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Synchrotron radiation in Brazil. Past, present and future

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ABSTRACT

The first discussions which later led to the creation of the Brazilian National Synchrotron Light Laboratory started in 1981 at the Brazilian Center for Physics Research in Rio de Janeiro. The decisions of the Brazilian National Research Council regarding the foundation of the National Synchrotron Light Laboratory and its location in Campinas, in Sao Paulo State, were taken in 1985. During the second semester of 1986 the activities at this new National Laboratory started from scratch. The construction of a second-generation synchrotron source consisting of a 120 MeV electron Linac and a 1.37 GeV electron storage lasted 10 years. In parallel, eight beam lines were designed and built up. After commissioning, in July 1997, the synchrotron source and the first set of beam lines were opened to users. Later, a 500 MeV booster accelerator and two wigglers were added and, more recently, a first undulator was installed. During the past 20 years several thousand scientists used the different beam lines for applications to many research fields and published more than 3500 indexed articles. During the current decade, a new fourth-generation 3.0 GeV synchrotron source with an extremely low emittance was designed, is under construction, and will be soon commissioned and open to users. The forthcoming availability of this new synchrotron source will allow for experiments with extremely high time, spatial and energy resolutions. Moreover, the coherence properties of the X-ray beams emerging from this source will allow for obtaining lens-free high-resolution images of the structures of inorganic materials and biologic systems.

1. Introduction

After five years of preliminary discussions, from 1981 to 1986, the Brazilian National Synchrotron Light Laboratory (LNLS) was created. From 1987 to 1997 the LNLS staff designed and built a second-generation synchrotron source consisting of a 120 MeV linear electron accelerator and a 1.37 GeV electron storage ring, which is used from 1997 until now by many scientists from Brazil and foreign countries. Moreover, a new source consisting of a fourth-generation 3.0 GeV electron storage ring is now in final phases of construction.

The history of synchrotron radiation in Brazil can be divided in four activity phases, namely (i) preliminary discussions and viability studies, from 1981 to 1986, (ii) design and construction of a second-generation synchrotron source and seven associated beam lines, from 1987 to 1997, (iii) upgrading of the synchrotron source and installation of eleven additional beam lines, from 1998 to 2015, and (iv) design and construction of a new fourth-generation 3.0 GeV synchrotron source, from 2012. The 1.37 GeV synchrotron source was open to Brazilian and foreign users in 1997 and will be shut-down in July 2019, just before the commissioning and first operation of the new 3.0 GeV source.

The purpose of this article is not to report detailed technical information on the currently used synchrotron source nor about the new source under construction. The relevant parameters associated to the technical features of both electron storage rings and associated beam lines are reported in articles to be referenced in next sections. An important parameter to be mentioned in this article is the electron energy from which the practical upper limit of the continuous photon emission spectrum depends. Particularly, the photon emission spectrum of synchrotron sources with increasing electron energies extends toward higher photon energies. Another relevant parameter to be mentioned is the emittance of the electron storage ring which is the product of the size of the light source (i.e. the size of the electron beam cross-section) and the narrow opening angle of the light emission cone. Notice that decreasing emittance induces higher brightness and larger volume of coherence of the emitted photon beams.

2. Preliminary discussions and viability studies (1981–1986)

The first discussions in Brazil that later led to the creation of LNLS started in 1981 in Rio de Janeiro at the Brazilian Center for Theoretical Physics (CBPF), when its director and a small group of scientists exchanged preliminary ideas about the viability of building a synchrotron radiation source in Brazil. At that time, most of the scientists working at CBPF were mainly dedicated to research in theoretical physics, and the

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eventual availability of a synchrotron source was considered as a significant action for enhancing experimental scientific activity at this Center. After preliminary discussions at CBPF the basic ideas for the construction of a synchrotron source in Brazil were spread among the Brazilian scientific community.

In 1982 the president of the Brazilian National Research Council (CNPq) decided to create the Synchrotron Radiation Project and nominated an executive committee and scientific advisory board. The basic ideas developed by the Synchrotron Radiation Project were presented and discussed in different scientific meetings held in Brazil and Argentina. Soon after, many workshops and scientific meetings were organized with participations of Brazilian and foreign scientists. From 1982 to 1984, several scientists and accelerator physicists from LURE-Université Paris-Sud, SSRL-Stanford University, and other synchrotron centers visited CBPF and collaborated with members of the executive committee of the Synchrotron Radiation Project. Soon after, a preliminary proposal for the construction of a synchrotron facility was submitted to the authorities of CNPq.

