SYNCHROTRON RADIATION
AND
APPLICATIONS

The following brief description of synchrotron radiation, its applications and demand is provided for the benefit of the non-specialist; for a fuller, more scientifically based, survey the reader is referred to the citations and many other published papers.

A. SYNCHROTRON RADIATION AND SYNCHROTRONS

Synchrotron radiation When electrons (or any charged particle) are accelerated to keep them in a circular path they will radiate electromagnetic radiation *in a narrow beam* in the direction that they were traveling. This radiation is called synchrotron radiation (SR) or synchrotron light (SL). SL has a particularly interesting history. The first observation, literally since it was visible light that was seen, came at the general Electric Research Laboratory in Schenectady, New York, on April 24, 1947. Although natural SR from charged particles spiraling around magnetic-field lines in space is as old as the stars – for example the light we see from the Crab Nebula – it was necessary to wait until the 1960's to see the start of the use of SL in accelerator; short-wavelength SR generated by relativistic electrons in circular accelerators is only a half-century old. "Synchrotron" is the name given to any circular accelerator that uses microwave electric fields for acceleration and magnets for steering.

Synchrotron facility A synchrotron facility has a size of football field *or more*. It produces light with special qualities such as extreme brightness and short wavelength that permit unprecedented scientific and technological research. Like a giant microscope, this brilliant light source allows matter to be "seen" at the atomic scale. With a fine, intense beam that's only about the width of a human hair, scientists can analyze molecules, biological samples, and materials with higher accuracy and precision than has hitherto been possible.

Advanced Photon Source at Argonne National Laboratory

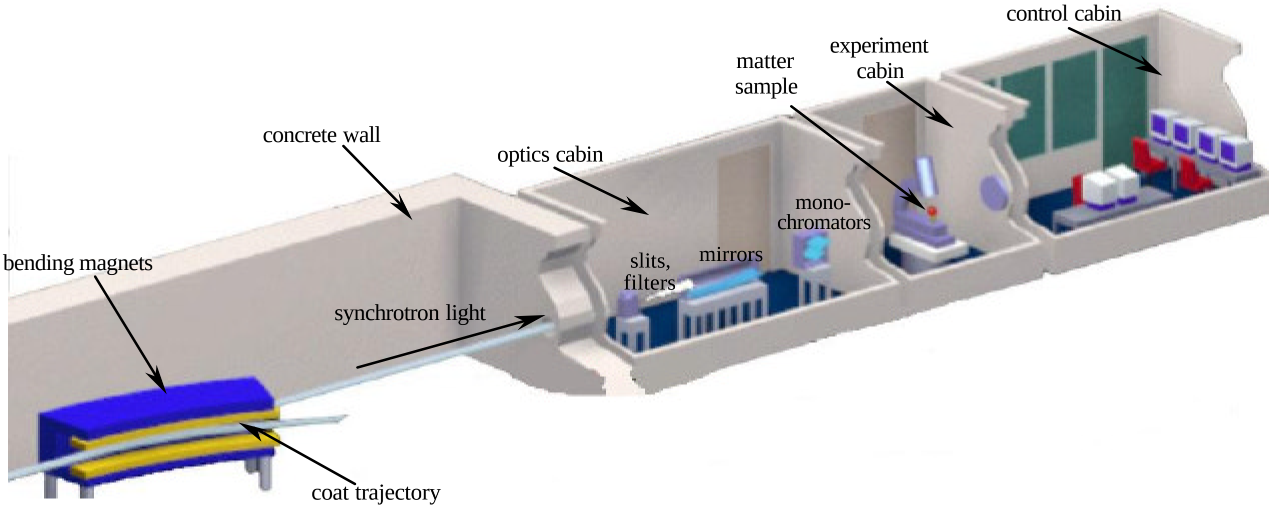
SR Facilities in the World In the 50 years since SR discovery facilities around the world constantly evolved to provide this light in ever more useful forms. These sources are expensive and few therefore exist; these are operated as either national or international facilities. Total investment in these facilities now is in the billions of dollars. There are now about 50 operational rings of all generations used as SR sources in 17 countries, about 10 of which are third generation sources. With the exponential type growth in the usage of synchrotron radiation, the number built in the past twenty years has increased to over 40 facilities throughout the world, concentrated principally in the U.S., Europe, and Japan. More recent and notable efforts to develop such facilities are ongoing in the United Kingdom, Canada, and Australia together with upgrades of existing facilities in the U.S.

