# SpLine: Spanish BM25 CRG X-ray beamline at the European Synchrotron Radiation Facility

# Juan Rubio-Zuazo and Germán R. Castro

SpLine Spanish CRG Beamline at the ESRF, ESRF-BP 220-38043 Grenoble cedex-France.

Instituto de Ciencia de Materiales de Madrid-ICMM/CSIC Cantoblanco E-28049 Madrid, Spain

# Abstract

The main goal of the interdisciplinary and multipurpose Spanish BM25 CRG Xray beamline SpLine is to satisfy the needs on the use of synchrotron radiation, in the region of the hard X-ray, of the Spanish Scientific Community. The beamline covers a broad range of interests crossing very different research areas: physics, chemistry, material sciences, geology, mineralogy, biology, environmental sciences, and culturage heritage. In this sense, the Spanish CRG beamline contributes to increase long term research projects using SR and provides competitive facilities to the Spanish Scientific Community interested in the use of hard X-ray radiation. By using the advantages of a wide Front End (9 mrad), SpLine is split in two braches covering a wide energy range: 5 - 45 keV. Both branches are fully equipped with its focusing optics and experimental stations.

Each branch has allocated two experimental stations with similar technical constraints and use 2 mrad of the available radiation fan. Branch A is allocated on the soft edge (critical energy of 9.6 keV) and branch B on the hard edge (critical energy of 20.6 keV).

The experimental stations installed in branch A enables the performance of X-Ray absorption Spectroscopy and High Resolution Powder Diffraction measurements. While in Branch B are allocate facilities for Surface X-Ray Diffraction and Hard X-Ray Photoelectron Spectroscopy measurements.

#### Introduction

The main goal of the interdisciplinary and multipurpose Spanish BM25 CRG Xray beamline SpLine is to satisfy the needs on the use of synchrotron radiation, in the region of the hard X-ray, of the Spanish Scientific Community. The beamline covers a broad range of interests crossing very different research areas: physics, chemistry, material sciences, geology, mineralogy, biology, environmental sciences, and culturage heritage.

The BM25 CRG beamline SpLine is operated and allocated on the D25 bending magnet at the European Synchrotron Radiation Facility (ESRF) in Grenoble France. The BM25-SpLine was built through a coordinated project with the participation of different groups. The project coordination for the beamline construction was led by the Material Science Institute of Madrid, Spanish National Research Council ICMM-CSIC. The construction project has been regulated, followed, and supported by the Spanish Government through its Science and Technology Offices and Ministries OCYT, MCYT, MEC and MICINN. The beamline operation has started in 2005 and is regulated by a contract between the CSIC/MICINN and the ESRF. The MICINN (formel MEC) is responsible for the beamline operation and its accessibility to the Spanish scientific community. The MICINN appointed the Spanish National Research Council (Consejo Superior de Investigaciones Científicas-CSIC) as the beamline management body to deal with BM25-SpLine beamline management.

The access to the BM25-SpLine infrastructures is open to all scientists with priority for the Spanish scientists. According to the operation contract signed between the MICINN and the ESRF concerning the BM25-SpLine, 30% of the beamtime is offered to the scientific community trough the ESRF as for any other public beamline. The other 70% is managed by an internal Spanish allocation committee. Therefore, there are two ways to apply for beamtime; either through the ESRF standard procedure or through the SpLine procedure. In both cases the same application form will be used but two different committees will revise the proposal. The protocol of access is detailed in the BM25-SpLine public web page. A submission of a proposal is compulsory in order to access the BM25-Spline infrastructures. In this proposal the potential user explains the proposed experiment emphasizing its scientific relevance and justifying the

use of the infrastructure. The proposal is evaluated technically by the BM25-SpLine staff and scientifically by an external access committee. If the proposal is approved then the BM25-Spline staff helps in the organization of the experiment. The BM25-Spline staff also helps, if requested by users, in the realization of the experiment and in the data analysis and interpretation.

