
Again, it is not that simple. Once the magnets are on their girders and assembled into the EBS storage ring, it is no longer possible to see the most crucial points – their centres. For that reason, the first stage of EBS alignment was a process of "fiducialisation", from a Latin word meaning "trust". On the exterior surfaces of the magnets are inverted cones, on which reflectors for measurement could be placed securely. Before the girders were assembled, and while the magnet interiors were still visible, the SA group used a sophisticated laser-tracking system to make sure that these cones were in known positions relative to the magnet centres.

What were the next stages?
In assembling the magnets on the girders, the engineers used the same tracking system to make sure the cone reflectors were precisely aligned with the girders' edges. Then, once the girders were installed to make the EBS storage ring, their edges could be aligned relative to key points inside the ESRF buildings. By the start of commissioning last December, most of the magnets were installed to within 60 µm – about half the width of a human hair – of their target positions, as fig. 1 shows.

EBS commissioning is nearly complete. Does that mean it's a job well done?
The fact that electrons can now continuously circulate the EBS storage ring is proof of the SA group's success. But as they learn more about the machine, the engineers are still making refinements to the alignment to optimise the magnet behaviour, and they will soon be involved with the alignment of the beamlines. Anyway, since the ground beneath the ESRF is shifting, their job is never truly done. "It's like being on the open sea," says David Martin, the head of the SA group. "Everything is moving all the time." ■

Jon Cartwright

THE NANO TRAIL

We're leaving manmade nanoparticles behind in the environment.
What happens to them? ESRF users are exploring the potential impacts.

WASH your veg: that is always the first step in any recipe, to remove any harmful bacteria and traces of toxic pesticides. But consider this result of a 2014 study by Camille Larue at the University of Grenoble and colleagues, performed at the ESRF. When the researchers sprayed lettuce plants with silver nanoparticles – which are being explored in the agricultural industry for use as pesticides – they found that the nanoparticles became trapped inside the leaves (*J. Hazard. Mater.* **264** 98). "They couldn't be washed off," says Hiram Castillo-Michel (pictured right), a scientist at the ESRF's ID21 beamline and one of the study's authors.

More and more scientists are concerned about the fate of synthetic nanoparticles. While nanoparticle toxicity has been studied for decades, only in the past 15 years or so has there been a concerted effort to explore the journey of nanoparticles beyond their original application, into the broader environment and the food chain. There are questions aplenty. What happens to a nanoparticle-based pesticide once it has killed the microbes that it was intended to kill? What happens to the nanoparticles in high-performance sportswear when it is laundered? What happens to the nanoparticles inside consumer technology when it is thrown away?

These are harder to answer than you might think. Obviously, the size of nanomaterials (generally defined to be less than 100 nm) makes them highly inconspicuous. But their exceptionally high surface-to-volume ratio also gives them more unusual properties, and sometimes much greater reactivity, than their bulk counterparts. This is what makes them so desirable in the first place, but it also means that a nanoparticle fabricated in a factory will often have taken on a completely different guise by the time it crops up miles away in, say, an agricultural field.

Synchrotron radiation techniques, such as those provided by the ESRF, are particularly well suited to this new, cat-and-mouse science of "nanoecotoxicology". Spatially resolved X-ray absorption spectroscopy, for instance, can accurately distinguish one chemical species from another, while synchrotron X-ray fluorescence mapping can detect multiple elements down to resolutions of tens of nanometres. Used in conjunction with one another, synchrotron techniques – which are becoming more powerful with the ESRF–EBS (see "The EBS: get real", right) – are enabling scientists to work out what synthetic nanoparticles exist within plants and where they come from.