Location	SR Facility/ Storage Ring	Energy, GeV	Note*
NORTH AMERICA			
Canada,			
Saskatoon	CLS	2.5	DS
USA,			
Berkley, CA	ALS	1.5-1.9	DE
Argonne, IL	APS	7	DE
Baton Rouge, LA	CAMD	1.4	DE
Ithaca, NY	CHESS	5.5	PD
Stoughton, WI	SRC	0.8-1.0	DE
Stanford, CA	SSRL	3-3.5	DE
Gaithersburg, MD	SURF III	0.28	DE
Upton, NY	NSLS I	0.75	DE
	NSLS II	2.5-2.8	DE
Durham, NC	DFELL	1-1.3	DE/FE
SOUTH AMERICA			
Brazil,			
Campinas	LNLS-1	1.15	DE
	LNLS-2	2.0	DS/DE
EUROPE			
Germany,			
Karlsruhe	ANKA	2.5	DE
Berlin	BESSY I	0.8	DE
	BESSY II	1.7	DE
Dortmund	DELTA	1.5	DE/FE
Bonn	ELSA II	1.5-3.5	PD
Dresden	ROSY	3	DS/DE
Hamburg	HASYLAB/ DESY: PETRA II	7-14	PD
	DORIS III	4.5	DE
Denmark,			
Aarhus	ISA: ASTRID I	0.6	PD
	ASTRID II	1.4	PL/DE
Spain,			
Barcelona	LLC	2.5	DS/DE
France,			
Greboble	ESRF	6	DE
Orsay	LURE: DCI	1.8	DE
	super ACO	0.8	DE
	SOLEIL	2.5-2.75	PL/DE
Italy,			
Frascati	DAFNE	0.51	DE
Trieste	ELETTRA	1.5-2	DE
United Kingdom,			
Didcot	DIAMOND	3	PL/DE
Daresbury	SRS	2	DE
	SINBAD	3.0	DS/DE
Sweden,			
Lund	MAX: MAX I	0.55	DE
	MAX II	1.5	DE
Switzerland,			
Villingen	SLS	2.4	DE
Nitherlands,			
Amsterdam	AmPS	0.9	PL
Eindhoven	EUTERPE	0.4	PL
CIS COUNTRIES			
Russia,			
Moscow	KSRS: Sibir-1	0.45	DE
	Sibir-2	2.5	DE
Novosibirsk	BINP: SSRC	0.8	DE
	VEPP-2M	0.7	PD
	VEPP-3M	2.2	PD
	VEPP-4M	5-7	PD
Armenia,			
Yerevan	CANDLE	3	DS
ASIA			
South Korea,			
Pohang	PLS	2	DE
China,			
Beijing	BSRF	1.6-2.8	DE/PD
Hefei, Anhui	NSRL	0.8	DE
Shanghai	SSRF	2-2.5	DS/DE
India,			
Indore	INDUS I	0.45	DE
	INDUS II	2	DS/DE
Japan,			
Hiroshima	HISOR	0.7	DE
Ichihara	NANO-HANA	1.5-2	DS/DE
Tsukuba	PHOTON FACTORY	2.5	DE
Nishi Harima	SPRING 8	8	DE
Okazaki	UVSOR	0.75	DE
Tokyo	VSX	1-1.6	PD
	SOR-Ring	0.5	DE
Tsukuba	NIJI-II	0.6	DE
	NIJI-IV	0.5	DE/FE
	TERAS	0.8	DE
	NIJI-III	0.6	DE
Nishi-Harima	NewSubaru	1-1.5	DS/DE
Kusatsu	AURORA	0.6	DS/DE
Kashiwa	HBLS	2-2.5	DS/DE
Osaka	Kansai SR	1.8	DS/DE
Kyoto	KSR	0.3	DS/DE
Sendai	TLS	1.5	DS/DE
Singapore,			
Singapore	HELIOS 2 or SSLS	0.7	DE
Taiwan,			
Hsinchu	SRRC	1.3-1.5	DE
AUSTRALIA			
Australia,			
Viktoria	BOOMERANG	3 GeV	DS

* **DE** – dedicated (synchrotrons built solely to access the electromagnetic radiation emitted); **PD** – partly dedicated; **DS** – design; **PL** – planned use; **FE** – FEL (free electron lasers) use

In comparison to the U.S. and Asia the present situation in the field of synchrotron –radiation research in Europe can be described as follows: Europe has played a leading role in the development of the field, starting from the pioneering work at DESY in the early seventies, the construction of early dedicated XUV-sources, like BESSY and ACO in the eighties, up to the creation of more modern third-generation synchrotron-radiation sources in the nineties, like ESRF, ELETTRA, MAX II, BESSY II, and the Swiss Light Source. Europe is again leading in the construction of free-electron X-ray lasers, like the TESLA Test Facility at DESY, Hamburg, and there are further new facilities in various European countries in planning and under discussion.

How does synchrotron work? SL is produced by charged particles (electrons) circulating in a ring at almost the speed of light. Electrons are deviated by the magnetic field of the bending magnets distributed all along the circumference. When they are forced onto a coat trajectory electrons emit the SL composed of bright infra-red, ultraviolet and X-ray light. The beams of light are emitted tangentially to the the trajectory of the electrons. They follow a straight direction towards the beamlines that are installed in the experimental hall on the other side of a concrete wall. Beamline is an instrument, tool, designed for certain experiments. A beamline is composed of a series of cabins. The first one is called the optic cabin and comprises such instruments as slits and filters, mirrors and monochromators. Close to it is the experiment cabin that with a sample of matter to study and the detector. At the end we find the control cabin. In front of their computer screens scientists monitor the experiment and the data acquisition.



What makes SR unique?

- The intensity of radiation emerging from storage ring can be of the order of a **billion** times greater than radiation from a typical laboratory x-ray source.
- The emerging beams are extremely **fine** – just a few microns across.
- The divergence of the beam is very low – that is, the beam is highly **collimated**.
- The radiation extends from the infra-red to hard x-rays. Unlike most sources it is **tunable**.
- Intense brightness and tenability across the spectrum combine to give SR its invaluable uniqueness.
- SR is pulsed with pulses typically 10 to 100 picoseconds in length separated by 10 to 100 nanoseconds (**time structure**).

B. RESEARCH USE AND TRENDS

SR is used to study structural details of matter, on a scale that is sensitive to the placement of individual atoms. The various synchrotron-radiation-based methods provide information on the spatial structure of the materials, on the chemical and electronic structure, on microstructure, on the properties of surfaces, interfaces, thin films and multilayers. As a result, materials research now has a tool that can probe in minute detail the interior and surface of all manners of samples, large and extremely small, including non-crystalline and heterogeneous materials. SL is one of the most powerful (including and exchanging many methods) tools available for exploring matter – from cross-sectional images to the nanosecond-by-nanosecond behavior. Therefore, it is an indispensable tool for pure and applied research in a great variety of areas, offering new opportunities for state-of-the-art investigations. The investigations are usually academically oriented and are relevant to many disciplines:

Physics Techniques using x-rays provide physicists with varied information not only about the geometrical structure of matter, but also about its fine structures. SL, with its high brilliance, opens completely new fields of research, like the study of surfaces (work functions, boundary features, thin films, surface depositions, contamination, etc.), magnetism, and samples submitted to extreme conditions of temperature and pressure.