The synchrotron radiation at the ESRF, which is emitted by very high energy electrons moving inside a ring, consists essentially of X-rays and serves mainly to study the atomic and molecular structure of matter. The synchrotron radiation is applied to different areas: physics, chemistry, biology, materials science, geophysics, environment or medicine. The beamline BM25-SpLine offer their users a research tool of exceptional characteristics, leading technologies, support and the competence of a multidisciplinary team of researchers, engineers and technicians.

The technical characteristics of hard X-ray synchrotron radiation and the experimental possibilities that offers go beyond those from conventional laboratories sources and other national facility in Europe and worldwide, both with regard to the brightness and stability, and tuning feasibility of the produced beam.

Due to the complexity of this unit and the tasks that involves both the operation and construction have required and will require the participation of a large number of people working in very special working conditions and great coordination, which involves close cooperation with high-level specialists in various fields. In this sense, the BM25-SpLine beamline will contribute to increase long term research projects using SR and provides competitive facilities to the Spanish Scientific Community interested in the use of hard X-ray radiation.

The beamline has been designed to give the Spanish Scientific Community access to a third generation synchrotron radiation facility to perform X-ray absorption and diffraction experiments in a broad energy range.

#### **Beamline design**

By using the advantages of a wide Front End (9 mrad), SpLine is split in two braches covering each a wide energy range (5 - 45 keV) with a high photon flux, Fig. 1. The Spanish SR necessities and interests may be served providing a beamline with experimental facilities for X-ray absorption spectroscopy (XAS), high resolution powder diffraction (HRPD), single crystal and interface diffraction (SCD) and hard X-ray photoemission spectroscopy (HAXPES).



Figure 1. (Right) Photograph of the SpLine optical hutch. (Left) Flux curve as a function of the photon energy for Branch A (top) and B (bottom). The energy covered by each branch ranges between 5 keV and 45 KeV with a photon flux of  $10^{13}$  pht/s/0.1%b.w

Branch A of the Spanish CRG SpLine beamline is allocated on the soft edge of the D25 bending magnet with a critical energy of 9.7 keV. It accepts, in horizontal, 2mrad of the beam delivered by the D25 dipole. The energy range covered by this infrastructure is between 5 keV- 45 keV. The spot size can be changed between 120 mm x 8 mm (H x V) and 0.3 mm x 0.1 mm. The optics accept the 2 mrad horizontal aperture and produce a small focal spot at different sample positions. The optics configuration is mirror-monochromator-mirror, as depicted in Figure 2. The beam is first vertically collimated by a one meter long cylindrically bent mirror of fixed meridional radius. The horizontal focusing is achieved by the second monochromator crystal through a sagittal cylindrical bending. By a second vertically bendable mirror the beam can be focused to different sample positions 40-45 meters away from the source. The XAS station is allocated at the sample position A1 (40m), while the HRPD station is at the sample position A2 (45 m). All optical components of the beamline are operated under vacuum. A fixed Be (500 microns) window at the front end is used to separate vacuum between the beamline and the electron ring. Therefore the effective cut-off energy is around 4 keV. In order to reduce the influence in the region of low energy (increasing the real cut-off energy) vacuum gate valves with a beryllium disk are used to separate the different components. In some special cases, if necessary and if the vacuum conditions are good the Be windows can be opened for maximum flux. The first mirror (collimating mirror) with a constant curvature radius is coated with Rh and Si. The angle of incidence (glancing angle) is set at 2.5 mrad, which corresponds to a cut-off energy of 26.8 keV for the Rh coating and 12.4 keV for Si. Due to the high power loads of the ESRF beams (~ 150 Watts) cooling of the mirror is mandatory.



*Figure 2. Schematic representation of the beam path for the configuration of Branch A. Dimensions are given in millimeters* 

The monochromator is a pseudo channel-cut with two fixed Si(111) crystals moved together by a simple goniometer circle, in the (-n,+n) configuration. The possibility to change both crystals on the monochromator according to resolution

requirements (i.e. Si(311)) has been considered. The first monochromator crystal is water cooled while the second is kept at room temperature. The second crystal is equipped with a piezoelectric driver that allows to change very slightly the Bragg angle (pitch adjustment) in order to reduce the harmonic content of the beam, if necessary, and to keep the transmission of the monochromator constant during long time intervals. Also, a bender curves sagittally the second crystal in order to dynamically focus the beam at the sample positions A1 and A2 (see figure). The position and dimension of the focused beam are kept constant during a  $\sim$  1 keV energy scan, standard in a EXAFS measurement.