Since the synchrotron source to be designed and constructed in Brazil was expected to be used by a high number of scientists working in many research fields, the president of CNPq invited the board members of most of the Brazilian scientific societies to attend a meeting for discussing the basic ideas risen by the members of the Executive Committee of the Synchrotron Radiation Project. The recommendation of the scientific societies to CNPq was to perform a viability analysis of the preliminary project for local design and construction of the proposed synchrotron source. Soon after, a working group of Brazilian scientists and engineers spent several months at Stanford University, USA, for practical training. During the stay at Stanford this group completed a first conceptual design of a 2–3 GeV electron storage ring to be used as a synchrotron radiation source. The electron energy of the storage ring was chosen in order to provide users with high fluxes of VUV, soft X-rays and also hard X-ray beams which are usually required for crystallographic studies by X-ray diffraction.

In December 1984, the president of CNPq created the National Synchrotron Radiation Laboratory (LNRS) and nominated an ad-hoc committee for proposing the site where the new Laboratory would be located. This committee organized an open bidding process for hosting the LNRS. Four proposals were submitted by two institutions from Rio de Janeiro State (Rio de Janeiro and Niteroi), and two from Sao Paulo State (Sao Carlos and Campinas). After a comparative analysis of all proposals and in agreement with the ad-hoc committee, the president of CNPq decided that LNRS be located in Campinas.

In 1985, the minister of science and technology of the new national government nominated an ad-hoc committee for performing an independent reanalysis of the project leading to the creation of LNRS, including the decision regarding its geographic location. This committee agreed with the basic ideas and previous decision regarding the creation of the synchrotron facility in Campinas. Soon after, in 1986, the Brazilian National Synchrotron Light Laboratory (named from then LNLS instead of LNRS) was formally created.

3. Construction of a second-generation synchrotron source and associated beam lines (1987–1997)

The activities at LNLS started from scratch by the end of 1986. After additional discussions about technical viability and taking also into consideration funding availability, the LNLS leaders modified the initial project of the synchrotron source and instead of a 2–3 GeV electron storage ring they decided construct a smaller storage ring with lower electron energy. Thus, the Project Department of LNLS started the design and construction of the UVX synchrotron source composed of a 120 MeV linear accelerator and a 1.15 GeV electron storage ring. A further upgrading led to an increase in electron energy of the storage ring from 1.15 GeV up to 1.37 GeV.

From 1987 until 1997, the staff of LNLS designed, built and commissioned the UVX electron storage ring and a first set of beam lines. Most parts of the electron storage ring - including all electromagnets and power supplies - and many critical components of the beam lines - including all X-ray monochromators - were designed and made at LNLS. The electron storage ring and the first set of seven beam lines were commissioned an open to users in July 1997. The UVX synchrotron source exhibits typical features of a second-generation electron storage ring, i. e. rather high emittance (100 nmrad) and photon beams emerging from bending magnets. The UVX electron storage ring installed at LNLS is the first synchrotron source in the southern hemisphere.

During the construction phase of the UVX synchrotron source, an International Review Panel composed of prestigious scientists and accelerator physicists met several times at LNLS. This Panel evaluated the progresses achieved during the design and construction of the electron storage ring and advised the direction of LNLS about the planning for future actions.

In 1989 the first LNLS Users Meeting was held, and from then this event is being organized every year. During the first decade, these meetings were dedicated to (i) provide the scientific community of future users with information related to progresses in LNLS activities, (ii) report relevant applications of synchrotron radiation in other synchrotron centers of the world, and (iii) discuss technical features of the first set of beam lines to be installed. From 1998 until now these meetings mainly focused on reports by LNLS users of the results of their experiments. In addition to these annual meetings, the LNLS organized many topical workshops related to specific applications with participation of prestigious scientists working at different synchrotron facilities in France, Germany, UK, USA, Japan and Italy. The purpose of these events was to inform, motivate and consolidate the community of future users of the LNLS synchrotron source.