Through the food chain

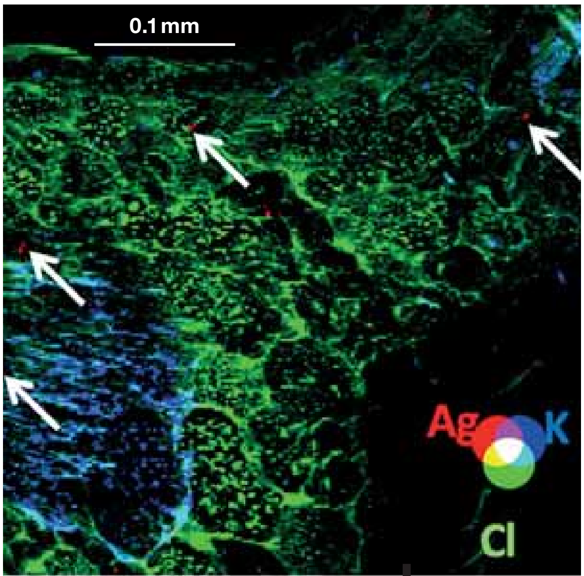
One ESRF study that neatly illustrates the longevity of nanoparticles in the food chain – and the power of the ESRF to help track them – was performed in 2017 by Alia Servin of the Connecticut Agricultural Experiment Station in New Haven, US, and colleagues. The study concerned the presence in agricultural soil of nanoparticles of copper oxide, which is widely used as a wood preservative and which has also been touted as a nanofertiliser and nanopesticide. With use of micro X-ray fluorescence (μ-XRF) and micro X-ray absorption near-edge spectroscopy (μ-XANES) at the ID21 beamline, the researchers demonstrated how copper-oxide nanoparticles ascend the food chain, starting in soil before being taken up by a lettuce plant, being eaten by a cricket, and finally being eaten by a lizard (although the researchers could not ascertain whether the copper oxide was still in nanoparticle form in the last two organisms).

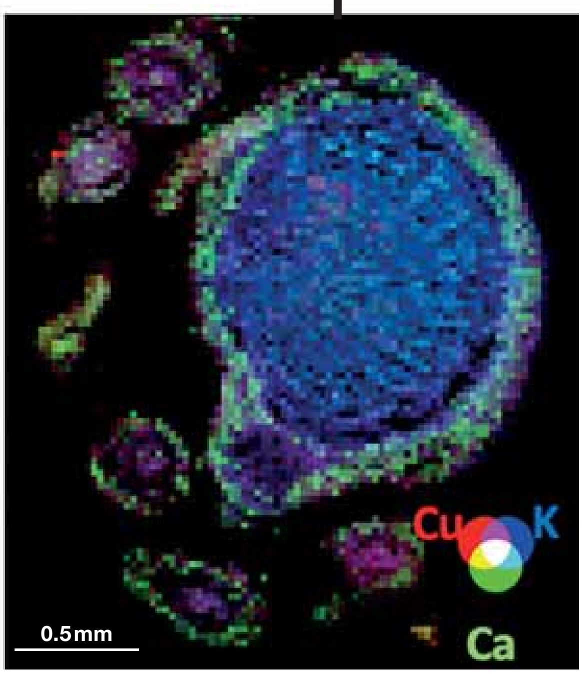
As there is already evidence for the toxicity of copper-oxide nanoparticles, Servin and colleagues' study could be taken as a straightforward example of the potential danger of nanoparticles introduced into the food chain. In fact, it is not so simple. When the researchers left the nanoparticles to age or "weather" in the soil for one month before growing the lettuce, they found that more than double were taken up by the plant roots; that they became more homogeneously distributed through the plant; and that they reacted with sulphur in the soil to form copper sulphide (*Nanotoxicology* **11** 98). Unfortunately, they could not determine the effect of these changes on nutrition.

"The chemistry of synthetic nanoparticles is very complex"

THE EBS: GET REAL

One hundred times more flux and coherence: that is the overall improvement that the ESRF's upgrade, the Extremely Brilliant Source (EBS), is set to offer when it opens to general users in August. For nanoecotoxicology and beamlines such as ID21 (above), however, the specific benefit of the EBS will be the step-change in detection limits and spatial resolution. As a result, scientists will be able to study the journey of nanoparticles at lower – and hitherto undetectable – doses that more accurately portray the real environment. Moreover, they will be able to detect important intermediary species that potentially affect the final toxicity, and increase the robustness of conclusions by taking more data in one go.