Branch B is located on the hard edge of the bending magnet D25 device with a critical energy of 20.6 keV. As in the case of branch A, the optics accept the 2 mrad horizontal radiation and produce a small focal spot at different positions (B1, B2, and B3) in the experimental hutch B. The optics layout is equivalent to branch A with a mirror-monochromator-mirror configuration, Figure 3.

The first mirror is collimator and is located before the monochromator beamstream. The horizontal focusing is achieved, as in the case of branch A, by the second monochromator crystal through a sagittal cylindrical bending. For focusing in the meridional plane a cylindrical bent mirror is used. In all the energy range the beam spot size is around  $300 \times 100 \text{ }\mu\text{m}^2$  in the horizontal and vertical direction, respectively. The vacuum of the main components is separated by fixed Be Windows. The main function of the first mirror is to reject the higher order harmonics, produce a non dispersive optical setting and reduce the heat load on the monochromator. Since a Rh coating is used a cut-off energy of 26.8 keV is achieved for a glancing angle of 2.5 mrad. A pseudo channel-cut type monochromator is used. Two Si (111) crystals placed at  $\sim$ 30 meters from the source serve as a double-crystal monochromator, which gives an energy resolution of  $\Delta E/E=1.5 \times 10^{-4}$ . The beamline X-ray energy ranges between 5 keV and 45 keV with a X-ray flux of  $10^{13}$  photons/s flux, that is well adapted for the HAXPES and XRD requirements. Contrary to the case of Branch A the beam is moved upward in order to gain more free space at the sample position in the experimental hutch A.

The Si(111) Darwin width meets the bandwidth necessities for resonant anomalous scattering measurements. A high-resolution channel-cut (Si(311), Si(333), and Si(400)) post-monochromator is located close to the vacuum chamber for cases where a better excitation source resolution in the HAXPES measurements is required.



*Figure 3. Schematic representation of the beam path for the configuration of Branch B. Dimensions are given in millimeters.* 

# **Experimental stations**

In 2007 and 2008 the BM25-SpLine has carried out two upgrading programs in order to improve the beamline capabilities and the user service. In 2008 special effort has been allocated to the development of new and more efficient photon detectors. For the Hard X-ray Photoemission station a novel 2D detection system has been developed. A novel and innovative multi-crystal (10) detector has been developed for High Resolution Powder Diffraction. The installation of these detectors are programmed for the end of 2009 and beginning of 2010, respectively.

A standard multi-elements solid-state fluorescence detector has been purchased and installed for the X-ray Absorption Spectroscopy technique, as well as a optic bank, cryostats and diverse support material.

#### **Branch** A

This infrastructure is devoted to the realization of material science experiments. The experimental stations installed in this branch enables the performance of X-Ray absorption Spectroscopy and High Resolution Powder Diffraction measurements. Branch A of the Spanish CRG SpLine beamline incorporates high technology equipments for the realization of material science experiments.





Figure 4. (Top) Photograph of the HRPD experimental set-up installed on Branch A. it consists on a one-circle diffractometer, for the detector movement, a gas blower for the sample heating placed below the capillary container and a cryostream for the sample cooling placed above the sample position. (Bottom). Representative HRPD spectra measured at SpLine together with the corresponding fit.

The optical elements installed in the infrastructure have been designed at SpLine so to fulfill the specific requirements imposed by the allocated experimental stations.

High Power slits, beam attenuators, beam shutters, X-Ray mirrors, X-Ray double crystal monochromator, beam position monitors, X-Ray fluorescence screens, high precission diffractometer, scintillator detector, capillary sample holder, gas blower, cryostream, ionization chambers, UHV baby chamber, high pressure chambers, reactors, fluorescence detector, microposition sample holder are the main high technology components installed in this infrastructure.

#### BM25-SpLine High Resolution Powder Diffraction station

High Resolution Powder Diffraction measurements could be performed within a photon energy range between 5 and 45 keV. Hence, K and L-edge resonance experiments could be performed together with non-resonance experiments.