Materials science covers the very wide field of human made-materials, from the most traditional ones, like glass, to the newest ones, like composites. A large variety of techniques are used in order to cover the diversity of the material structures studied: plastics, colloids, glass, cement, composite alloys, superconductors, etc. The researchers are also interested in materials found in nature, particularly in biological materials, which are very complex, and thus could be a source of inspiration for the materials of the future. Some biological materials like wood and spider silk have remarkable properties, associating strength and flexibility. Other biological materials are used in the food industry, such as chocolate, made from cocoa. SR provides information about arrangement of the atoms in complex materials including chemical and biological materials. It also provides information about the changes in these atomic arrangements as these materials carry out their biological or chemical function. Since structure of chemical or biological materials often determines function, knowing the detailed structure can give us insight into their chemical or biological activity or tell us how to improve manufactures materials. This is why researchers are trying to elucidate their microstructures.

Materials research is concerned with both bulk condensed matter and surface phenomena investigating both steady state and dynamic phenomena. The construction of many dedicated synchrotron-radiation facilities in most of the technologically advanced countries in the eighties, and especially the appearance of third-generation synchrotron-radiation facilities in recent years, has enormously expanded the classical means of characterizing materials. Worldwide, about 70% of all beam time on synchrotrons is used for materials research in which SL has been used to explore the properties of materials. SL is used abundantly to explore chemical composition, crystal structure, electronic structure, to better understand the growth and properties of crystals such as those used in semiconductors, for the development of ceramics, to better understand such phenomena as corrosion and metal fatigue.

-**New materials, their contribution to our future** The last decade in particular has seen an explosive increase in the rates of design, production and utilization of new materials. The remarkable advances that have been made will continue to pervade every corner of our lives and will increasingly influence manner in which we live, work, travel, communicate with each other and spend our leisure time. These advances have occurred right across the spectrum of physical, chemical, biological and engineering sciences so that it is now feasible to design, synthesize and to characterize substances on the molecular level, to assemble them into everyday use in new applications. Further progress in almost every field of human endeavor depends on the availability of new materials: their production is now a key enabling technology for the future.

Scientists work in the atomic and molecular world and their task in making new materials is to assemble combinations of the atoms of the more than 100 known chemical elements into larger structures that constitute useful materials. Advances in a range of scientific disciplines mean that materials that will display particular desirable macroscopic properties and functions can now be designed at the atomic level. Educational programmers and research initiatives must seek to build and encourage a real multidisciplinary ethos between subjects and institutions. The research programmers at SR sources that are essential for the characterization of materials, promote and foster this ethos and could be further promoted as a focus for such developments in the future.

The presence of new materials is now ubiquitous. Here are some specific examples drawn from the more obvious areas in which they have a major influence.

- Nanotechnology This covers a whole spectrum of science incorporating molecular structures that are 1-100 nanometers in size and offers new perspectives in improving existing technologies. Nano-powders will provide radically new ceramics, catalysts, lubricants and coatings. Self-assembly of molecules into larger structures will revolutionize synthesis: early applications are likely to be in electronics and to include the production of very finely divided materials showing quantum effects.

- Electronics and communications The new technologies used in these areas provide the most visible and widely known applications of new materials. Brighter and faster liquid crystal displays based on new liquid crystalline materials will displace our traditional screens. Miniaturization and enhanced performance in the computer industry will exploit organic conductors: perhaps the greatest challenge here is the design of self-assembling molecular computers. The speed and efficiency gains in fibre-optic communication will continue to depend on challenging functional materials such as non-linear optic materials. Rapidly developing printing and imaging technologies will demand novel synthetic colourants and formulation processes.

- Energy generation and storage The energy requirements of the world will continue to increase, making new and sustainable technologies for the generation, storage and transport of energy essential. The materials challenges for high-temperature solid oxide fuel cells and for the direct methanol fuel cell are now well defined. New catalysts and the ability to make ceramic membranes are being developed. Improved batteries for applications ranging from portable electronics to vehicle propulsion and peak-shaving of power demands will find universal use.

- Medicine and health care The ever-increasing contributions of high-technology science to medical practice will continue. Biocompatible materials, surface modifications and coatings are required for the production of artificial tissues, bone, implants, grafts, joints and medical devices. Rapid diagnostic kits, including those sold over the counter for self-diagnosis, will become commonplace, Biologically active electrochemical sensors will be developed to provide therapy, preventative action and biofeedback to controlled drug delivery systems such as electrophoretically assisted transdermal patches. Nanoscale chemistry will lead to new devices for monitoring specific functions and disease progression.

- Transport Here the need is to reduce energy consumption and the pollution caused by exhaust fumes. The lightweight engineering materials required range from composites for applications in power units and vehicle interiors to structural components designed to reduce injuries by absorbing the energy of collisions.

- The environment Many pressures, including resource conservation and cost reduction, combine to drive research efforts to find simpler materials that can easily be recycled. The 'green' chemistry initiative, which seeks to lessen or eliminate the use of hazardous substances and to promote source reduction, will lead to innovative cleaner methodologies and more specific and careful control of the production of materials in chemical syntheses.

The industrial contribution of SL to the development and application of new materials is described in the next section.

Biological and Life Sciences Most of the biological functions (from respiration to digestion) involve very large molecule, the biological macromolecules (proteins, enzymes, viruses…). The function of a biological macromolecule is directly linked to its structure, i.e. its spatial arrangement. The knowledge of this structure is a major goal for scientists. Structural analysis techniques have progressed and the latest breakthroughs due to SL allow numerous structure-function studies, particularly the quantitative identification of the atomic assembly of large biomolecules and the determination of the microstructure of biological tissue (muscle, ligament, tendon) in dynamic situations.