The operation and use of the UVX synchrotron source started in July 1997 (Rodrigues et al.,1997, 1998). At this time, the electron beam emerging from a 120 MeV linac was directly injected through a transport line into the electron storage ring in which the electrons were additionally accelerated up to 1.37 GeV by means of a radio-frequency cavity. Because of the rather low electron energy of the UVX storage ring, the critical photon energy of its emission spectrum is also low (E = 2.1 KeV). This implies that the maximum flux over the continuous photon emission spectrum lies within the soft X-ray energy range while the emission flux of harder X-ray photons (E > 2.1 KeV) decreases. For example, the fluxes of photons with energies E = 8.0 KeV ($\lambda \sim 1.5$ Å) and E = 15.0 KeV ($\lambda \sim 0.8$ Å) - typically used in X-ray diffraction experiments - are approximately 30 and 500 times, respectively, lower than the maximum flux of soft X-ray photons with critical energy E = 2.1 KeV ($\lambda \sim 6.0$ Å).

The rather low electron energy of the UVX synchrotron source was considered by crystallographers as a handicap for diffraction experiments, because, as mentioned before, this technique typically requires rather hard X-ray photons, within the energy range from 8 KeV to 15 KeV. Despite this drawback, four beam lines using photons within this energy range were constructed, which are dedicated to X-ray diffraction (XRD1), X-ray absorption (XAFS1), small angle scattering (SAXS1) and protein crystallography (PCr, later named MX1). Another beam line (SXS) was built for soft X-ray spectroscopy studies and other two operate in the VUV energy range (TGM and SGM). As will be reported in the next section, the preference of users for X-ray beam-lines was also evidenced and accentuated during the following twenty years, during which eleven new beam lines were added.

4. Upgrading and use of the UVX synchrotron source (1998–2015)

Along two decades of operation, from 1998 to 2018, the UVX synchrotron source was several times upgraded. In 2001 a 500 MeV booster electron accelerator was placed between the 120 MeV linear accelerator and the storage ring (Tavares et al., 2001). The booster accelerates the electrons from 120 MeV up to 500 MeV and then injects them to the

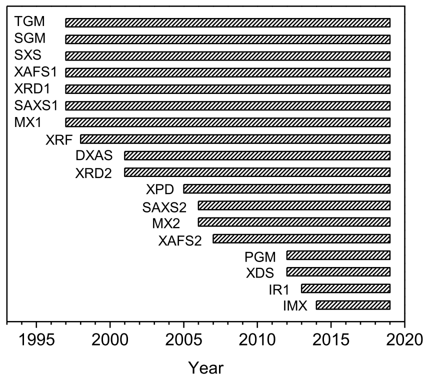

Fig. 1. Horizontal bars display the periods of operation of the 18 beam lines of the UVX synchrotron source. TGM (Toroidal Grating Monochromator), SGM (Spherical Grating Monochromator), SXS (Soft X-ray Spectroscopy), XAFS1 and XAFS2 (X-ray Absorption Fine Structure), XRD1 and XRD2 (X-ray Diffraction), SAXS1 and SAXS2 (Small-Angle X-ray Scattering), MX1 and MX2 (Protein Crystallography), XRF (X-ray Fluorecence), DXAS (Dispersive X-ray Absorption Spectroscopy), XPD (X-ray Powder Diffraction), XDS (X-ray Diffraction and Spectrocopy), PGM (Polarizing Grating Monochromator), IMX1 (X-ray Imaging) and IR1 (Infrared Spectroscopy).

main electron storage ring. After this upgrading several insertion devices were installed, namely a normal magnet multipole wiggler, a superconducting wiggler and an elliptically polarized undulator. The radiation beams emerging from both wigglers exhibit higher flux of hard X-rays while the undulator produces polarized VUV beams much brighter than those emerging from bending magnets.

During the period from 1997 to 2014, the number of beam lines in operation and open to users increased from seven to eighteen. The sequence of the additions and periods of operation of all beam lines are shown in Fig. 1. It can be noticed in this figure that thirteen beam lines use hard X-rays (5 KeV $<$ E $<$ 20 KeV), while five of them provide soft X-rays, VUV or IR radiation beams.

