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SCHOOL ON THE USE OF SYNCHROTRON RADIATION IN SCIENCE AND TECHNOLOGY:
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The Brazilian Synchrotron Radiation Source: Commissioning and First Experiments

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LNLS synchrotron source and beamlines: status, first experiments and user access

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Abstract

The synchrotron radiation source constructed at LNLS is composed of a 1.37 GeV electron storage ring and a 120 MeV LINAC injector. Seven beamlines are in the final phase of commissioning and will be opened to external users during the second semester of 1997. These beamlines allow for experiments of VUV and X-ray spectroscopies and X-ray diffraction and scattering. A number of experimental results were already obtained using the beamlines which are currently in operation. X-ray absorption spectroscopy and small-angle X-ray scattering results, associated with a metal (pure Ni) and a biological sample (collagen), respectively, are described. The perspectives and user access policy for the utilization of the LNLS synchrotron radiation source are reported.

1. Introduction

Synchrotron radiation is produced by high-energy electrons circulating inside the vacuum chamber of *storage rings*. The electron energy in storage rings dedicated to the production of synchrotron radiation ranges from about 400 MeV up to 8 GeV. The radiation is generated in the parts of the orbit in which the electrons are accelerated centripetally by the action of dipolar magnets. The photons extracted from the electron storage ring are monochromatized and focused by the optical components of the *beamlines* and finally reach the *workstations* where are used by scientists and technologists for a number of applications. Synchrotron radiation i) has a continuous spectrum from infrared to X-rays, ii) is extremely intense if compared with classical VUV and X-ray sources, iii) is linearly polarized in the orbit plane, and iv) is naturally collimated, having a low vertical divergence angle (1). These features make synchrotron radiation very useful in a number of applications for the characterization of atomic, electronic and magnetic structures of inorganic and biological materials(2).

The availability of synchrotron radiation sources allows for experiments which are impossible using classical sources, such as those involving high energy, angle, space and/or time resolution (high-resolution band electron energy structure, mapping of structure in heterogeneous materials, chemical reactions and phase transformation in real time, etc). These singular possibilities opened for material science and biological research caused the scientific and technological communities of industrialized countries, and also of some developing countries, to become interested in the use of synchrotron radiation and led them to request the construction of electron storage rings dedicated to the production of synchrotron radiation.

2.The Brazilian National Synchrotron Light Laboratory (LNLS) source

The Brazilian scientific community started the discussions about the interest and technical viability for the construction of a synchrotron light source in 1981. The debates with the participation of scientists from many fields and most of the Brazilian scientific associations took 6 years. The Ministery of Science and Technology decided to found the National Synchrotron Light Laboratory (LNLS) in Campinas, São Paulo State, by the end of 1986. LNLS is one of the research institutes of the National Foundation for Scientific and Technological Development (CNPq).

LNLS is currently installed in a 400,000 m^2 site donated by the São Paulo State Government, on which four main buildings were already constructed and another is under way. The LNLS currently has 140 staff members (about 80 are engineers and technicians and 20 are scientists).

The synchrotron source is composed of a 1.37 GeV electron storage ring, the electrons being injected at low energy (120 MeV) by a linear accelerator (LINAC). Originally the storage ring was designed for 1.15 GeV/100 mA with some allowance in the power supplies and vacuum chambers to deal with higher synchrotron light power. However, the care taken in the design and construction of the magnets proved sufficient to increase the energy to 1.37 GeV, which means twice as much synchrotron light power and 30 times more flux at 10 keV. The energy of the electrons is ramped up to the final energy inside the storage. LNLS designed and constructed the synchrotron source which is presently in the final steps of commissioning. Most of the components were constructed at LNLS, including all the magnets and power supplies.

The picture in Fig.1 shows a general view of the LNLS electron storage ring. The LINAC injector is not visible because it is located underground. Around the storage ring there are now seven beamlines and workstations, some of them already in operation.