The SR techniques used for structural biology can be sub-divided into four principal areas: crystallography, spectroscopy, scattering from noncrystalline materials and imaging. The first major step in the biotechnology revolution was unravelling the structure of DNA. During the past ten years the number of protein structures elucidated by X-ray crystallography has risen more than tenfold. At last count there were more than 17,000 protein structures available in the Protein Data Bank. Driving this increase has been the use of SL sources to solve protein structures: from less than 30% in 1990 to more than 80% at the end of the last decade. SL provides two features essential to protein crystallography: speed and accuracy. The former is important in any structural genomics project and the latter is considered vital in structure-based drug design as many diseases are due to malfunctioning proteins. Research into the 3-dimensional structure of proteins underpins the development of 'designer drugs' with specific therapeutic activity. Such designer drugs are being developed in the pharmaceuticals industry and are widely seen as the way of the future.

SL has revolutionized the field of protein crystallography, but it can also be used to study enzymes that are not available in a crystalline form. It is also useful for the study of fibre structures, such as muscle tissue and the structure of wool fibres. Membranes, inherently difficult to study, can also be explored using SL.

The Structural Biology Synchrotron Users Organization (BioSync), formed in 1990 to promote access to SR for scientists whose primary research is in the field of structural biology, published a report in 1991 on the status of structural biology research using SR in the U.S. based on the results of surveys of both the SR facilities and the structural biology research community. The main conclusions of the report were that structural biology was a very rapidly growing field with a growing impact on basic and applied biology, and that the SR facilities available at the time were insufficient for the needs of this ever-expanding community. Both construction of additional beamlines and improved support for existing beamlines were recommended to meet the predicted need. In its most controversial conclusion, the study group predicted a very large “latent” demand for synchrotron beam time from biologists who were not specialists in structural methodologies. More streamlined structural experiments coupled with an intense demand for new macromolecular structures were the driving forces for the “latent” demand. The report has been updated in 1997 and, as anticipated, the impact of structural biology in all areas of biological sciences has expanded greatly. With this continued development has come an increase in both the size and the complexity of the macromolecular structures that are being determined and in the difficulty of the experiments that are being pursued. This increase in complexity, which was not expected to appear so quickly, has meant that synchrotron-based structural biology has expanded its role and now makes a significant contribution in addressing the fundamental questions of how life processes are carried out and the practical applications of treating diseases at the molecular level. So, some of the main conclusions are the following:

- Structural biology research is producing results of high biological impact that have a direct bearing on human health issues. Structure-based drug design, which seemed merely a trendy phrase a few years ago, has become a reality. The design of new medically important drugs is a direct consequence of research in structural biology. Structural biology is also becoming increasingly important in biotechnology, as for example, in the design (or re-design) of enzymes to degrade pollutants or to act as thermostable industrial catalysts or in the design of insecticides with increased efficacy. These applications can have huge environmental and economic impacts.

- SR is now a dominant contributor to new macromolecular structures. The role of SR in structural biology has been growing rapidly over the last twenty years. A recent survey of the literature shows that SR was used in nearly half of the new structure determinations. The benefits of SR include substantial increases in resolution over those available with laboratory sources and the ability to study crystals that are too small or have a unit cell too large to be studied using home x-ray sources.

- The general demand for structural information in all molecular fields of biology continues to grow very rapidly, and is paralleled by a growth in the demand for synchrotron time. Three factors contribute to the substantially increased demand:

a. Technological improvements in synchrotron facilities, x-ray detectors and crystal handling have brought many more biological problems into the range of structural biology and have significantly improved the success rate and quality of synchrotron experiments.

b. As the complexity of the biological project increases, there is a greater demand for synchrotron time to tackle more difficult problems. For such complex problems, the ability to integrate information from several types of experiments is often critical.

c. As anticipated a significant new demand has indeed come from a “latent” community of users who are not specialists in crystallography but who have biologically significant structure determinations to carry out. Latent demand is difficult to quantitate, but it is already clear that many non-specialist laboratories are embarking on structure determinations. Non-specialist users are a greater challenge for synchrotron facilities because they usually need a higher level of assistance from scientific staff. In addition, the synchrotron facility may be their only available x-ray source.

- Regional facilities will grow in importance. Without question, there is a strong demand for regional facilities that can provide service to the regional scientific community. It was deemed of extreme importance that research groups be within driving or short flying with distance of synchrotron facilities to exploit their resources fully. The ability to drive to a local facility with samples in hand was rated as extremely important by a majority of users. Graduate students and postdoctoral fellows are the majority of scientific workers who actually go to the synchrotron, and it is essential that proximity to synchrotron facilities allows them to travel in large numbers for training.

Medicine Medical procedures consist of identifying illnesses with the aim of treating them effectively. Therefore, one of the objectives of medical research is to deepen the knowledge of pathologies. To make a diagnosis about a patient, in addition to questioning him, a physician can have disposal a collection of complementary tests, among which imaging techniques are increasingly used. Medical applications of SL span from diagnosis to the treatment of diseases. Medical research benefits from recent progress made in x-ray imaging techniques using the coherence of SL. It can also apply some of the results obtained by biologists using other SL techniques.

- Brain: Computed Tomography; Microbeam radiation therapy
- Heart: Angiography (x-ray study of the heart and blood vessels to reveal obstructions)
- Lungs: Imaging of tumors
- Breasts: Mammography
- Immune system: AIDS
- Nervous system: Alzheimer's disease; Neuron demyelination
- Skin: Collagen fibers
- Arteries: Atherosclerosis
- Bones: Osteoporosis

Some medical scanning applications are moving into hospitals. In Japan, for example, one hospital is being sited near to the Spring 8 synchrotron so that it can be supplied with SL via a 3 kilometre beamline.