A combination of micro X-ray fluorescence, for localisation, and X-ray absorption spectroscopy, for speciation, exposes the uptake by lettuce plants of copper-oxide and silver nanoparticles – both of which are touted as potential nanopesticides due to their antimicrobial and antifungal properties. A cross-section of the lettuce roots (bottom) shows copper oxide dissolving at the root surface, increased copper uptake and, inside the roots, copper sulphidation. Meanwhile, the foliage (top) exhibits an internalisation of silver nanoparticles and ions, as well as sulphidation.

The following year, Servin and colleagues came upon another example of how nanoparticles in soil enter the food chain, this time those made of cerium oxide, which is also a potential nanopesticide. Again using μ-XRF and μ-XANES at ID21, the researchers evaluated the location, speciation and persistence of the nanoparticles in earthworms living in the contaminated soil. The results were in part good news, revealing little evidence for toxicity, and moreover that a popular charcoal-based soil improver, biochar, can help to reduce nanoparticle accumulation. On the other hand, the results also showed that the nanoparticles were still present in the earthworms at the end of the study, three days later, raising a question mark over the organisms' long-term health (*J. Agric. Food Chem.* **66** 6609).

Indeed, the story gets even more complicated for cerium-oxide nanoparticles once the type of soil is placed under consideration. A few months before the previous study, Clément Layet of Aix-Marseille University in France and colleagues studied the uptake of the nanoparticles in tomato plants and fescue grass, growing in either sandy soil poor in organic matter or clay soil rich in organic matter. The results – some of which came from the ESRF's BM16 collaborating research group beamline – painted a mixed picture: the clay withheld the nanoparticles, whereas the organic matter released them to the roots. A further test in a hydroponic growth bed, designed to normalise the tomato and fescue root systems, showed that both species of the plants responded the same, and moreover that nanoparticle transfers took place at lower concentrations than normally cited in the research literature (*Environ. Sci. Technol.* **51** 9756).

All these studies concern nanoparticles introduced more or less directly into the soil, which closely matches reality for nanopesticides, for example. But nanoparticles have many other intended uses beyond agriculture, and they can originate far and wide. In 2018, for instance, using XANES at the ESRF's ID26 beamline, Alain Manceau of Grenoble Alpes University and colleagues showed that mercury in the atmosphere – a by-product of centuries of mining and other human activities – can be absorbed by foliage, where come leaf-fall, it is transformed into mercury-sulphur nanoparticles (*Environ. Sci. Technol.* **52** 3935).

The journey for silver nanoparticles is much faster. Thanks to their unique optical, electrical and antimicrobial properties, silver nanoparticles are now used in hundreds of applications, from consumer technology to cosmetics, and from fabrics to food containers. When items like these last ones are washed, inevitably some of their nanoparticles go down the drain.

In 2016, Ana Pradas del Real at Grenoble Alpes University and colleagues revealed the fate of silver nanoparticles once they had been injected into a waste-water treatment plant, to simulate silver nanoparticles released from consumer products. Via a range of techniques at the ESRF beamlines ID21, ID16B and BM30, the researchers showed that the nanoparticles that emerged in the resulting sludge had reacted to form silver sulphide, and other potentially less stable silver-sulphur species that may dissolve into silver and sulphide ions (*Environ. Sci. Technol.* **50** 1759).

Such sludge is commonly applied to agricultural fields as an inexpensive fertiliser, opening a door to crop transference. Although the researchers found little evidence for silver uptake in plants grown on sludge-amended soil, they did find that the plants' growth was significantly less vigorous.