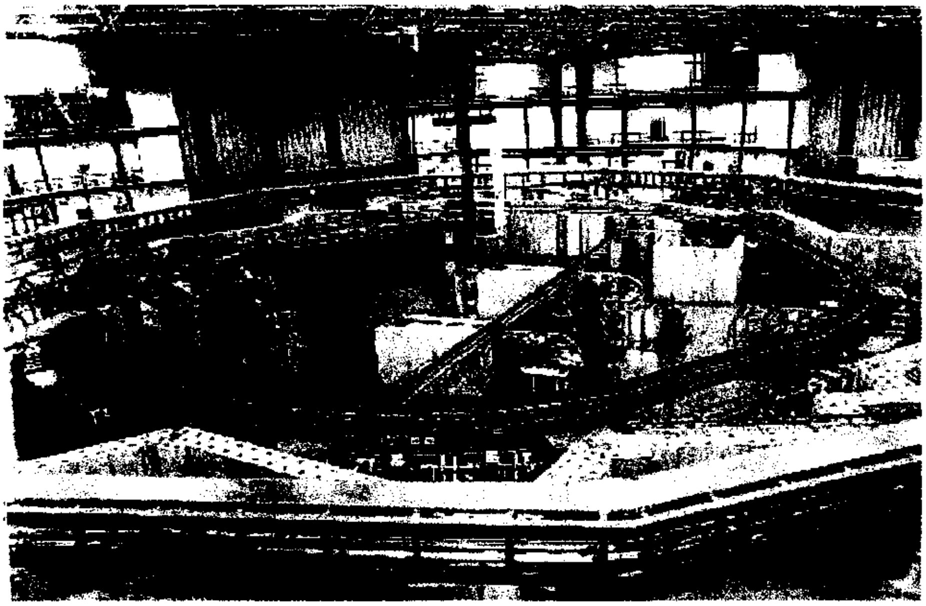

Figure 1: The LNLS 1.37 GeV electron storage in December 7, 1996 (3). All twelve dipolar magnets are visible. Inside the ring two klystrons and their associated modulators are apparent; they feed the LINAC located underground with RF.

Three modes of operation of the electron storage ring are possible. They are the high, normal and low emittance modes. The main design parameters of the LNLS synchrotron source for the normal mode of operation are listed in Table 1. Presently the synchrotron source is operating routinely and is used by the LNLS technical and scientific staff for commissioning and first experiments.

Table 1: Main design parameters of the LNLS electron storage ring

Operation energy	1.37	GeV
Injector:	linear accelerator	
Injection energy	120	MeV
Nominal electron current	100	mA
Circumference	93.21	m
Mean diameter	30	m
Magnetic structure	double bend achromat	
Lattice symmetry	6-fold.	
Revolution frequency.	3.2	MHz
Harmonic number.	148	
RF frequency.	476	Mhz
Natural emittance	70	nm.rad.
Horizontal betatron tune	5.27	
Vertical betatron tune	2.17	
Synchrotron tune (at 500 kV rf gap voltage)	9.19×10^{-3}	
Natural horizontal chromaticity	-7.8	
Natural vertical chromaticity	-9.5	
Momentum compaction factor	8.3 x 10-3	
Natural energy spread	6.0 x 10-4	
Horizontal betatron damping time	13	ms
Vertical betatron damping time	13	ms
Synchrotron damping time	6	ms
Dipole bending radius	2.735	m
Bending field	1.67	T
Number of dipoles	12	
Number of straight sections for insertion devices	4	
Length available for insertion devices	2.95	m
Energy loss per turn from bending magnets	114	keV
Total radiated power from bending magnets	11.4	kW
Critical photon energy from bending magnets	2.08	keV
Electron lifetime	10	hours

At high energy, the maximum value of the initial electron current inside the storage ring obtained up to now is 70 mA and the average electron lifetime is about 3 hours. The electron lifetime is progressively increasing as commissioning goes on. The design lifetime (10 hours) is expected to be reached by August 1997. The initial electron current at high energy increased from some mA in October 1996 up to about 70 mA in March 1997. A typical time dependence of the electron current at 1.37 GeV is plotted in Fig. 2. In order to attain, and eventually exceed, the nominal current, an upgrading of the electron LINAC is planned:

modifications to increase the injection energy to 170 MeV will be carried out during a shutdown programmed for November-December 1997.

