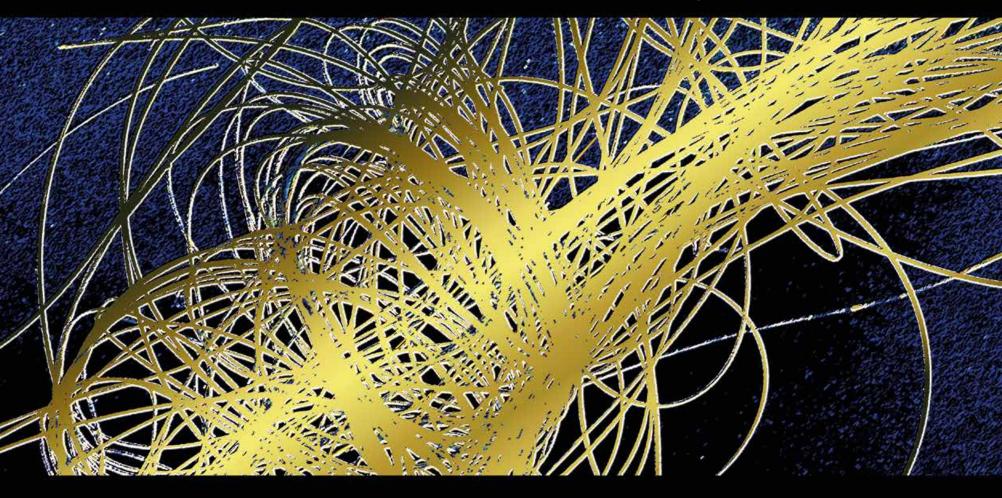
Advanced Light Sources and Crystallography

Tools of Discovery and Innovation



Published by LAAMP, Lightsources for Africa, the Americas and Middle East Project, an International Union of Pure and Applied Physics (IUPAP) and International Union of Crystallography (IUCr) project funded by the International Council for Science (ICSU)

Advanced Light Sources and Crystallography

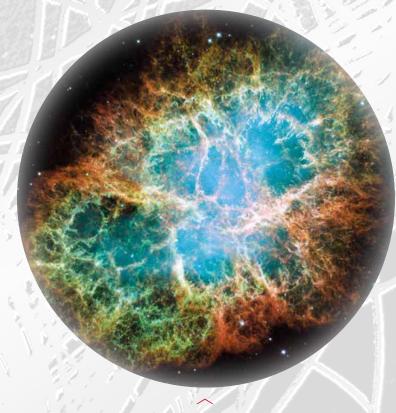
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The Crab Nebula is the remnant of a supernova explosion that occurred in AD 1054. It is about 6,500 light years from the Earth. Energetic electrons from the explosion follow curved paths caused by the strong magnetic field in the nebula and emit synchrotron light, just as do advanced light sources here on Earth.

The cover of this LAAMP Brochure was designed by Italian artist, Luciano Martinis (Aquielia, Italy).

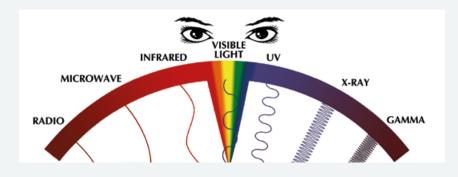
Martinis writes: "The image comes from my personal interpretation of the light emanating from matter. In fact, having no real pictures of what occurs inside the synchrotron, I have worked as an artist inspired by my personal relationship with matter, time and light."

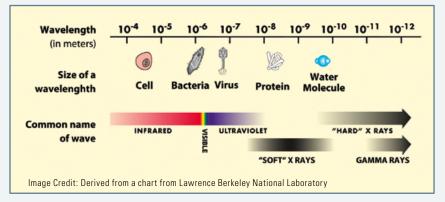
What are light sources? What is crystallography?

Not all light is visible. In science and technology the word **light** applies generally to electromagnetic **radiation**. Most wavelengths of light are not visible.

Light sources generate radio, microwave, infrared, visible, ultraviolet, X-ray and gamma-ray light.

Advanced light sources are much more intense than conventional sources, such as light bulbs and traditional lasers.





Different wavelengths of light compared to sizes of various objects. To see an object we need light with a wavelength that is equal to or less than the size of the object.

The smallest object we can see with visible light is a cell. To see the details of the constituents of a cell, *e.g.* proteins, we need a shorter wavelength such as X-rays.

Crystallography is the science that examines the arrangement of atoms in solids and there is a close connection between the science of Cyrstallography and much of the work done at Advanced Light Source Facilities.