From 1997 until now, several thousand scientists from Brazil and from foreign countries have conducted research projects at LNLS for applications to many research fields such as materials science, physics, chemistry, biology, environmental sciences, and others. LNLS operates as a National Laboratory which opens its beam lines to interested users after evaluation and approval of submitted scientific proposals. The use of the LNLS beam lines for performing scientific projects is free. Furthermore, users from Brazil and other Latin-American countries receive total or partial financial support for their travel and lodging expenses during the period of their experiments at LNLS. During the last year (2018), 1680 scientific users performed experiments at LNLS, 1403 of them from Brazil, 182 from other Latin-American countries and 96 from the rest of the world.

From 1998 until 2018, the users of the UVX source published more than 3500 indexed articles related to experimental work performed at LNLS. The total numbers of published articles corresponding to every beam line are displayed in Fig. 2. In this Figure the beam lines are grouped in order to visualize and compare the numbers of articles associated to different types of experimental techniques: (i) VUV and IR spectroscopies, (ii) X-ray spectroscopies, (iii) X-ray diffraction, (iv) small-angle X-ray scattering and (v) X-ray imaging.

The historical evolution of the total number of articles published annually containing experimental results obtained at LNLS is displayed in Fig. 3. This figure evidences an overall trend characterized by a continuous increase in number of published articles and a progressively decreasing growth rate. During recent past years, the users of the UVX synchrotron source are annually publishing 270 articles circa. Another analysis not shown here indicates an also significant growth in the average impact factor of the journals in which the articles were published.

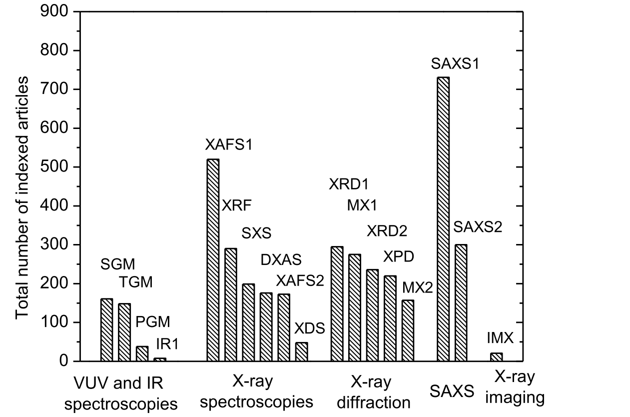

Fig. 2. Vertical bars indicate the total number of indexed articles based on experimental results obtained using the different beam lines from 1998 until 2018. The beam lines were grouped in order to visualize the relative production in number of articles associated to different types of experimental techniques.

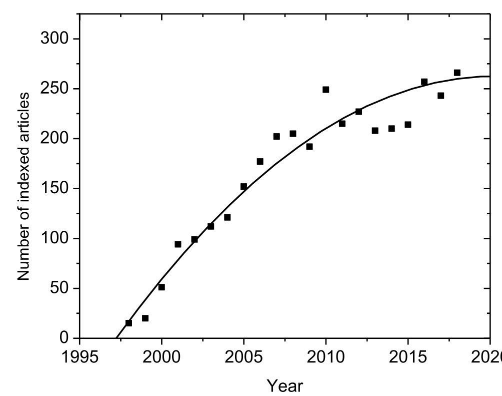

Fig. 3. Annual number of indexed articles associated to experimental results obtained at LNLS (LNLS records). The solid line is a guide for the eye.

An example of the relevant contribution of LNLS to the development of research fields in Brazil and Latin-America is related to the quantitative scientific output associated to the SAXS beam lines. The LNLS records regarding the scientific production associated to its beam lines indicate that the annual number of published articles related to results obtained by using the SAXS beam lines increased from a few articles in 1998 up to about 80 in recent years, most of them being authored by Brazilian and Argentinian users. By an independently data mining procedure, the total numbers of scientific articles related to SAXS published by authors from Brazil and Argentina were also determined (Fischer and Craievich, 2018). By comparing both determinations, it was established that the annual numbers of scientific articles related to the SAXS technique based on experimental results obtained at LNLS are essentially the same as the total annual number of articles related to SAXS published by Brazilian and Argentinian scientists. This implies that the strong growth of research fields related to SAXS applications in these countries is essentially due to the open availability of the SAXS beam lines installed at LNLS. Another relevant finding of our search refers to the annual numbers of articles related to the SAXS beam line currently published by Brazilian authors and their comparison with those published by authors from other countries of the world. While in 1998 the total annual number of articles related to SAXS published by Brazilian scientists was very low, this number strongly increased and is now essentially the same as the number of articles published by authors from France and Japan, and only lower than the number of those published by authors from China, USA and Germany.