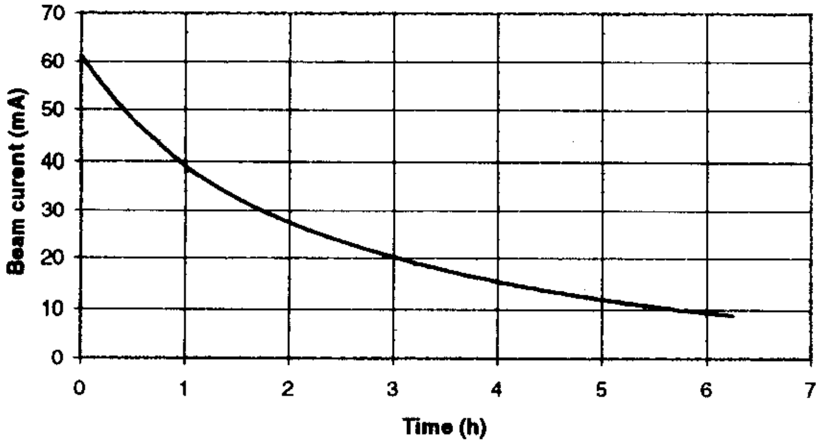

Figure 2: Typical time dependence of electron current in the LNLS storage ring (April 1997).

Fig. 3 exhibits the photon flux produced by the bending magnet sources. The useful photon energy range (i.e. the range for which the photon flux is much higher than that produced by classical sources as, for example, a rotating anode source) extends up to E_{max}= 15 KeV. In order to increase the photon energy range for users interested in applications of high energy photons (E_{ph}>10 keV), the insertion of superconducting wigglers in the ring's straight sections is planned. In Fig. 3, the photon flux expected from a planned 1/2-1-1/2 wiggler (or wavelength-shifter), with a magnetic field of 7 Tesla,is also plotted.

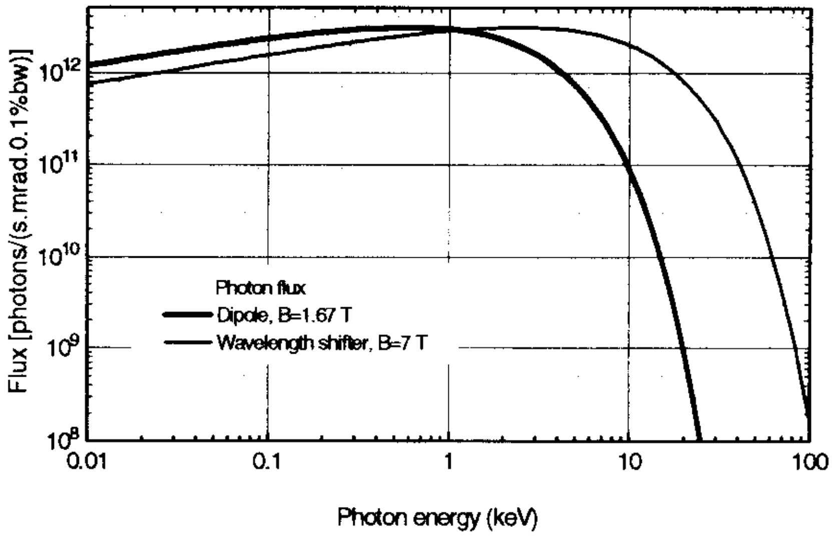

Figure 3: Photon flux from the bending magnets of the storage ring and from the planned 7 Tesla wave-shifter.

3. LNLS beamlines and workstations

Parallel to the construction of the synchrotron light source, LNLS designed and constructed seven synchrotron light beamlines corresponding to different experimental techniques and applications: 1) Toroidal Grating Monochromator (TGM), 2) Spherical Grating Monochromator (SGM), 3) Soft X-Ray Spectroscopy (SXS), 4) X-Ray Absorption Fine Structure (XAFS), 5) X-ray Diffraction (XRD), 6) Small-Angle X-ray Scattering (SAXS) and 7) Protein Crystallography (PCr).

Two additional beamlines for X-ray Fluorescence (XRF) studies and Microfabrication (MF) are currently under construction and will be open to users in 1998.