Well-focused X-ray beams, generated by advanced synchrotron radiation facilities, yielded high-resolution diffraction data from crystals of ribosomes, the cellular nano-machines that translate the genetic code into proteins.

Ada Yonath, 2009 Nobel Prize in Chemistry



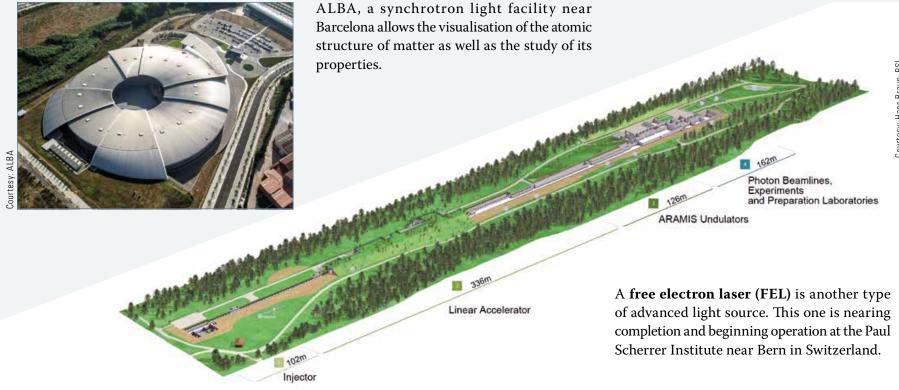
Why care about light sources?

BSERVING MATTER and decoding its secrets are at the heart of humanity's quest to understand the world around us. Advanced light sources offer unique tools to expand the boundaries of scientific investigations into new materials and living matter. As centres for fundamental and applied research, light sources play a key role in stimulating innovation and enhancing industrial competitiveness.

Advanced light sources are revolutionising a myriad of fundamental, applied and industrial sciences, including agriculture, archaeology, biology, biomedicine, chemistry, cultural heritage studies, engineering, energy, environmental science, forensic science, geology, materials science, nanotechnology, new drugs, palaeontology and physics.

Advanced light sources are the ultimate means of characterising materials in our age. They open insights to micro- and nano-structures of manufactured materials that are not possible to obtain in any other way. Industry recognises this, and is an increasing user of advanced light sources to support research and innovation in product development.

These facilities have major impacts on the education and training of graduate students, our future scientists.







More than 50 advanced light source facilities are in operation, construction or planning.

Advanced light sources are key to research frontiers in many disciplines and industries - sometimes addressing current scientific, commercial and educational needs of a particular country or region. Thousands of graduate students in biology, chemistry, environmental,

material and medical sciences, and other disciplines have done world-class research for their Ph.D. theses at light sources around the world. Providing such a resource was a major motivation for many countries to start their light sources in the mid-1980s.







The National Synchrotron Light Source II (NSLS-II) at Brookhaven National Laboratory in the United States is one of the world's newest and brightest light sources, serving up to 6,000 users per year.



How does a light source work?

A CCELERATION is a change in speed or direction of motion. When they are accelerated all electrically charged particles emit light (produce radiation.)

Both protons and electrons when they are accelerated can emit light but electrons emit vastly more than protons. All advanced light sources use electrons.

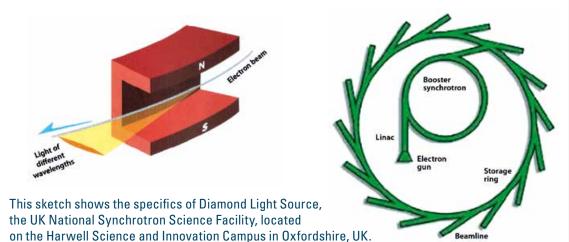
There are two major types of advanced light sources: **circular**, doughnut-shaped electron storage rings called synchrotrons or synchrotron light sources, and **linear**, free-electron lasers (FELs).

Each synchrotron radiation facility is different and has somewhat different capabilities. However, in general, the complex is like a chain composed of links.

69995

Each link in the chain accelerates the electrons further and injects them into the next link in the chain. Details below are for the medium-energy Diamond ring.

CIRCULAR. When an electron beam passes between the north and south poles of a magnet its path is curved. It changes direction and emits light. The electrons can be stored and circulate in a ring for periods of up to a day. As the electrons turn they emit electromagnetic radiation, both visible and invisible, from very long wavelengths (infra-red) to very short wavelengths (X-rays). Since the radiation is emitted tangentially to the ring, many beam lines can be constructed, and scientists can take data simultaneously.