Because of the low electron energy - and thus rather low photon flux in the hard X-ray range - and high emittance of the UVX electron storage ring, it was soon apparent that this source exhibits serious limitations for applications to studies that require high spatial, temporal and/or energy resolutions. In addition, because of the rather large size of the electron beam, another drawback was the lack of coherence of the emitted beams. Comments about the limitations associated to the use of incoherent instead of coherent or partially coherent beams for X-ray diffraction and scattering experiments are included in Appendix A.

5. New four generation synchrotron source

During the last two decades, new synchrotron radiation sources with novel characteristics were proposed. These new sources are designed for providing very bright photon beams from the IR up to the hard X-ray range, with high degree of coherence and/or very short photon pulses. New photon sources that started to be constructed during the last two decades are fourth generation synchrotron sources and X-ray free electron lasers (XFELS). New possibilities open by the availability of new advanced synchrotron sources and some applications are described in Appendix B.

After open discussions with users, the LNLS staff started in 2009 the design and in 2012 the construction of a new fourth-generation 3.0 GeV synchrotron source named Sirius (Liu et al., 2013), which will soon replace the current 1.37 GeV UVX source. Because of the higher electron energy of Sirius, the emission spectrum of photons emerging from its bending magnets will extend toward the high energy range. On the other hand, the undulators to be installed will deliver VUV and also X-ray photon beams. Another relevant feature of Sirius will be its extremely low emittance, namely ~0.2 nmrad, i. e. about 500 times lower than the emittance of UVX. These features of Sirius together with those of the new beam lines to be installed will allow for performing many novel experiments which are not now possible using the UVX synchrotron source.

Since the electron energy of Sirius is much higher than that of UVX, the undulators to be installed will produce very bright beams also in the X-ray range. In addition, because of the extremely low emittance of the source (~0.2 nmrad instead of 100 nmrad of the UVX source), the beams emerging from undulators will be highly coherent. These features will make it possible different applications of coherence properties of the X-ray beams to diffraction analyses of varying structures with high spatial and time resolutions.

As reported in Table 1, the first set of six beam lines to be installed at Sirius during its first operation phase will be dedicated to (i) X-ray nanoscopy, (ii) X-ray spectroscopy and diffraction under extreme conditions, (iii) coherent and time-resolved X-ray scattering, (iv) high resolution UV and soft X-ray spectroscopies, (v) infra-red micro and nano-spectroscopies and (vi) macromolecular micro and nano-crystallography (www.cnpem.lnls.br). These beam lines with highly coherent radiation beams will be equipped with new advanced instrumentation for novel applications associated to the use of coherent X-ray scattering and diffraction techniques, fast time resolved measurements and probing of very small sample volumes. Other beam lines which do not necessarily need the use of highly coherent radiation beams will be installed later. These beam lines will be dedicated to powder diffraction, small-angle X-ray scattering, X-ray spectroscopy, high-energy X-ray tomography, inelastic X-ray scattering, X-ray micro and nano tomography, and soft X-ray absorption spectroscopy and imaging.

It should be noticed that the efficient use of the different techniques associated to novel applications of fourth-generation synchrotron sources requires challenging developments of new instruments such as extremely stable optical components, complex control systems and very fast detectors with high spatial resolution and dynamical range. Progresses in all these technical issues are being achieved at LNLS, so as the forthcoming opening of Sirius is expected to bring soon new exciting research opportunities to users from Brazilian and foreign scientific communities.

An example of a novel instrument for Sirius is a high dynamics double-crystal monochromator recently developed by LNLS researchers in collaboration with scientists from MI Partners, Netherlands (Geraldes et al., 2016). This instrument is based on a new concept and achieve the crystal parallelism to be kept within +- 20 nrad, which is at least one order of magnitude better than the best existing monochromators, and this performance is maintained even for scanning speeds as high as 1000 eV/sec.