Chemistry The remarkable properties of SL make it an ideal tool to study chemical reactions and compounds, as well as complex processes not easily addressed by 'wet' chemistry. For example, many industrial processes rely on the use of catalysts which can be analyzed using specific techniques. There is a strong interest in polymers, a class of macromolecules with an extremely wide range of applications. Moreover, SL has allowed breakthroughs in our understanding of some fundamental properties of water.

Environment Protection of the environment has become one of the main issues of world community. Significant effort has been made in the last years in scientific research. SL can be an invaluable resource in some studies related to the environment: for example the improvement of different energy sources, the fight against pollution as well as a better understanding of natural phenomena. SR is greatly used in the analysis of contaminants and in understanding their release into the environment, their mobility and toxicity are also becoming evident.

Earth science The underlying goal of earth science is to understand the processes that formed the earth, and the concentrations of metals, minerals and fossil fuels in its crust. The unique and very powerful characteristics of SR provide wonderful new opportunities for increased understanding of earth science processes, in the natural environmental and in the laboratory and industrial context. SR provides natural environmental researchers with the ability to study a range of fundamental and applied problems falling in two main areas: Molecular Environmental Science, and Mineral Physics and High Pressure Petrology. In the first, the aim is to study the fundamental processes controlling the geochemical and biological influences on element cycling between the crust, hydrosphere, biosphere and atmosphere at a molecular level. In the second, the aim is to obtain a better fundamental understanding of the relationships between physical properties and atomic structure, and of the mineral stabilities and equations of state for minerals in the deep crust and mantle.

The earliest applications and development of conventional X-ray technologies at the beginning of the last century were strongly focused on earth science materials. Over the past 20 years minerals have continued to play a central role in the development of the new generations of SR techniques and the range of applications of synchrotron X-ray technique to the study of the earth sciences has grown enormously. It is anticipated that in the next ten years not only will the current techniques continue to be developed in terms of elemental sensitivity, the range of environments available, mapping capabilities, surface sensitivity, etc, but also that new techniques will be developed.

Synchrotron facilities are among the most versatile of all research facilities. Synchrotron research is developing rapidly in a number of key areas, and there is ample confirmation of increasing demand for synchrotron facilities world-wide from both scientific and industrial users. That is evidenced in research use levels and trends and in the willingness of an increasing number of countries to construct new facilities. Sweden's MAX I facility is reported to be overbooked by 3 or 4 times its availability. And in Asia, it is reported that full capacity has been reached with existing machinery at most sites. International experience suggests that use of national facilities doubles within the first 4 years, and each 6 years after that. It is worth noting that none of the older 2nd generation facilities in the US has been decommissioned as newer 3rd generation facilities come online – all remain in active use.

C. INDUSTRIAL APPLICATIONS AND DEMAND

Early SR sources were constructed principally to provide facilities for multi-discipline academic studies but it was soon realized that they also represented a unique source of radiation for a wide range of industrial research and development studies. As synchrotron provide high intensity light across a wide spectral range including infra-red, visible, ultraviolet and x-ray radiation, they are used as an effective probe to understand the underlying structures and properties of matter, and analyze physical, chemical, geological and biological processes. Hence, the information that can be generated through synchrotron research is of immense importance to various industries. SL opens the door to many applications in the pharmaceutical, mining, petrochemical, advanced materials, electronics, manufacturing and health industries. A number of impressive examples of SR usage in applied research and industries are the development of new drugs, design of new microchips for more powerful computers, manufacturing of tiny biomedical implants, creation of new materials such as stronger metal alloys for airplane wings, development of new injection-moulded materials such as jogging shoes, car bodies and bumpers, and furniture foam, development of solvent-free paints, manufacturing of biodegradable plastics that could be eaten by bugs, studying the surfaces and interfaces between materials which can help tackle corrosion problems in cars, planes and pipelines, manufacturing of microscopic machines such as motors so small they can fit through the eye of a needle, evaluation of the performance of microchips, tracing the distribution of pollutants in natural systems, determination of the lead content in clay, performing research which helps develop ultra-thin lubricants, establishment of the value of a possible mining site by analyzing ore samples.

From the experimental stations that are requested, it is known that **the most frequently used industrial techniques** are:

- *glancing angle x-ray reflection/diffraction*, probing the surface of a material at variable depth;
- *energy dispersive x-ray diffraction*, a fast process which allows the identification and classification of phase transitions during dynamic changes (temperature, pressure, chemical reactions, etc.);
- *high resolution powder diffraction*, a technique allowing the identification of atomic structures of micro-crystalline materials; the technique is available in a conventional x-ray laboratory, but SR provides much enhanced speed and resolution;
- *x-ray spectroscopy (XAS)*, a technique unique to SR, which resolves atomic structures in non-crystalline/amorphous materials, sometimes with very low concentration of the 'target' element;
- *small/wide angle x-ray scattering*, two separate techniques which, when combined, provide structural information of one and two dimensionally ordered systems from the molecular to the macroscopic level;
- *protein crystallography*, a major technique allowing the full identification of the atomic structure of large bio-molecules, but only if three dimensional fully ordered crystals of the materials are available.

The techniques described above are all concerned with investigation and measurement. However, **SR is also used as a manufacturing tool**:

SR for Lithography The lithographic process used to manufacture microchips conventionally makes use of ultra-violet radiation and currently is able to produce structures with minimum line widths of the order of 0.25 µm. To obtain further reduction in size, achieving higher component densities with a corresponding increase in operating frequency, the use of x-rays for the exposure of the photo-resist is necessary; this appears to offer the possibility of resolving features of 0.07 µm or less. The parallelism and intensity offered by SR sources makes them eminently suitable for this work and investigations have been carried out at a number of laboratories. Whilst this technique has not yet matured to the stage of supporting routine manufacture, purpose built sources suitable for use in a manufacturing environment are now being designed and constructed.