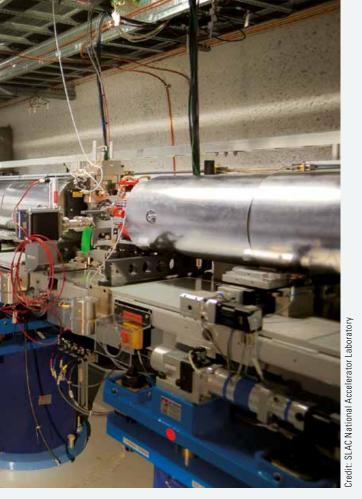




LCLS, The Linac Coherent Light Source at Stanford University's SLAC National Accelerator Laboratory in California, one of the world's first linear freeelectron lasers.

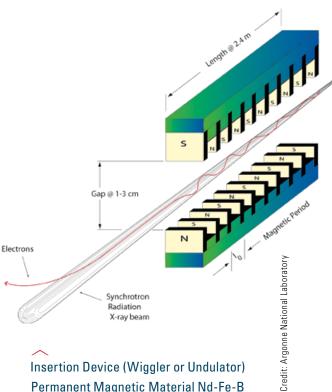
Electron gun. Electrons "evaporate" from a hot surface and are accelerated to 90,000 electron volts (90 keV) of energy. **Linac** (linear accelerator) is the first of 3 particle accelerators in this chain; electrons are accelerated from 90 keV to 100 million electron volts (100 MeV). **Booster**. Bending magnets curve the electrons around a circle, and a radio-frequency source accelerates them to 3 GeV





LINEAR. Making light from electron beams.

Conventional lasers make their extraordinarily useful light by jiggling electrons bound in atoms. Accelerator-driven free-electron lasers (FELs) make light by using magnets called undulators to jiggle electrons that are freed from atoms. The pulses from a FEL are a thousand times shorter than those from a storage ring and many times more intense. This has opened up exciting new possibilities for research in many diverse fields. A disadvantage of FELs is the comparatively small number of beamlines that can be operated simultaneously. An **undulator** is an "insertion device" because it is "inserted" into the accelerator track. It is used in both circular and linear advanced light sources and is a periodic magnetic structure with the north and south poles of the magnets alternating in orientation. The undulator stimulates highly brilliant, forward-directed synchrotron radiation emission by forcing a charged particle beam to perform wiggles (accelerations), or undulations, as it passes through the device.



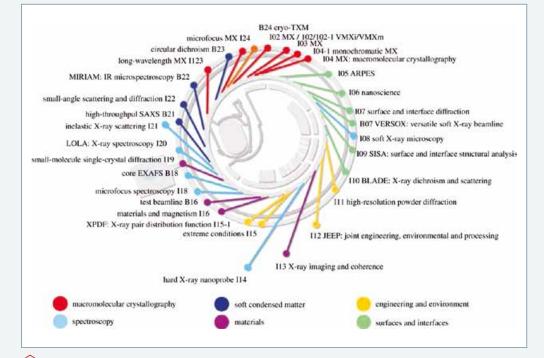
(3 giga or billion electron volts) before being transferred into the storage ring. **The storage ring** consists of straight sections angled together to form a closed loop approximately 600 m in circumference. It is filled every 10 minutes with a new batch of electrons. At some facilities it is filled every few hours. **Beamlines** operating simultaneously carry different wavelengths of light to the user groups.

Brilliance

When comparing light sources, an important measure of quality called brilliance, takes into account the number of photons produced per second, how fast the beam spreads out, the beam size and the spread in frequencies or wavelengths in the beam. X-ray beams from advanced light sources are many orders of magnitude more brilliant than from conventional X-ray tubes.

> 5 INTRODUCTION

Research at an advanced light source facility



The ambiance of working at science and technology frontiers

A group of scientists, usually from a university, but sometimes an industry or government laboratory, and often in a collaboration among several institutions, will write a proposal. They will describe the study, specify which beamline they want to use and how many hours of beam time they require. A committee appointed by the facility Director chooses the best science, usually without regard to institution or country.

Often the group is led by a Professor with postdoctoral scientists and doctoral students. Most facilities have on-site housing, a cafeteria, a library, and a computing centre. They are unique hubs for training and education and the interactions between groups is part of the experience.

Diagram of Diamond Light Source, UK, specifying the typical area of research of each beamline.

Beamlines in the Experimental Hall at the Photon Factory, KEK, Japan.





Education and outreach

ONE of the most successful outreach programmes is the International Union of Crystallography (IUCr)-UNESCO OpenLab programme. OpenLabs is a network of operational crystallography laboratories based in different countries worldwide, mainly in Africa, South and Central America and South Asia and is aimed at allowing access to knowledge gained from crystallography in all parts of the world. Funds for additional OpenLabs are part of LAAMP.