However, another study by Basilius Thalmann and others at the Swiss Federal Institute of Aquatic Science and Technology (EAWAG) in Dübendorf at the ESRF's Dutch–Belgian CRG beamline BM26, exploring the effect of the concentration of soil humus on silver-nanoparticle sulphidation, exposed an intriguing fact: that not all silver-sulphide nanoparticles are the same. Compared with those formed in a lab, those formed in real soil, in the presence of humic acid, had a hollow and potentially more reactive structure (*Environ. Sci. Nano* **3** 203).

When questioning the toxicity of synthetic nanoparticles in the environment, then, it is hard to get a straight answer. Indeed, the same is true of potential solutions to nanoparticle ecotoxicity. Last year, research undertaken at the ESRF, involving scientists at EAWAG and elsewhere, lent support for one potential solution: the treatment of sludge by incineration, which is a common way of generating energy at waste-treatment plants. Working at BM31, Alexander Gogos and colleagues found that incineration totally destroys nanoparticles made of cerium oxide (*Environ. Sci. Nano* **6** 1765).

"The engineered nanoparticles were still in the earthworms at the end of the study, three days later"

Such a result might suggest incineration is a safe bet. But other studies have come to the opposite conclusion. Working at BM26, Jonas Wielinski at EAWAG and colleagues have reported that incineration converts sulphidised copper and zinc nanoparticles back to their original species (*Environ. Sci. Technol.* **53** 11704). Meanwhile, use of the same beamline – as well as the Swiss Light Source in Villigen – by Christoph Meier at the Zurich University of Applied Sciences in Switzerland and colleagues, has shown that incineration can convert nanoparticles of silver sulphide back into those of pure silver, which is more reactive and potentially more toxic (*Environ. Sci. Technol.* **50** 3503). Results like these have led Wielinski and colleagues to recommend that the transient nature of synthetic nanoparticles should be taken into account in waste-processing risk assessments.

Perhaps, as some research indicates, we should be looking for biological solutions to nanoparticle contamination (see "Natural defenders", below). But if one thing is clear from nanoecotoxicology so far, it is that there will always be more to the story. Castillo-Michel, who has co-authored much of the work at the ESRF, knows the importance of finding out what the ending is. "The full periodic table seems to be available to produce nanomaterials, and their chemistry is very complex," he says. "We need to investigate their risks to the environment before we use them in commercial products if we want to avoid possible consequences to human health such as those seen with asbestos." ■

NATURAL DEFENDERS

The natural world might have its own defences against synthetic nanoparticles, according to ESRF research. In 2017, for instance, Eva Kovačec of the University of Ljubljana in Slovenia and colleagues started off trying to find out whether synthetic copper-oxide nanoparticles could be used to kill the fungus *Botrytis cinerea*, a scourge of farmers worldwide (as the diseased grapes above show). In the end, with help from extended X-ray absorption fine structure spectroscopy at the BM23 beamline, they found that the fungus actually degrades the nanoparticles to copper oxalate – suggesting it could instead be used to clean an area suffering from copper-oxide nanoparticle contamination (*Chemosphere* **180** 178e185).

The soil can create its own nanoparticles in response to non-nano contaminants, too. In 2018, Manja Vogel and colleagues at the Helmholtz-Zentrum Dresden-Rossendorf (HZDR) in Germany investigated the fate of selenium – an essential trace element that becomes toxic at higher concentrations – in the presence of the soil bacterium *Azospirillum brasilense*. Taking XAS measurements at the HZDR's ESRF ROBL beamline, the researchers found that the bacterium reduces the common oxide of selenium, selenite, to much safer selenium-sulphide nanoparticles (*J. Hazard. Mater.* **344** 749). "Azospirillum … may help to prevent accumulation of selenium in crops cultivated on selenium-contaminated soils," they conclude.

Centre of attraction

Sergio Bertolucci, independent chair of the EU project ATTRACT, explains why scientific instrumentation has so much to offer.