LIGA This technology was developed in Karsruhe, Germany, to produce micro-mechanical components for isotope separation. The required accuracy indicates that lithographic methods are necessary but the specified thickness of the components calls for a process able to penetrate photo-resists to a substantially deeper level than the thin-film lithography used for microelectronics. The resulting LIGA (Lithographie, Galvanoformung, Abformtechnik) process uses SR to penetrate to depths of 0.5 mm or greater in the photo-resist. After development, to remove the exposed material, deposition by electrolysis fills the resulting voids and either directly produces the required micro-structure or provides a mould for replication. The micro-components so produced can be of a variety of materials (metals, polymers, ceramics), have height to width aspect ratios of up to 100:1 and side wall accuracies usually better than 1 μm. Unlike thin-film lithography, where soft x-rays provide the necessary penetration, the LIGA technique requires x-ray in the 5 keV range for optimum performance.

Pharmaceuticals Perhaps the most widely known industrial application of synchrotron-based research, and perhaps the most economically significant in the near term, is in the exploration of protein structures. Structural biology has developed rapidly over the last decade. The best technique for structural biology studies is x-ray diffraction. It is used to reveal the atomic arrangement of proteins, nucleic acids and viruses. For large protein complexes it is used to study the binding modes with substrate molecules, and therefore gives a unique understanding of the biochemical role. The outstanding properties of the x-ray beam provided by synchrotron sources are now essential for collecting high-quality diffraction data. Pharmaceutical companies appreciate the rapid data collection on very small crystals at higher resolution than with conventional x-ray sources for the research and development of innovative medicines. Traditional methods of drug development, based on the identification and isolation of active ingredients, selection involve a high degree of risk both in the drug development cycle and in the control of side effects. With R&D in pharmaceuticals accounting for an increasing share of revenue, and companies being so dependent on 1 or 2 major products, firms are vigorously pursuing the opportunities that such research presents to shorten the development cycle, create more certainty in that cycle and build more a reliable and predictable new drug product development 'pipeline', as well as reducing the cost. Rather than waste millions on the hit-and-miss discovery of drugs, pharmaceutical companies are now able to design drugs based on being able to observe and record how chemical molecules interact with living enzymes.

Cosmetics Developing new cosmetic products and assessing their effectiveness and safety for future consumers calls for very high-performance characterization tools. As a complementary technique to electron microscopy, x-ray analysis provides in-depth information about beauty creams, hair products, lipsticks or nail varnishes and their tolerance by skin or hair. By examining their microstructure, the cosmetics engineer is able to develop more stable products with longer-lasting effects.

The sound example is L'Oreal - one of the leading companies producing cosmetics. Since its creation, L'Oreal's policy has been to invest in R&D. Over the last ten years, the group has devoted nearly 2 billion euros (3% of consolidated sales) to cosmetics and dermatology research. A strategy that stimulates creativity: L'Oreal is number one in cosmetics patents. L'Oreal benefits from a complete research structure with a staff of more than 2300 people working in ten research centers all over the world and collaborates with several international scientific institutes. SL has been used several times to look at hair by L'Oreal researchers allowing them to study the molecular and super-molecular arrangement of hair keratin, as well as the effect of water and perms on this structure. They also showed that hair growth in vitro, in cellular culture, has the same structure as that naturally growing on the head.

Food products The food industry is constantly developing new food products to satisfy consumer trends in terms of novelty, ease of preparation and nutritional value (e.g. low calorie meals), whilst maintaining high standards of quality. To respond to this challenge, the sector is moving away from traditional cooking towards rationalized processing. This implies a huge nutritional research effort since foods are very complex mixtures of different components with widely diverse thermal, mechanical, rheological and ageing properties. Several food companies have chosen to take advantage of the wide range of analytical SR tools to improve existing products and explore the potential of new developments for such different foods as chocolate, butter, creams and drinks.

Plastics The place of plastics in everyday life is increasing rapidly. They compete successfully with traditional materials in widely different sectors such as transportation, home equipment, furnishings, clothing and packaging. The reasons for this are multiple: their low cost, their versatility, their ease of manufacture and of course their excellent properties. Whereas traditional materials – wood, leather, metals, glass, ceramics and natural fibers – have reached a steady state in their development and their full potential has been exploited, plastics still offer extensive development possibilities: from the synthesis of new polymers with better properties and the creation of composites and blends, to the improvement of manufacturing processes. All this requires a vast R&D effort to predict and control the behavior of future polymers, based on the understanding of their properties on a microscopic scale. The high performance characterization tools such as SL play an important role in extending this knowledge which is vital to the continuous progression of the properties.

Papermaking Trees represent a large part of all the biomaterials produced on the planet. The understanding of the structure and metabolism of wood is vital for the economy, due to its use as construction material and as a basic raw ingredient in the manufacture of paper. The papermaking industry, through recycling and improvement in processing, is trying to reduce negative ecological impacts. For example, a clean process for wood bleaching is called TCF (Totally Chlorine Free), using hydrogen peroxide as the bleaching agent. The efficiency of this process is influenced by the presence of various ions (Fe, Mn, Cu, Mg and Ca) in the wood fibers and, to improve the process, it is necessary to analyze the distribution of the ions inside the fibers. SL allows a quantitative microanalysis of very high quality.

Chemistry The knowledge of chemical reactions and compounds has significant implications in industrial research, in particular in the field of petrochemistry. For example, although present in almost all chemical engineering processes, catalysis mechanisms are generally not yet analyzed on an atomic scale. The demand for more efficient and selective catalysts, driven by economic and environmental factors, is at the origin of a world-wide research effort to rationalize the synthesis of new catalysts. Understanding the reaction mechanisms on an atomic scale is now a priority and is being addressed through absorption spectroscopy, a major x-ray technique that can be used to follow the local chemical environment of catalyst atoms during the reaction, and thus identify transient compounds not seen otherwise.