IUCr-UNESCO OpenLab, Rabat. Morocco, May 2014.

This OpenLab was run with the formula of the travelling lab: a portable diffractometer was moved through different locations in the country (Rabat, El Jadida and Agadir) and at each stop it served as the basis for a crystallographic school, including tutorials about the use of the instrument and related software.

TO LEARN MORE

http://iucr.org



Students at the microscope are preparing crystals for an X-ray diffraction measurement while other students in the room analyse data from a previous experiment.

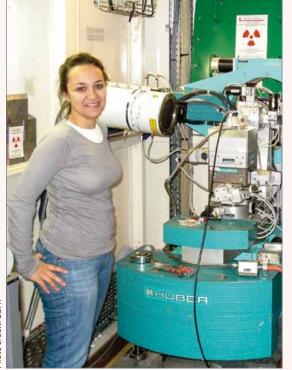


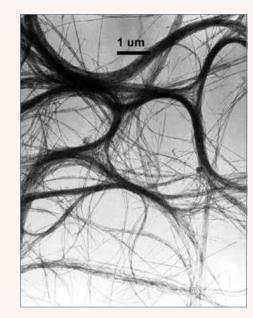
Applications to nanomaterials

NANO is the prefix for one-billionth. A nanometre (**nm**) is one-billionth of a metre. A nanosecond (**nsec**) is one-billionth of a second. A convenient way to remember how long is a nsec, is that a signal travelling in a wire or piece of fibre optic 30 cm long takes about one nsec to go from one end to the other.

Protein molecules are a few nanometres in size (page 1) and the soft X-ray beams from advanced light sources have wavelengths of a few nm's so this is an ideal match for basic studies of protein molecules.

Nanotechnology and nanomaterials are general terms for the design and creation of materials whose use depends on structure at the nanoscale, generally 100 nanometres or less. They include devices or systems made by manipulating individual atoms or molecules, as well as materials which contain very small structures. Nanomaterials may have different physical and chemical properties than the same materials in bulk form. The number of products produced by nanotechnology or containing nanomaterials is increasing. Current applications include healthcare (in targeted drug delivery, regenerative medicine, and diagnostics), electronics, cosmetics, textiles, information technology and environmental protection. Nanomaterials appear in a range of products, including food packaging, wound dressings and food supplements.







Nanowire images from Dr. Öztürk

Özgül Öztürk doing powder X-ray diffraction measurements to study the effect of doping on semiconductor nanowires. The work was done at the European Synchrotron Radiation Facility in Grenoble. Dr. Öztürk chairs the LAAMP Middle East Regional Committee as well as the SESAME Users' Committee.



APPLIC



Elettra Sincrotrone in Trieste, Italy, is an international research centre serving science and industry. The centre hosts two synchrotron light sources: FERMI, a free electron laser, and Elettra, a storage ring. The light produced is transferred to over 30 experimental stations specialising in chemistry, microscopy, materials science, electronics and information technology.

hata: Elattra Cinaratrana

Sunlight-driven photosynthesis is the energy source of all green plants. The invention and development of FELs has opened a fantastic new window into fundamental research. The extremely short duration pulses from a FEL, only 30 femtoseconds in length (one femtosecond is one millionth of a nanosec!) allow a movie to made of fundamental molecular processes as they occur.

The Linac Coherent Light Source (LCLS) at Stanford University produced a "molecular movie" of a bacterial molecular complex that catalyses photosynthesis as it splits water into hydrogen and oxygen. A deeper understanding of photosynthesis could aid the development of better solar cells and might advance the quest for artificial photosynthesis.



Structural biology

Powerful tools to study the biology of diseases

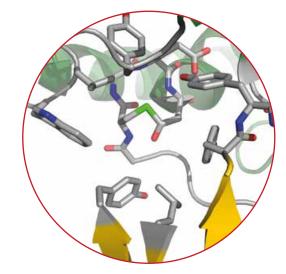
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and Human Services.

MALARIA

Using the Advanced Photon Source at Argonne National Laboratory, researchers from the University of Texas Southwestern Medical Center contribute to the fight against malaria, a disease that kills millions. They study how a protein in the mosquito immune system operates against the parasite that causes malaria.



Thanks to Richard Baxter and Johann Deisenhofer for providing the image.

APPL

ZIKA

ZIKA virus is spread by daytime-active aedes mosquitos and has become a world-wide scourge. Below is a digitally-colourised image of Zika virus particles (coloured blue) about 40 nm in diameter.