BORN: 1950, La Spezia, Italy.
EDUCATION: PhD in particle physics, (1976).
CAREER: Researcher, DESY (1974–1978) and Fermi National Accelerator Laboratory (1979–1996); director, INFN National Laboratory of Frascati (2002); vice-president, INFN (2005); director of research, CERN (2009–); professor, University of Bologna (2016–).

Sergio Bertolucci knows a thing or two about adapting instrumentation for new purposes. In the late 1990s, the experimental particle physicist designed a huge calorimeter to accurately measure the energy, and especially the timing, of kaons generated by meson-decay at the DAFNE particle accelerator at the INFN Frascati National Laboratory in Italy. Two decades later, he is having the same 100 tonnes of equipment shifted to the DUNE experiment at Fermilab near Chicago, US, to help measure neutrino oscillation. "I don't design detectors for the sake of better detectors; I adapt them for the physics I want," he says. "After all, if you're always making a better detector, you're never doing an experiment. We have a proverb in Italian: the better is the enemy of the good."

Bertolucci is no longer limiting his technological nous to academia. Since 2017, he has chaired the independent research, development and innovation committee for ATTRACT, an EU project designed to foster co-development of breakthrough sensor and imaging technologies to the needs of industry, science and society at large. "Why is there no European Google or Amazon?" asks the ATTRACT website. "It is not for lack of great technology or breakthrough science. It is because the mechanism for scaling up promising ventures to global markets is simply not working."

Why is this? Bertolucci explains: "Our national scale in Europe is too small, and our commercial appetite for risk is not very high." Science can live with the high risk on return for inventing new instrumentation, he continues – it is the only chance of gaining insight into the unknown. But industry needs more confidence, and even once novel scientific instrumentation exists, it is usually too clumsy for the purposes of non-scientists, and there is no guarantee that its adaptation will not be an expensive failure.

The idea of ATTRACT is for the EU to shoulder this initial risk of innovation, not industry. To do this, it has invited users of Europe's research infrastructures, in collaboration with entrepreneurs, to come up with bold ideas for how imaging and detector technology can have a decisive impact on society. Last year, it allocated €100,000 each to 170 projects – including four ESRF projects – out of more than 1200 proposals. In June this year, Bertolucci and his committee partners will have to start a thorough review process to whittle down those to just 20 or so projects for further funding. "It will be tough, because the number of projects we can support will not be the number that we want to support," he says.

The ESRF – one of nine ATTRACT partners – has played a "fundamental role" in getting ATTRACT off the ground, he adds. "Your director-general, Francesco Sette, has promoted the idea in Europe. And Michael Krisch, who now chairs our consortium board, really understands the potential of this bottom-up approach, which exploits the creativity of our researchers and innovators."

Bertolucci's greatest claim to fame is that he designed the central calorimeter for Fermilab's CDF experiment, which in 1995 discovered the top quark, the most massive of all elementary particles. Now he hopes that scientific instrumentation adapted under ATTRACT can have just as strong an impact in the marketplace. If it works, he believes the tech-transfer model can be extended beyond detectors and imaging to all sorts of other areas of scientific innovation – artificial intelligence, for example, or infrastructure. "For any of these things, there could be a zillion industrial applications," he says. ■

"For any of these things, there could be a zillion industrial applications"

Jon Cartwright

EVENTS

Dark-field X-ray microscopy for EBSL2 workshop

15–17 April 2020

Dark-field X-ray Microscopy is a newly developed technique to measure orientation and strain in crystalline materials with a spatial resolution down to 100 nm. When the ESRF–EBS opens to scientific users in August, the hard X-ray microscope at the ID06 beamline will be the first instrument worldwide to offer the technique. Later, the flagship beamline EBSL2, constructed on the current ID03 port, will improve spatial resolution 10-fold and make experiments hundreds of times faster. This workshop will present the new instrument and help form a new user community. Via discussions with potential users, ESRF scientists aim to determine their needs for instrumentation such as sample environments, auxiliary measurements and sample preparation.