Certainly, the forthcoming availability of Sirius will open new research fields and new opportunities to users. However, for achieving an efficient utilization of this fourth-generation photon source, users should be clearly aware of recently developed analysis tools, such as those involving theoretical aspects related to coherence properties of X-ray beams, new methods for data analysis and modern procedures for big-data processing.

Final comments and acknowledgements

The author of this article participated in different activities along the whole historical evolution of synchrotron radiation in Brazil. He was coordinator of the Executive Committee of the Synchrotron Radiation Project during the early times at CBPF (1982–1985), adjoint director of LNLS during the construction of the UVX synchrotron source (1986–1997), and frequent user of LNLS beam lines from 1998 until now. His scientific production along the last twenty years was almost exclusively based on experimental results obtained at LNLS.

The author thanks the current directors and leaders of LNLS - R. Rodrigues, Y. Petroff, H. Westfahl, L. Lin and H. Tolentino - for providing information on different features of Sirius, and about the first set

Table 1
First set of six beam lines to be installed at Sirius (www.cnpem.lnls.br).

Beam line	Application	Main features
Carnaúba	X-ray nanoscopy	X-ray absorption, scattering and emission using coherent radiation and nanofocussing (beam size down to 30 nm)
Ema	X-ray spectroscopy and diffraction under extreme conditions	High brilliance for time-resolved experiments, and small beam size (down to 0.1 × 0.1mm2) for experiments at extremely high pressure
Catereté	Coherent and time-resolved X-ray scattering	Coherent X-ray scattering and diffraction imaging (CXD) for structural studies at the nanoscale and photon correlation spectroscopy (XPCS) for investigations of the dynamics of biological molecules
Ipé	High resolution UV and soft X-ray spectroscopies	Inelastic and photoelectron spectroscopies. Ambient pressure X-ray photoelectron spectroscopy (AP-XPS) and resonant inelastic X-ray scattering (RIXS)
Imbuia	Infra-red micro and nano-spectroscopies	Infrared spectroscopy studies in the medium IR range. Composition analyses of materials and other chemical applications
Manacá	Macromolecular micro and nano-crystallography	X-ray diffraction by biological macromolecules (proteins, viruses and complexes) using micrometric and submicrometric beam sizes.

of beam lines and their applications. The author also acknowledges a research fellowship and associated grant assigned by the Brazilian National Research Council (CNPq).

Appendix A. Limitations of classical investigations of materials structure

Structures of crystalline materials currently determined by classical crystallographic methods are time and spatial averages over atomic configurations inside all unit cells. On the other hand, all crystalline materials - with essentially periodic atomic structure - exhibit many types of defects in their volume and structural disorders close to their external surface. Furthermore, the properties of many materials depend more on the detailed features of their defects than on their average structure. For this reason, the knowledge of time and spatial averages of instantaneous and local structures - determined by classical X-ray diffraction procedures - may not be sufficient for explaining a number of properties of materials.

The most frequently used experimental procedure for studying the structure of materials is X-ray diffraction. Analyses of X-ray diffraction patterns reveal the geometry of unit cells and determine the average coordinates of the atoms inside them. The problem is that atomic structures determined by applying this technique are time and spatial averages of many instantaneous and local structures, respectively. For this reason, the results derived from classical X-ray diffraction experiments do not describe neither instantaneous configurations of moving atoms nor detailed local structures of point, linear, surface and volume defects. This restrains our understanding of many properties of materials that often depend more on their instantaneous structure and/or local configurations of structural defects than on the features of their time or spatially averaged structures. Characterizations of static and dynamical defects such as those associated to vacancies, impurities, quantum dots, stacking faults and others are performed by using X-ray diffuse scattering techniques which analyze the weak diffuse intensity between Bragg peaks.

The problem related to the average nature of structural information derived from both, classical and diffuse scattering measurements, is even more relevant for nanomaterials. The volume fraction of the disordered structure near the external surface of nanocrystalline materials depends on nanocrystal sizes and shapes. For this reason, the melting and crystallization temperatures and other properties of nanomaterials also depend on crystal sizes and shapes. This example clearly shows that the exclusive knowledge of the average atomic structure of the unit cell derived from classical crystallographic procedures is not enough for explaining the properties of nanomaterials.