Image: Centers for Disease Control and Prevention, U.S. Department of Health

Adobe Stock



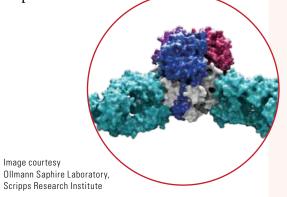
Image: National Synchrotron Light Source, Brookhaven National Laboratory

HIV/AIDS

Globally, about 35 million people are living with HIV, which constantly adapts and mutates creating challenges for researchers. Scientists armed with a clearer image of the HIV virus and its constituent proteins are able to learn how the body can combat the virus with the ultimate aim of producing more effective antiviral drugs.

LASSA

Perseverance pays off in the fight against the deadly Lassa virus. Investigation revealed the first-ever image of its elusive viral protein. An antibody from a human survivor (light blue or turquoise) is shown inactivating the Lassa virus surface protein. The work shows how to engineer vaccine strategies to elicit protective immune responses.

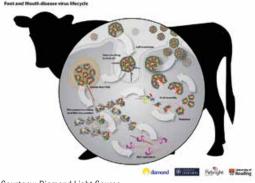




Ebola, a viral hemorrhagic fever of humans and other primates, caused by Ebola virus, spreads by direct contact with body fluids of an infected human or other animal. The largest outbreak to date was the epidemic in West Africa from December 2013 to January 2016. It was declared no longer an emergency in March 2016. Another outbreak in Africa began in May 2017 in the Democratic Republic of the Congo.

Source: PureLife Pulse. PureLife is a manufacturer of health and safety products.

Foot-and-mouth disease



Courtesy: Diamond Light Source

SCIENTISTS at Diamond Light Source and Oxford University have produced a vaccine for foot-and-mouth disease which is caused by a virus. The 2001 outbreak in the UK caused the deaths of over 7 million livestock. Globally it remains one of the most economically important diseases in livestock. It is endemic to central Africa and some parts of the Middle East and Asia. The image shows the the virus lifecycle.

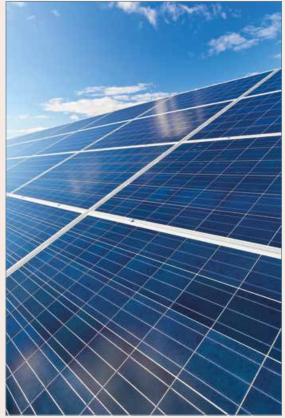


Applications to energy

Тне modern world consumes ever ⊥ increasing amounts of energy. Current reserves of fossil fuels are limited. One of the greatest challenges in the 21st century is providing the world's population with the energy it needs without significantly raising the concentration of greenhouse gases in the atmosphere A significant fraction will have to come from solar cells taking advantage of the sunlight bathing our planet. Organic photovoltaics show great promise for providing cost-effective and lightweight solar panels.

Development of new energy sources and improving the efficiency and exploitation of existing systems requires detailed understanding of both structure and behaviour at the fundamental microscopic level. This is an area where the powerful X-ray beams of a synchrotron radiation source play a major role.

Research at many light source facilities is aimed at understanding and improving the multilayer materials that compose an organic solar cell. One such effort is at the Molecular Sciences Research Center at the University of Puerto Rico, which is leading the LAAMP effort to advance light source and crystallographic sciences in the Caribbean.



P3HT:PCBM:Au NPs ITO Glass Image: B. Paci, Istituto di Struttura della Materia, C.N.R., Rome.

Schematic diagram of the organic photovoltaic cell used for the experiment. Organic photovoltaic device local structure revealed by combined X-ray diffraction and fluorescence. adobe.

Large arrays of solar panels are becoming more and more common



Study of materials under extreme conditions

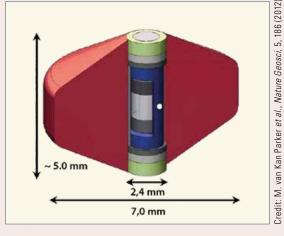
THE study of the effects of pressure and temperature on material properties f L is fundamental. These studies are relevant to many problems in condensedmatter physics and chemistry, Earth and planetary sciences, and materials science and technology.

In particular, high-pressure and high-temperature research is vitally important for studying the composition, thermal state and properties inside the Earth and other planets. Measurements of properties of rocks and plausible mantle and core mineral phases under appropriate conditions of pressure and temperature, together with the interrelationship of such data with geophysical and geochemical observations, are indispensable for the understanding and the modelling of planetary interiors.