Cryo-electron microscopy workshop

9–11 June 2020

Lasting two and a half days, this practical workshop on sample preparation for single-particle cryo-electron microscopy (cryo-EM) is aimed at doctoral students, postdocs and scientists who are new to the technique. Jointly organised by the ESRF, the EMBL Grenoble outstation, the ILL and the IBS, it is the fourth in a series of hands-on workshops, and will teach potential users the theoretical and practical aspects of sample preparation for single-particle cryo-EM, including prior quality control by negative staining. The grids prepared in the workshop will be screened at the ESRF's Krios cryo-EM facility as well as at the Glacios facilities at the IBS and EMBL to provide immediate feedback on their quality. Cryo-EM is the perfect technique for biological complexes that cannot be crystallised.

Third workshop on studies of dynamically compressed matter with X-rays

7–9 July 2020

Aimed at scientists from both the static and dynamic extreme conditions communities, this workshop (DyCoMaX) provides a forum to discuss new scientific frontiers in the new combination of shock-inducing lasers with brilliant X-ray probes. It hopes to inspire new projects and to prompt new collaborations among workshop participants, particularly in view of the opening of the ESRF's high-power laser facility (HPLF-I), which will couple a nanosecond, 100-joule laser with time-resolved X-ray absorption at the ID24 beamline. The coupling of dynamic compression with time-resolved X-ray diffraction and X-ray imaging will also be covered, as well as plans for a future extension of HPLF (HPLF-II) to other X-ray techniques.

MOVERS & SHAKERS

Caterina Biscari, director of the ALBA synchrotron in Spain, became the new chair of the League of European Accelerator-based Photon Sources (LEAPS) in January. Launched in 2017, LEAPS is a partnership of 16 light sources across Europe that aims to offer a "common vision" of using scientific excellence to solve global challenges, while boosting European competitiveness. One of its main aspects is an agreed roadmap for the development of next-generation light sources and instrumentation, and tackling big data. Serving a combined 24,000-strong user community, the initiative also aims to maximise the strengths of individual facilities through coordinated specialisation, and to expand industry services. Meanwhile, it plays an integrating role for countries with less-developed communities and infrastructure.

Previously the vice-chair of LEAPS, Biscari studied physics at the Complutense University of Madrid in Spain before working in a number of different laboratories, such as the particle-physics lab CERN and the Frascati National Laboratory – where she served as director of technology and deputy director of science – at the Italian National Institute of Nuclear Physics. She has also presided over the accelerator group of the European Physical Society executive committee, and participated in advisory boards for projects in several countries, including CERN accelerators and the National Centre for Oncological Hadron Therapy in Pavia, Italy.

She takes over the LEAPS chair from Helmut Dosch, chair of the board of directors of the particle-accelerator lab DESY in Germany.

Harry Westfahl Junior became director of the Brazilian Synchrotron Light Laboratory (LNLS) in January, taking over from the former ESRF director-general Yves Petroff. Having defended a PhD in physics at the State University of Campinas in Brazil in 1998, Westfahl became a postdoc at the University of Illinois Urbana-Champaign under the supervision of Anthony James Leggett, a superfluids expert who would go on to win the 2003 Nobel Prize in Physics. In 2001, Westfahl became a postdoctoral fellow at the US Department of Energy's Ames Laboratory in Iowa, before joining the LNLS in the same year. He was manager of the LNLS programme for beamline research and development between 2004 and 2009, and later coordinator of its X-ray scattering beamlines. In 2013, he became the facility's scientific director.

Westfahl is an expert in the use of synchrotron radiation for the study of materials and condensed matter physics, with an emphasis on polymers and magnetic materials, and the development of instrumentation for synchrotron light. He has also coordinated the design and construction of the first beamlines at the new Brazilian synchrotron light source, Sirius.

At an inauguration ceremony, the director-general of the Brazilian Centre for Research in Energy and Materials, Antonio José Roque da Silva, sang the new director's praises. "At this key moment for the institution, in which we will have to structure the start of operations at Sirius, Harry's experience, competence, and multidisciplinary background will be a key factor."