The main characteristics and applications of the first seven beamlines, which are now under commissioning or in final phase of construction, are listed below(4).

3.1 Toroidal Grating Monochromator (TGM)(5).

Startup: July 1997

Coordinator: A. Rubens Brito de Castro, e-mail: tgm@lnls.br

Applications: Surface physics, surface chemistry, molecular spectroscopy, electronic structure of condensed matter, mass spectroscopy. *Source*: Bending magnet D05 (4°), s_y=0.14 mm. *Monochromator:* Three toroidal gratings. *Energy range*: 300 - 100 eV (40-120 Å), 100 - 35 eV (120-360 Å), 35 - 12 eV (360-1,000 Å). *Spectral resolution:* Better than 0.1 Å (40-120 Å), better than 0.3 Å (120-360 Å), better than 1.1 Å (360-1,000 Å). *Dispersion:* Better than 30.2 Å/degree. *Focusing element:* Two toroidal mirrors. *Flux on sample:* 10^{13} photons/sec (1.3 GeV @100 mA). *Spot size at sample:* 2 x 5 mm^2. *Detectors:* Electron energy analyser, fluorescence detector, electron time of flight.

3.2 Spherical Grating Monochromator (SGM).

Startup: September 1997

Coordinator: A. Rubens Brito de Castro, e-mail: sgm@lnls.br

Applications: High-resolution soft-X-ray spectroscopy, electronic and magnetic properties of materials. *Source:* Bending magnet D08 (4°), s_y=0.10 mm. *Monochromator:* Two spherical gratings. *Focusing elements*: Two spherical mirrors and one toroidal mirror. *Energy range:* 250 - 1000 eV (250 - 1500 at reduced flux and resolution). *Spectral resolution:* Better than 3,000. *Spot size at sample:* 0.5 x 0.5 mm^2. *Detectors:* Electron energy analyser, channeltrons, microchannel plates.

3.3 Soft X-ray Spectroscopy (SXS).

Startup: July 1997

Coordinator: Miguel Abbate, e-mail: sxs@lnls.br

Applications: Soft-X-ray spectroscopy of transition metals and rare earth systems (thin films, multilayers, alloys and compounds), core level spectroscopy. *Source:* Bending magnet D04 (4°), s_y=0.10 mm. *Monochromator* (6): Double-crystal with constant offset, beryl: 790-1,550 eV, quartz: 1,480-1,800 eV, InSb: 1,680-2,000 eV, Si: 2,050- 4,000 eV. *Energy resolution:* 0.2 eV @800 eV ($E/\Delta E$ =4,000). *Focusing element:* Toroidal mirror (gold-coated zerodur mirror). *Detectors:* Total electron yield, electron energy analyser, photodiode array.

3.4 X-ray Absorption Fine Structure (XAFS).

Startup: July 1997

Coordinator: Hélio Tolentino, e-mail: xafs@lnls.br

Applications: Local atomic structure of disordered materials (glass, multilayers, composites, catalysers, etc), electronic and magnetic structure of solids. *Source:* Bending magnet D04 (15°), s_y=0.12 mm, spot size: 550 x 700 mic.m^2, flux on sample: 2 x 109 photons/s.mrad @

8keV. *Monochromator:* Double-crystal (6) and four-crystal (7) with constant offset; *energy range*: Si(111) (2d=6.271 Å): 2.010 - 11.390 keV, Si(220) (2d= 3.84 Å): 3.300 - 1.850 keV, Ge(111) (2d=6.53 Å): 1.920-10.930 keV; *resolving power* (E/ΔE): 10,000-100,000. *Focusing system:* Cylindrical bent mirror (gold or rhodium-coated) sagittal focusing on crystal. *Detectors:* Ion chambers, fluorescence and electron detectors.