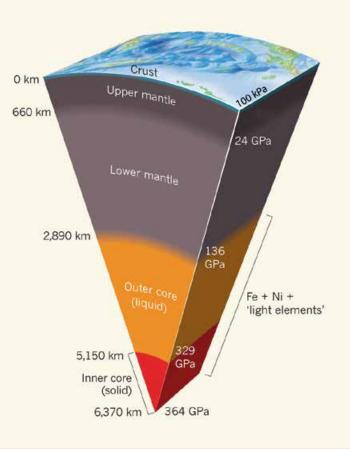
Synchrotron radiation is powerful and can penetrate the highly absorbing walls of the pressure vessels in which the samples are contained.

Furthermore the high brilliance beams are ideal for micron-sized foci needed

Some of these studies have been conducted by a group Johannesburg using the beams at the ESRF, the European Synchrotron Radiauon Facility in Grenoble, France. South Africa recently became a Scientific Associate



Mechanical cell for exposing materials under high pressure to the beam.



Cross section of the Earth's interior. The lower mantle extends from a depth of 660 km to a depth of 2,800 km. The pressures are in units of GPa (giga Pascals; one GPa is roughly 10,000 times atmospheric pressure at sea level.)



Applications to forensic science

Synchrotron radiation provides a tool to study trace evidence: glass, gunshot residues, pigments and biological samples such as human hair. Imaging can be done at high resolution and sensitivity. Energy tunability gives powerful chemical identification and mapping through X-ray absorption spectroscopy. These techniques are particularly suitable for small sample sizes.

Hair Analysis. Trace-level components of blood supply become incorporated into the growing hair cells in the hair bulb. As the hair grows each segment of growth shifts

The Case

Phar Lap

outwards, producing a timeline of the individual's blood concentrations. The hair may tell us a great deal, such as dietary intake, exposure to pollutants, effects of disease, and profiles relating to ingestion of drugs including performance enhancing drugs.



Phar Lap was a famous Australian horse with an incredible racing history in the late 1920s and early 1930s. Many acclaimed him to be the best race horse in history. In 1932 Phar Lap died suddenly in suspicious circumstances. Many people attributed this death to murder by arsenic poisoning but the autopsy proved inconclusive. Such was their love for Phar Lap that his skeleton, his heart, and his mane are displayed in museums in New Zealand and Australia.

APPLI

Forensic hair analysis was performed on a small sample removed from Phar Lap's mane. The X-ray beam from the Australian Synchrotron in Melbourne revealed a distinct band of arsenic in a location between the hair bulb and the skin level consistent with Phar Lap receiving a large acute exposure to arsenic just prior to his death. The announcement of this poisoning evidence resulted in major press coverage, not only in Australia but internationally.



Reference: I. M. Kempson "Synchrotron XRF and XAS Analysis of Hair for Toxicological and Pharmacokinetic studies, applications and artifacts," *IAEA TECDOC*, No. 1803, p. 93-102, 2016

Research on ancient fossils. Palaeontology



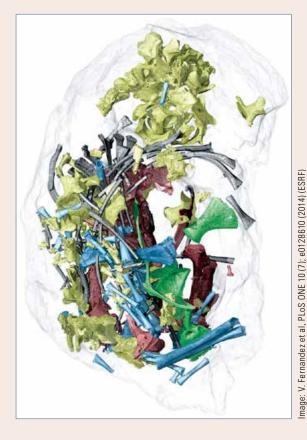
Skull of a 200-million-year-old fanged dinosaur. The fossil was scanned with X-rays at ESRF, using a beam 100 billion times more powerful than those used in hospitals. This allowed the research team from the Evolutionary Studies Institute at The University of Witwatersrand (Wits) in South Africa to peek "inside" it for the very first time.

The ESRF is supported by 22 partner nations, of which 13 are Members and 9 are Scientific Associates. Not limited to European nations, the ESRF's most recent affiliate is South Africa.

Palaeontology

Palaeontology is the scientific study of life that existed up to roughly 11,700 years before present. It includes the study of fossils to determine organisms' evolution and interactions with each other and their environments. The interior of a one-centimetre-long fossilised egg. The eggs were found in northeastern Thailand and turned out to be 125 million years old. Using micro tomography imaging to scrutinise the hidden embryonic skeletons preserved in the egg, the team now knows the egg layer's true identity—an anguimorph lizard, a category that includes Komodo dragons.





Research on ancient materials. Archaeology

Virtually reconstructed dentition of a Neanderthal child. Synchrotron virtual histology reveals precise developmental information that is recorded in the form of tiny growth tracks inside the teeth (background).