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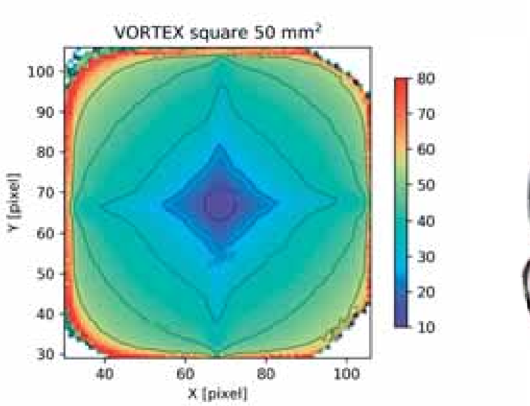

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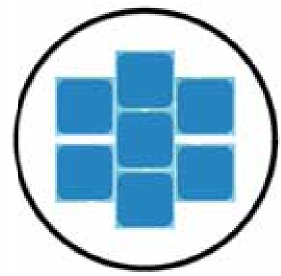

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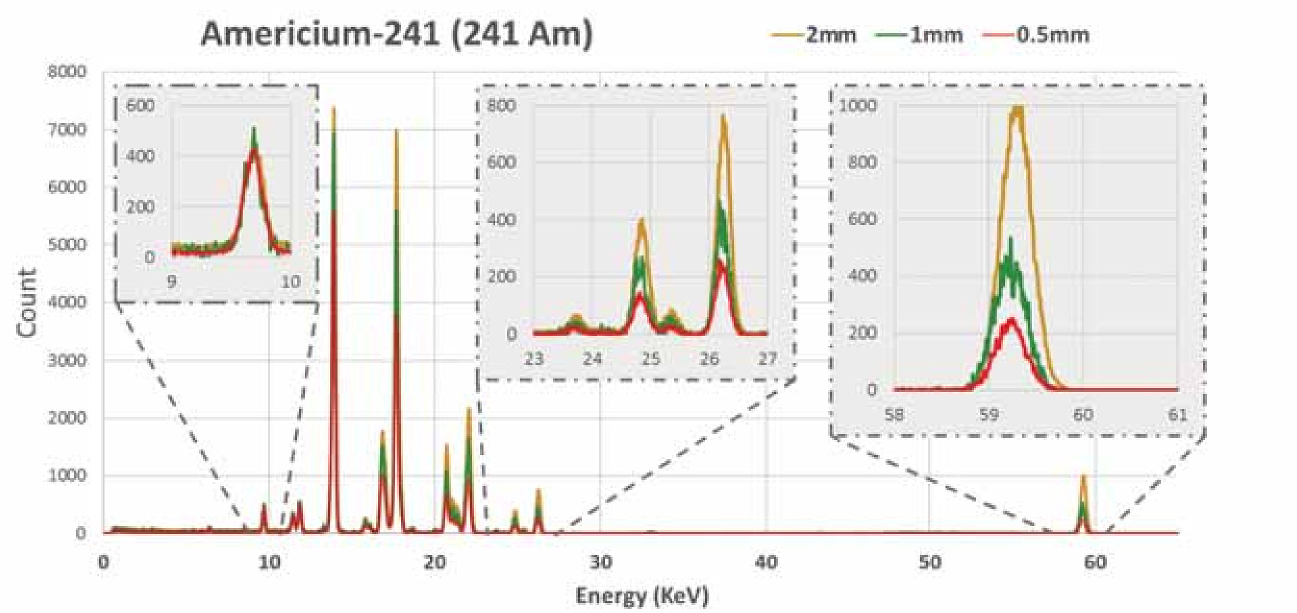

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Hitachi High-Technologies Science America, Inc.
21770 Nordhoff St., Chatsworth, CA 91311

www.hitachi-hightech.com/hhs-us/
Email: sales@hitachi-hitec-science.us
Tel: +1-818-280-0745