3.5 X-ray Diffraction (XRD).

Startup: July 1997

Coordinator: Cesar Cusatis, e-mail: xrd@lnls.br

Applications: Multiple-axis goniometry: rocking curves, standing waves, back-diffraction, topography, multiple diffraction, grazing incidence. θ and 2θ goniometry: Debye-Scherer powder diffraction, texture, structural characterization of epitaxial layers and nanostructures. Single- and multiple-crystal optical studies and devices. *Source:* Bending magnet D12 (4°), s_y=0.10 mm. *Monochromator:* Double- (2C)(6) and four- (4C)(7) crystal with constant offset; energy range: Si(111) (2d=6.271 Å): 2.010 - 11.390 keV, Si(220) (2d=3.84 Å): 3.300 - 18.500 keV, Ge(111) (2d=6.53 Å): 1.920 - 10.930 keV. *Energy resolution:* 4 eV @ 8000 eV (E/ΔE =2000). *Focusing element:* Sagittal focusing (10 mrad) by elastic bending of the second crystal (2C). *Detectors:* Fast scintillation detector, Si-Li solid state and linear position-sensitive detector, ionization chamber, fluorescence detector, imaging plate.

3.6 Small-Angle X-ray Scattering (SAXS) (8).

Startup: July 1997

Coordinator: Iris Torriani, e-mail: saxs@lnls.br

Applications: Heterogeneous materials, characterization of fractal structures, microporous materials, microphase separation, glass-semiconductor nanocrystals, gels, proteins in solution. *Source:* Bending magnet D11 (4°), s_y=0.14 mm. *Monochromator:* One horizontally bent and asymmetrically cut silicon crystal, reflection (111), asymmetry angle: 11 degrees, condensing mode; energy range: 6 -12 keV (1 - 2 Å). *Energy resolution:* (E/ΔE): 1,000. *Focusing element:* Cylindrical elastically bent mirror (vertical focusing), bent silicon crystal (horizontal focusing). *Detector:* One-dimensional position-sensitive gas detector, two-dimensional position-sensitive gas detector, imaging plate, scintillation monitors.

3.7 Protein Crystallography (PCr).

Startup: September 1997

Coordinator: Igor Polikarpov, e-mail: pcr@lnls.br

Applications: Monochromatic diffraction data collection of native and derivative proteins (MIR/SIR/MIRAS/SIRAS). 3D structure determination of biological macromolecules. *Source:* Bending magnet D3 (15°), s_y=0.12 mm. *Monochromator:* Single horizontally bent and asymmetrically cut silicon crystal, reflection (111), asymmetry angle: 7.25 degrees, condensing mode; energy range: 6 keV-12 keV (1 - 2 Å). *Energy resolution:* (E/ΔE): 3,000. *Focusing elements:* Cylindrical elastically bent mirror (vertical focusing), bent silicon crystal monochromator (horizontal focusing). *Detector:* Imaging plate

The locations around the storage ring of the first seven beamlines described above are indicated, together with those of the two beamlines to be completed in 1998, in Fig. 4. All beamlines were designed and constructed at LNLS. Some of the optical components developed at LNLS, such as two-crystal and four-crystal monochromators, use original and patented mechanical concepts (9,10). A picture of one of the beamlines (SAXS), which is now in preliminary operation, is shown in Fig. 5.