Source: Paul Tafforeau and Tanya Smith (ESRF)

Archaeology

Archaeology is the study of human activity through the recovery and analysis of material culture. The archaeological record consists of artifacts, architecture, biofacts, and cultural landscapes. Archaeologists study human prehistory and history, from the development of the



Restored in 1831, the **Temple of the Emerald Buddha** was redecorated with fine patterns of very thin coloured glass mirrors. The mirrors resemble natural gems more than ordinary mirrors. Unfortunately, this fine art of mirror decoration has disappeared for almost 150 years. The ultimate goal of exposing the glass mirrors to the beam at the Synchrotron Light Research Institute in Thailand was to quantify chemical composition and trace transition metals, and thus successfully enable restoration to their previous grandeur.

first stone tools at Lomekwi in East Africa 3.3 million years ago up until recent decades. Archaeology is a field distinct from the discipline of palaeontology, the study of fossil remains. Concerning prehistorical studies, archaeology is particularly important for learning about prehistoric societies, for whom there may be no written records. Reference: W. Klysubon *et al.*, "Characterization of the Ancient Decorative Mirrors from the Grand Palace Bangkok by SR-Based techniques," Forensics and Materials Science," *IAEA TECDOC*, No. 1803, p. 86-92, 2016



High technology centres feed industry

THE microscopic and nanoscopic structures of materials are directly related to their macro-scale properties and to their optimisation for modern day manufacturing processes and recycling.

One of the major users of advanced light source facilities is the pharmaceutical industry to obtain knowledge of the three-dimensional structure of protein targets and protein-complexes.

Herceptin – used to treat advanced breast cancer – benefited from synchrotron experiments. Using synchrotron light in the infrared range, pioneering research is underway into developing new cancer therapies that can be tailored to the individual patient.



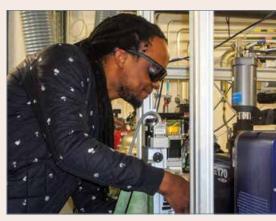
and SASOL





SASOL is an international corporation in South Africa and a leader in the industrial applications of light sources. Synchrotron radiation is used to study catalysts and their action on microstructures under varying conditions of temperature and pressure.

Hester Esna du Plessis is carrying out a high resolution X-ray diffraction experiment for catalyst characterisation.



Duminsani Kama is adjusting a microcrystal to be irradiated by a powerful short laser pulse. He is working inside the "hatch" of beamline ID09 at the ESRF and is wearing special eye protection. The catalyst's photo-chemical activation will be induced, and then the re-equilibrium will be studied with X-ray crystallography.

The impact of advanced light sources on science and society in developing countries

D RAZIL is a model for establishing a D synchrotron radiation facility in a developing country. It designed its first synchrotron light source, called UVX, in 1985 and launched in 1997. Initially, it was difficult to convince key parties of the benefits from having such a facility in one's own country. At its opening, researchers submitted only a few proposals, but the number of proposals grew quickly by orders of magnitude. Brazil is now internationally recognised for the quality of its research, particularly in structural biology, and currently is constructing a new light source called Sirius.

Similar experiences in South Korea and Taiwan led each of these countries to invest hundreds of millions of dollars in new light sources. These are examples of how excellent scientific research centres can improve local scientific and private enterprise opportunities. Dozens of mid-career diaspora scientists have returned home when they realised they can do world class research at their own light source.