Construction The mechanical and chemical properties of the materials used in the building industry (cement, alloys, glass, etc) are keys for their current performance as well as for the ageing processes. Cements, for example, are somewhat empirically formulated, in order to find a compromise between fluidity and setting time. To put cement formulation onto a more scientific basis, some cement companies are now engaged in extensive research efforts. One of their objectives is to understand the complex setting process and to observe the many intermediate phases involved in it. Time-resolved (second timescale) and in situ experiments are now possible in synchrotrons. The prediction of the performance of building materials exposed to aggressive environments (water, for instance) is economically and technically important. Modelling water-induced ageing processes in order to develop more resistant materials requires the knowledge of transport coefficients, which in many cases depend on the porous structure of the material. Visualization of porous materials on a microscopic scale is made possible by new imaging techniques using SR.

Metallurgy Metallic components are subject to considerable mechanical stress during manufacture and throughout their operating life. These stresses cause deformation strain and fatigue of the component, which affect its performance and can lead to failure. In the aerospace, automative and construction industries, it is essential to have a perfect knowledge of the stress/strain relationships in many components that are critical for safety and service lifetimes. X-ray strain measurement in scanning mode, which is a well-adapted tool for stress analysis, provides this understanding. This technique reveals the deformation strain in metals by measuring the structural deformation of the metallic lattice at the atomic level. The stress forces are calculated from the strain map to identify zones with critical levels. The intense x-ray beams in synchrotrons can be used to analyze steel to a depth of a few mm with a very high dimensional resolution.

Mining and Minerals Synchrotron research and analysis also has enormous potential significance for the mining industry. It is increasingly being used in the identification and analysis of minerals, and in understanding their precise composition, crystal and electronic structures in exploration, and increasingly in remediation. Exploration can be assisted by the early identification of indicator minerals, and by the use of SL techniques to investigate very small samples or individual crystals within larger samples, or to characterize slight differences in crystalline structures. Synchrotron techniques are also of importance in understanding surface properties vital in reducing cost and increasing the effectiveness and efficiency of retrieval and separation of minerals.

Synchrotron techniques have been employed in the US in a number of environmental cleanup situations, including the remediation of soils contaminated with heavy metals and radioactive elements. Australian researchers are also using microprobe techniques for the study of airborne pollutants.

Micromachining High-technology materials used in the microelectronics industry deserve special attention, since much effort is put into their research and development. SL is used an increasingly wide range of micromachining and microfabrication applications that range from the control of silicon wafers to the characterization of semiconductors, including chip etching (lithography); new industrial techniques make possible the fabrication of microstructures - miniature mechanical components like motors, pumps, gears, valves and sensors. These devices are used in a range of industries, such as medical instruments and implants, automotive and aerospace applications, electronics and electrical equipment, for things like antilock braking systems, airbag systems, computer disk drives, and so forth.

In the *Automotive* industry, x-ray radiation is the critical emerging process technology for the fabrication of a wide range of micro-devices which are finding increasing application in vehicles. The development and production of cost effective actuators and sensors in applications like anti-lock braking systems, airbag safety and ignition systems depends upon micro-machining using x-ray technologies for cutting and shaping.

Micromachining is also emerging as the critical technology in many areas of *scientific and medical instruments* where miniaturisation and lowering production costs are particularly important. Over the next 5-10 years local manufacturers are looking to develop products based on the “lab-on-chip” concept. These plans involve the use of x-ray radiation for micromachining.

A recent US Government report on Microelectromechanical Systems (MEMS) indicates that some 20 large US companies have incorporated MEMS into the products in order to increase efficiency and reduce costs. They include: Honeywell, Motorola, Hewlett- ackard, Texas Instruments, Xerox, GM Delco, Ford and Rockwell. A further 60 companies have MEMS activities of a more preliminary nature.

There are numerous applications of SL for the *microelectronics* industry, but the best known is microlithography. We are all familiar with lathes and milling machines for shaping parts in machine shops and factories. But what if the parts we need to make are significantly smaller than a millimeter, and featuring details even smaller? Semiconductor chip manufacturers have faced these problems and have learnt to use new ways to make devices. No longer are transistors made one at a time, but rather are ‘printed’ millions at a time, together with their interconnection wiring in a process called photolithography. Photolithography is today a standard process in semiconductor FAB plants and has several critical advantages in terms of cost, reproductivity, reliability and its ability to scale towards ever smaller and more complex systems. Compared to the resolution obtained in optical lithography, which is limited to about 500 nm, the SL offers a resolution better than 100 nm. One can then speak of “nanolithography”. Considering the progress in miniaturization of electronic devices, it becomes essential to characterize these materials on a submicronic scale and even, sometimes, on the atomic scale! The growth and the structure of magnetic thin films, for example, can be studied in a very sharp way using synchrotron techniques, so as to improve the storage capacity of magnetic memories. The quality of the silicon wafers used for the manufacturing of chips also needs to be checked with high precision: undesired metallic contaminants present in the wafers in minute quantity (traces) can be detected. This checking step is necessary to improve the manufacturing processes of the new generation chips.

As semiconductor fabrication reaches the limits of photolithography many of the larger players in the industry are increasingly developing their capabilities in x-ray lithography using SL. The US-based Semiconductor Industry Association suggests that x-ray lithography is the likely next step in the development of semiconductor production technology, and estimate that it will be used in commercial production by 2003. IBM and several Japanese companies have already invested in dedicated synchrotron sources. In Australia, Telstra researchers have already begun to experiment with SL methods on overseas facilities.

SL facilities are also used in photonics research and in a wide and increasing range of micromachining applications relating to *information and communication* technologies.