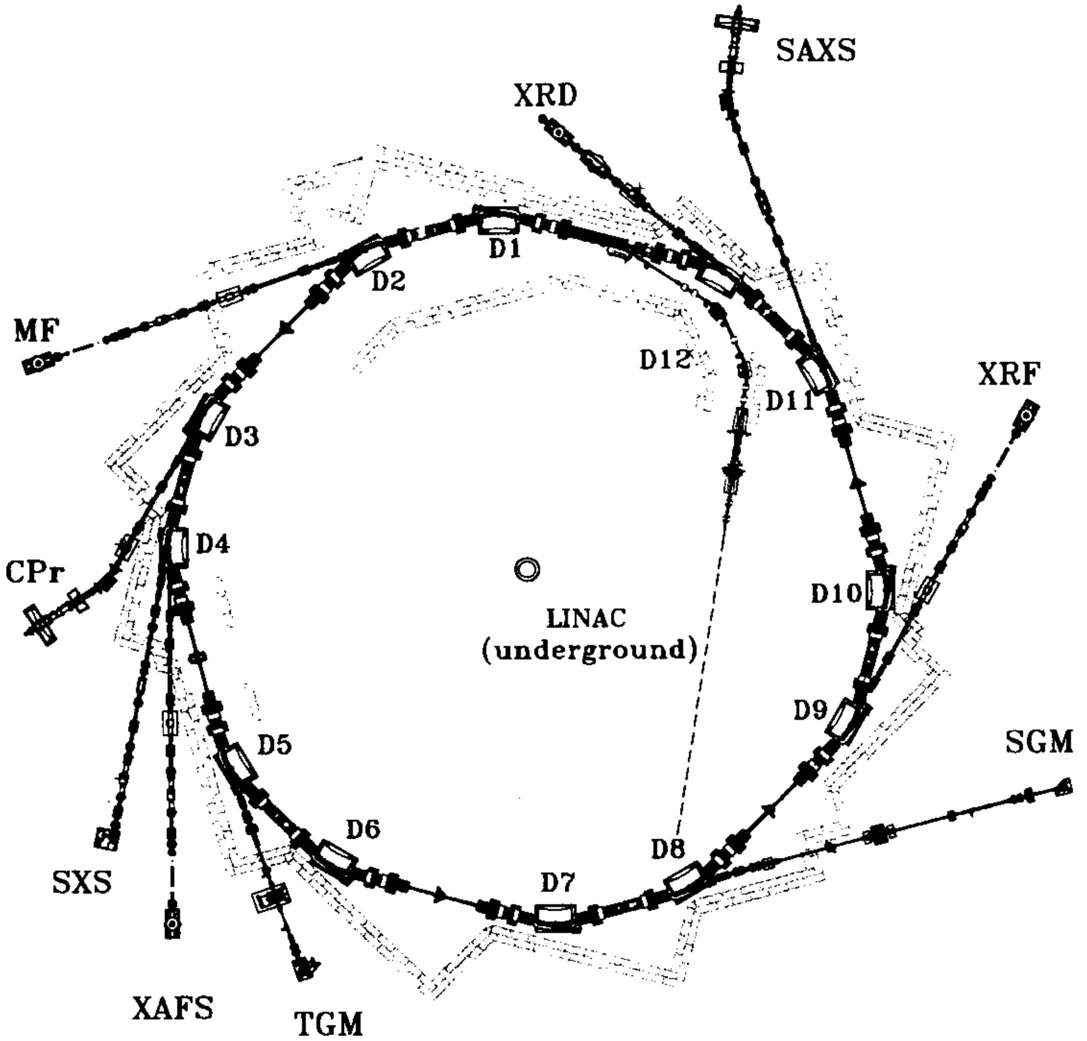

Figure 4: Location of the nine beamlines to be opened to users in 1997 (seven) and 1998 (two).