A high resolution powder diffraction set-up is installed at the focal plane A2 (~ 45 meter from the source). A heavy-duty Theta-2theta diffractometer with a distance from the center to the detector of at least 50 cm has been installed in order to study samples in different environments, such as ovens and cryostats, Figure 4. The detector arm is rigid enough to accept heavy detectors (~ 7 kg). The angular resolution for the detector movement is 0.0001 degrees. The main axis for the theta and two-theta circles is horizontal so diffraction measurements are realized on the vertical plane, profiting from the high polarization degree of the synchrotron radiation.

The samples could be accommodated in capillaries of different diameter according to the available amount of sample (as in the case of most of the cultural heritage experiments) or by the concentration of the chemical species present on the studied sample and the photon energy used. In-situ thermal treatment could be performed on the sample. The sample could be cooled down up to 80K with a cryojet system which does not disturb the detector movement. The sample could be also heated up to 1000K with an external stream oven which also does not interfere with the detector movement. A typical HRPD spectra measured on this experimental set-up is shown in Figure 4.

This experimental station is operational since July 2005. It shares experimental hutch and optics with the X-Ray Absorption station of the BM25-SpLine branch A

infrastructure. Hence the use of this service is coordinated with the use of the beamtime on the XAS station service. Both services cannot be offered simultaneously.

# BM25-SpLine X-Ray Absorption Spectroscopy station

X-Ray absorption spectroscopy measurements could be performed within a photon energy range between 5 keV and 45 keV. Hence K and/or L edge for the most relevant chemical species can be scanned. The sample environment has enough free space to install baby chambers, reactors, cryostats, ovens, etc...



Figure 5. Photograph of the XAS experimental station installed on Branch A. The 13-element Si(Li) detector is placed parallel to the X-Ray electric field. The beam pipe of Brach B crosses the experimental hutch of Branch A. The space left for the XAS station is enough for the installation of different sample environments as baby chambers, gas reactors, etc.

In-situ thermal treatment could be performed on the sample within a temperature range between 4 K and 1300 K. A gas reactor and a baby chamber are available within the XAS infrastructure. The baby chamber incorporates many surface science facilities as ion gun, evaporators, gas leak valves, low and high mass spectrometer, He-cryostat, high temperature heater, etc.

Experiments could be performed under a pressure range between ultra-high vacuum and 3 bars. Different detection modes are available. For fluorescence yield measurements a solid state detector placed parallel to the electric field of the incident

X-Rays is used, as shown in Figure 5. With this detection method thin films and multilayers can be well studied. A set-up for high resolution absorption experiments by using high resolution analyzer crystals in order to set the required fluorescence lines for fluorescence yield detection mode is being developed. Transmission experiments are performed by using two identical gas filled ionization chambers, in order to ensure the best linearity in the measurement of the photon intensity just before and after the studied sample. A third ionization chamber is present in order to measure simultaneously a reference sample. Total electron yield measurements can be also performed. The latter detection method could be performed simultaneous to the fluorescence detection mode without disturbing between each other. Within this framework different information depths are handled. Figure 6 shows a representative K-edge XAS spectra measured by the transmission mode on a Vanadium sample.

This experimental station is operational since February 2006. It shares experimental hutch and optics with the High Resolution Powder Diffraction station of the BM25-SpLine branch A infrastructure. Hence the use of this service is coordinated with the use of the beamtime on the HRPD station service. Both services cannot be offered simultaneously.



Figure 6. Representative spectra measured on the SpLine XAS station in the transmission mode. The XANES and EXAFS signals on the K-edge of a vanadium sample are shown. It can be seen the high signal to noise ratio.

#### **Branch B**

Branch B of the Spanish CRG SpLine beamline incorporates high technology equipments for the realization of material science experiments. The optical elements installed in the infrastructure have been designed at SpLine so to fulfil the specific requirements imposed by the allocated experimental stations. High precision diffractometer, scintillator detector, cryostream, beam intensity photodiodes, UHV baby chamber, high pressure chambers, microposition sample holder, charged-coupled devices, electron gun, ion gun, MBE evaporators, gas leak valves, mini-LEED, high kinetic energy electron analyzer, load-lock sample fast entry, UV-discharge lamp, high Power slits, beam attenuators, beam shutters, X-Ray mirrors, X-Ray double crystal monochromator, beam position monitors, X-Ray fluorescence screens, are the main high technology components installed in this infrastructure.