<u>Industrial Demand</u> Much of the commercial work carried out by or for industry at SR laboratories is confidential and contracts usually guarantee such secrecy; this makes it difficult to survey the work comprehensively. However, there is sufficient evidence to indicate that the work is concentrated into what could be classified as 'strategic research' – the investigation of products and processes – rather than fundamental research. Many SR sources now support commercial programmes involving both strategic research investigations, and industries with manufacturing bases as diverse as materials (semi-conductors, ceramics, polymers, etc.), chemicals, petroleum products, health-care products and pharmaceuticals; funding authorities are now reluctant to finance new projects without firm evidence of significant industrial interest. The range of applicability is increasing daily as new facilities are built, existing ones continuously upgraded and industrial development dynamics impel industries to search for ever greater technological developments. The data on the industrial use of the total instrument time of some US, Asian and European facilities are described below.

Laboratory	Industrial Use (percent)
Advanced Photon Source (USA)	23
Advanced Light Source (USA)	25
Stanford Synchrotron Light Source (USA)	30
National Synchrotron Light Source (USA)	15
MAX II (Sweden)	10-15 (planned)
Photon Factory (Japan)	17
Nano-Hana (Japan)	100
ANKA (Germany)	45 (planned)
BESSY II (Germany)	33 (by government decree)

Major international drug companies are located at synchrotron facilities. For example, one of the collaborative access teams at the US Advanced Photon Source (IMCA-CAT) includes Abbott Laboratories, Bayer, Bristol-Myers Squibb, Glaxo Wellcome, Eli Lilly, Merck, Monsanto/Searle, Parke-Davis, Pharmacia and Upjohn, Proctor and Gamble, Schering-Plough, and Smith Kline Beecham. In addition, there are a number of more or less dedicated industrial synchrotron facilities – 7 in Japan, and 1 in Singapore. A number of Japanese multinational electronics giants have there own in-house synchrotron facilities. There are at least 7 corporately owned synchrotron facilities focusing on microelectronics applications.

ESRF Commercial beam time at the European Synchrotron Radiation Facility (Europe's largest purpose-built, dedicated SR source) is about 1% of the total, but if collaboration between industries and academics entering through the peer reviewed route is included, this figures rises to 5%. There are three main fields of industrial research:

- pharmaceuticals, with a dozen companies mainly using the protein crystallography technique;
- materials, 15 companies working in areas such as chemistry, cements, cosmetics, glass, polymers, petroleum, metallurgy, etc.;
- microelectronics, 10 companies performing trace element detection on Si wafers, using the x-ray florescence technique. This last technique is proving so valuable that a joint ESRF/industry initiative is now funding a new experimental station dedicated to these investigations.

DESY Currently, industrial beamtime at HASYLAB at DESY (Hamburg) accounts for 1.5% of total. The Laboratory has long-term (3 year) contracts with 5 commercial companies, 3 of whom are using EXAFS for catalyst research. These companies provide funding for post-doc positions and, in return, obtain rapid access to beam, help and advice with experiments, etc. However, the companies make additional payments for beam-time and any further support they may require.

LURE is the French National Laboratory for SR research. Its direct commercial work accounts for 5% of beam-time but a more accurate indication of industrial use would be 15%, if academic/industry collaborations were included. The statistics provided by LURE relating to the nature of the industries using the facility is as follows: petroleum - 24%, chemicals - 15%, military - 17%, electronics - 13%, pharmaceutical/cosmetics - 11%, metallurgy - 8%, automobile - 6%, small companies - 6%. The industrial 'highlights' include:

- the structure of skin and hair for cosmetics development;
- the study of catalysts for automobile exhaust systems;
- fluorescence detection for environmental studies.

SRS at CCLRC, Daresbury Laboratory is a fifteen year old, medium energy source which supports a large academic programme. The Daresbury SRS has been used for industrial work for well over ten years. During this time the percentage of beam-time directly scheduled for commercial applications has varied but is typically between 2% and 3% of the total time available. However, as with the other sources, it is known that many industrial users obtain access through collaboration with academic. A number of major companies, including ICI, Unilever, Glaxo and Zeneca, have long term contracts, whilst others, including some major pharmaceutical and petrochemical companies, obtain SR and staff effort through shorter term arrangements. The analysis according to industrial sector is the following: general chemicals – 40% use, petrochemicals – 34%, health care – 10%, pharmaceuticals – 9%, LIGA – 4% and others – 3%. LIGA work has also been promoted, with a group of four interested industrial companies forming a 'club' to support the work and obtain micro-structures. These included representatives from the automobile engineering, nuclear materials processing, and micro-electronics sectors. Recently, CCLRC has launched a new initiative, named 'DARTS'. This is intended to make SR more easily available to small companies by offering a sample analysis service with a quoted price per sample and a fast turn-round time. DARTS is proving to be popular with companies which have never used SR in the past and the service has examined samples as diverse as foam rubber, bladder stones, solar cells, paint, deodorants and toothpaste. The DARTS service is expected to continue to expand and could, in the future, represent greater commercial use of SR than the earlier core activity of beam-time use by industry's own experts.

HELIOS, Oxford Instruments, UK is a 'compact' superconducting storage ring, intended for both the development and manufacture of micro-chips by x-ray lithography. The design of the accelerator was carried out through a joint Oxford Instruments/Daresbury Laboratory initiative and Oxford Instruments are now offering the source for sale to laboratories and manufacturers. Helios 1 was made to order for IBM, USA and is currently being used for x-ray lithography research.

It is clear that SR facilities provide a focal point for a wide range of interdisciplinary linkages and learning, and a mechanism for the kinds of interaction that encourage the commercialization of technologies and the capture of inter-industry and intra-industry spillovers. It is also anticipated that the remote operation of experimental stations, simultaneous multi-technique analysis, in-situ measurements, and a greater focus on industrial applications are likely to become a central focus of the future development of synchrotron facilities.

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