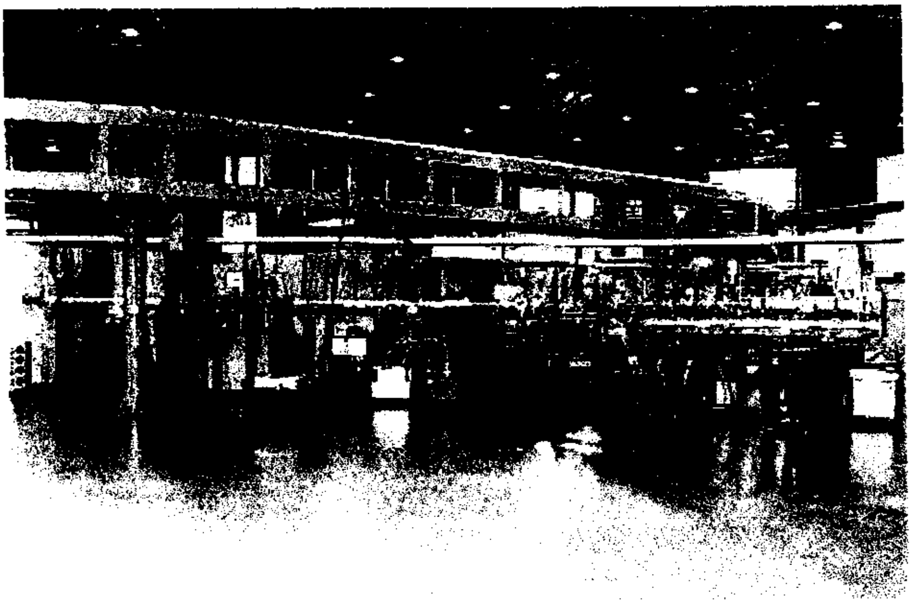

Figure 5: Picture of the SAXS beamline(3). The beamline passes across the shielding at the left. The sequence of optical components is: mirror chamber (for vertical focusing), first four-slit set, monochromator chamber (for monochromatization and horizontal focusing), second four-slit set, guard slit set, sample holder, beamstopper and vertical X-ray position sensitive detector.

4. Preliminary experimental results using LNLS beamlines

Four beamlines are presently (April 1997) ready for operation. The first experiments were performed in these beamlines by users at the end of October 1996, some days before the VII Workshop of LNLS users was held.

As an example of experimental results, an EXAFS spectrum of pure nickel is illustrated in Fig. 6. This absorption spectrum was obtained in the XAFS beamline using two ion chambers to record the direct and transmitted beam intensities as a function of the photon energy. The white beam was monochromatized by using the (111) Bragg reflection from a two-crystal silicon monochromator. The absorption coefficient of Ni was determined in an energy range of 500 eV, close to the K absorption edge. Atomic structure information is obtained by mathematical treatment of the "EXAFS oscillations" which are apparent in Fig. 6 above the K absorption edge.

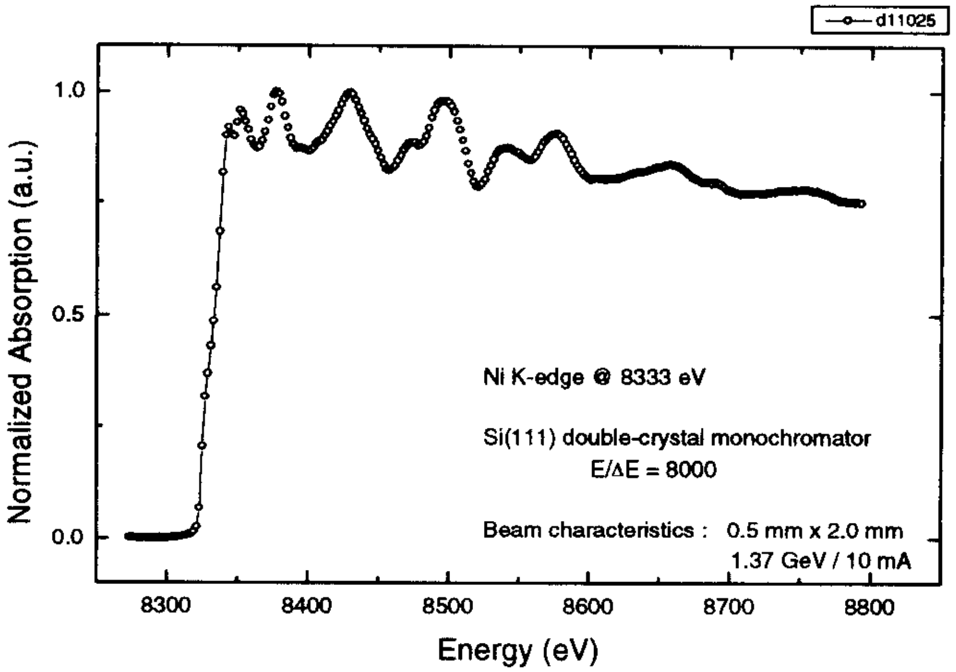

Figure 6: EXAFS spectrum of Ni close to the K edge[11].

A small-angle X-ray scattering spectrum of collagen obtained in the SAXS beamline is presented in Fig. 7. This experiment was performed using an X-ray beam vertically focused by a gold-coated mirror and monochromatized and horizontally focused by a (111) Bragg reflection from a bent silicon single crystal. A one-dimensional position-sensitive detector was utilized to record the X-ray photons scattered in the vertical direction at small angles. The SAXS spectrum plotted in Fig. 7 as a function of the modulus of the scattering vector, q, exhibits a number of peaks which are associated with the periodicity (about 630 Å) of the atomic structure along the direction of the collagen fibers.