UVX





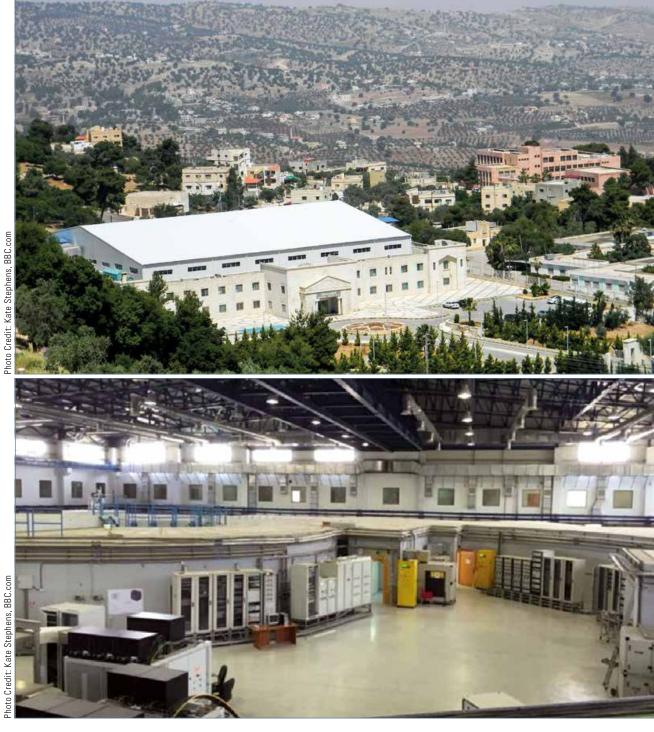


SESAME, Synchrotron light for Experimental Science and Applications in the Middle East, is an advanced light source facility in Allan, Jordan, near the capital of Amman, established under the auspices of UNESCO and closely modelled on CERN. This advanced synchrotron serves a wide spectrum of disciplines including biology and medical sciences, materials science, physics, chemistry and archaeology.

Beginning operations in 2017 the **SESAME** countries are Cyprus, Egypt, Iran, Israel, Jordan, Pakistan, Palestinian Authority and Turkey.

The European Union is an active supporter of **SESAME**, providing support via its Research Framework Programmes with projects such as CESSAMag (CERN-EC Support for SESAME Magnets) and OPEN SESAME.

SESAME combines capacity building with peace building through science and is a model project for other regions.





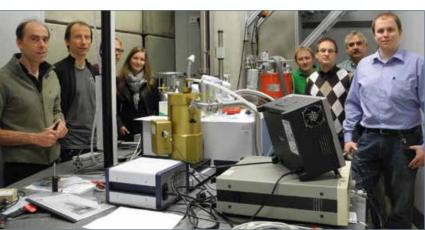
Jobs and careers at light sources

DESIGNING, building, maintaining, operating and using these complex machines require an enormous range of scientific, engineering, technological and industrial skills. There are career opportunities both at these facilities themselves and with the many scientific user groups that do experiments there. They are unique centres of education for young technicians, engineers and scientists. There are

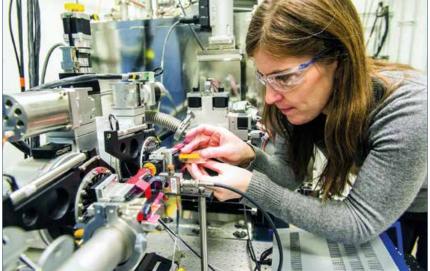
encounters among researchers from different groups studying different disciplines. There are budding friendships between researchers from different countries, universities and institutions. Encounters take place in the cafeteria, the library, the computing centre and at presentations of scientific results. This is part of the excitement of working at such a facility.

University of Saskatchewan bioarchaeologist Dr. Angela Lieverse conducts research on a rare skull from the Bronze Age.





Installing a new narrowband terahertz system as part of an accelerator improvement program by a group of scientists, technicians and engineers at DELTA, the light source in Dortmund, Germany.



Mary Upton (X-ray Science Division, Advanced Photon Source, Argonne National Laboratory) aligning a high-resolution monochromator in preparation for a resonant inelastic X-ray scattering experiment.

Future

THE international research community is collaborating to construct ever more intense sources of electromagnetic radiation to address the most challenging questions in living and condensed matter sciences. X-rays were used to determine the double helical structure of DNA by Rosalind Franklin to revolutionise biology. It must have taken Franklin weeks to get this pattern using conventional X-ray tubes in 1952. This can now be done with a synchrotron light source in seconds. In order to understand and deal with health problems associated with thousands of proteins, the detailed structure of these proteins must be determined. This is a main job of light sources and they are being used to study viruses. Biomedical, environmental, human heritage issues and concerns are local, which is why so many light sources are needed. Although there are more than 50 light sources and hundreds of beamlines, the user communities have grown faster needing light sources for their fundamental, applied and industrial research. There is often a wait of months or years to get access.

These new endeavours will face challenges. But they share with SESAME the goals of building regional capacity and promoting understanding, friendship and peace, bringing together scientists from different countries and ethnicities to perform world class science.

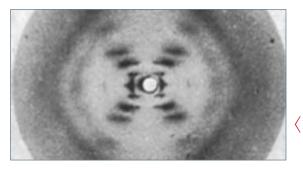
Sekazi K. MTINGWA and Herman WINICK, "SESAME and beyond," Editorial, *Science Magazine*, May 20, 2017



The High Energy Photon Source (HEPS) will be built in China in 2018.



MAX IV Laboratory, Swedish National Synchrotron, inaugurated 2016.



X-ray diffraction pattern of DNA by Rosalind Franklin in 1952



Photo: Roger Erikso

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Tools of Discovery and Innovation



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To learn more about LAAMP http://laamp.iucr.org



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