Challenges associated to the need of high space and time resolved experiments are now expected to be faced by using recently developed X-ray photon sources, namely fourth-generation electron storage rings and X-ray free-electron lasers (XFELs).

Appendix B. Fourth generation synchrotron sources and X-ray lasers. Basic features and some applications

The characteristics of synchrotron sources consisting of high energy electron storage rings have been changing along the history. These sources are often classified as belonging to different generations. *First generation* synchrotron sources are electron storage rings initially designed for studying collisions of high energy electrons and later used as a synchrotron source in parasitic mode. *Second generation* sources are electron storage rings specially designed for production of photons emitted by bending magnets. In *third generation* sources, photon beams are emitted by special devices, named undulators, inserted in the straight sections of storage rings. Modern *fourth generation* X-ray sources are electron storage rings with extremely low emittance which provide very bright X-ray beams with large coherence volumes.

The properties of coherent hard X-ray beams and their use in advanced structural analyses were detailed in a tutorial article by van der Veen and Pfeiffer (2004). The longitudinal coherence length is related to the monochromaticity of the X-ray beam while the transversal coherence length increases for decreasing size of the photon source. For these reasons, the availability of very bright fourth-generation synchrotron sources with extremely low emittance and other modern sources such as X-ray free electron lasers (XFEL) will open new opportunities for structural studies with high spatial and time resolutions of all kind of crystalline and amorphous materials (Weckert, 2015)].

The first fourth generation synchrotron source (MAX-IV) was built and is operating since 2016 in Lund, Sweden, while other two - Sirius at LNLS in Campinas, Brazil, and EBS (Extremely Brilliant Source) at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France - are currently under construction.

In order to determine the detailed structure of imperfect crystals without spatial averaging, the whole crystal should be considered as being the "unit shell". This type of structural analysis requires an X-ray beam with a volume of coherence larger than the crystal volume. This method is currently starting to be applied to small (nano) crystals by using third and fourth-generation synchrotron sources. The use of highly coherent X-ray beams allows for the analysis of X-ray scattering intensity over all accessible reciprocal space, and, through Fourier synthesis, the whole structure of the material is determined. In other words, this procedure yields "lens-free images" of the structures of perfect and imperfect crystals, and even of disordered and amorphous materials. An example of a real-time study of a fast varying structure is a recent time-resolved investigation - at the millisecond scale - of the propagation of cracks in silicon wafers (Rack et al., 2016).

Notice that lens-free X-ray imaging techniques currently are far from reaching atomic resolution. However, lens-free images with spatial resolutions at nanometric scale (~ 2 nm) were already obtained while a progressively higher spatial resolution (~ 2 Å) is expected to be achieved by increasing the mechanical stability of beam line components and improving the quality of photon detectors (Robinson, 2019).

In order to characterize materials with fast varying structure, other new X-ray sources named X-ray free electron laser (XFEL) are used. XFELs consist of a linear accelerator followed by a long undulator which provides incident X-ray beams composed of very short (~ 10 femtoseconds) bunches, each of them containing an extremely high number of photons. XFELs are currently in operation in USA (LCLS), Japan (SACLA) and Germany (European XFEL).

Problems risen in classical experiments by long time averaging are starting to be solved by using XFELs. For example, a structural study of metallic nanocrystals using a single ten-femtosecond XFEL bunch was achieved (Xu et al., 2014). Moreover, an X-ray diffraction study of protein nanocrystals performed at LCLS demonstrated that a single photon bunch fully destroys the nanocrystals (Chapman et al., 2011). Despite this drawback, since the photon-electron interaction is extremely fast, diffraction patterns associated to the initial structure of protein nanocrystals - before they be radiation damaged - are recorded. Since in the mentioned investigation a single photon bunch was not enough for obtaining a diffraction pattern with good statistical quality, many ($\sim 10^4$) protein nanocrystals were successively probed. This new experimental procedure is named serial crystallography. The results obtained demonstrated that the use of XFELs solves the old problem of radiation damage usually occurring in classical experiments of X-ray diffraction studies of protein crystals.

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