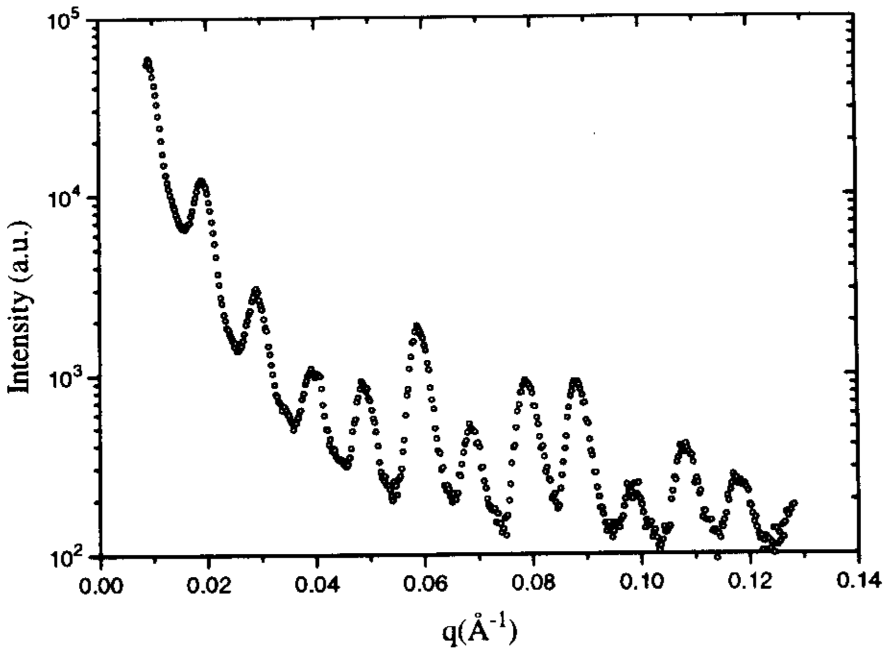

Figure 7: SAXS spectrum of collagen with fibers axes parallel to the direction of detection(12).

Other recent experimental results concerning i) XAFS studies of the atomic structure of nanocrystalline Cu-Co alloys and ii) SAXS investigations on the crystallization process in polymers and on conformational changes of lysozyme in aqueous solutions will be reported in the forthcoming XX Brazilian Meeting on Condensed Matter Physics.

5. Access of external users to LNLS beamlines

LNLS, being a National Laboratory, is open to all the scientific community interested in using the synchrotron radiation beamlines and experimental workstations for investigations in physics, chemistry, material science, biology and other fields.

As stated in previous sections, five beamlines will be available for external users in July 1997, two in September 1997 and two in 1998. Many other beamlines can be added (up to 24), the precise planning depending strongly on suggestions and specific projects which are expected to be submitted by users from Brazil and other countries. The schedule for the installation of additional beamlines will depend on demand, funding conditions and availability of technical manpower.

Several modalities for interaction between LNLS and users of the synchrotron source are possible:

- Qualified scientists from any institution and any country may apply for the use of the available beamlines and workstations for scientific or technological applications.
- Scientists from any institution may collaborate with the LNLS X-ray or VUV Instrumentation Groups for the design and construction of beamlines and workstations associated with experimental techniques of common interest.

- External users may be allowed to install their own workstations and associated instrumentation at LNLS, temporarily or permanently, using one of the available beamlines for scientific applications.
- Scientists or engineers interested in industrial applications, such as routine material characterization, microfabrication, microlithography, etc, may use the available beamlines and workstations or establish joint projects with LNLS for the installation of additional beamlines with special features and particularly relevant applications.

The research projects for the use of the LNLS beamlines will be reviewed by external referees as is usual in similar national laboratories. *Additional information and the application form for research proposals can be found on the LNLS Internet Home-Page (http://www.lnls.br/).*

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(11) Figure provided by H. Tolentino.
(12) Figure provided by C. Barberato.