### BM25-SpLine Grazing incidence and single crystal X-Ray Diffraction station



Figure 7. Photograph of the multipurpose diffraction set-up installed at Brach B. It is based on a six-circle diffractometer in vertical geometry, a punctual detector placed on the diffractometer arm and a 2D CCD detector placed on a decoupled motorized stage. The diffractometer loads weights up to 50 Kg so different sample environments could be mounted on, as the baby chamber shown in the right picture.

Single crystal diffraction including surfaces, interfaces, superlattices and thin films can be performed with a six-circle diffractometer in vertical geometry, Figure 7.

The diffractometer has its main axis in the vertical and is able to house loads up to 50 kg. In this way UHV baby chambers, magnets, cryostats and reactor cells are easily accommodated. In addition to the standard circles for orienting the sample and the detector, additional motions to align the surface normal along predefined directions (vertical or horizontal) are required for interface scattering experiments, as it is customary in surface diffraction. For high resolution experiments, a Theta-2theta set-up together with a goniometer head is installed in the detector arm in order to use a crystal analyzer.



Figure 8. Representative diffraction measurements obtained at the SpLine set-up. (Top). Reflectivity curve of a SrTiO3/La0.7Ca0.3MnO3/SrTiO3 sample. The layer thickness could be obtained from the interference fringes. (Bottom). Surface X-Ray diffraction measurements. At the Left the 22-CTR of a Cu-Alanine sample is shown together with the corresponding fit. At the Right a reciprocal space map is shown on a 2.4 nm thickness manganite layer, evidencing a layer relaxation.

The detector arm is rigid enough to allow the installation of heavy detectors if necessary. This is an accurate instrument with a confusion sphere diameter of  $\sim 100$  microns. The experimental station incorporates a large CCD detector with an input active area of 250 mm x 125 mm. The CCD has 3 x 11 Megapixel, with a pixel size of 32.8 microns square and a final image resolution of 7651 x 3825 pixels. The sample-detector distance can be varied between few mm up to 1500 mm. For short sample-detector distances the area detector covers a wide enough solid angle of the reciprocal space so to acquire reciprocal space maps from thin films and multilayers in few seconds. For large sample-detector distances the area detector distances the area detector distances the resolution as the solid angle accepted by each pixel is extremely low. Also Small Angle X-Ray Scattering measurements can be performed for different incident angles.

The tunability of the radiation delivered by branch B enables the measurement of anomalous X-Ray diffraction. The CCD area detector can be rotated and placed with the large side either vertically or horizontally so to maximize the accepted solid angle along the out-of-plane direction or along the in-plane direction. Hence, single crystal diffraction measurements can be performed under the same experimental set-up than Grazing incidence X-Ray diffraction experiments.

Figure 8 shows representative diffraction measurements obtained with this experimental set-up. Reflectivity curves, CTRS, reciprocal space maps can be obtained with high resolution on ultra-thin films or clean surfaces (de Andrés et al, 2003 and Rubio-Zazo et al.2005-a).

This experimental station is operational since December 2005. It shares experimental hutch and optics with the Surface X-Ray Diffraction and Hard X-Ray Photoelectron Spectroscopy station of the BM25-SpLine branch B infrastructure. Hence the use of this service is coordinated with the use of the beamtime on the SXD/HAXPES station service. Both services cannot be offered simultaneously.

# *BM25-SpLine Surface X-Ray Diffraction and Hard X-Ray Photoelectron Spectroscopy station*

Surface X-Ray Diffraction and Hard X-Ray Photoelectron Spectroscopy measurements could be performed within a photon energy range between 5 keV and 45

keV and an electron kinetic energy range between few eV and 15 keV. The experimental set-up is installed at the second focusing point of Branch B. It is devoted to the recent emerging technique Hard X-Ray Photoelectron Spectroscopy (HAXPES) and Surface X-Ray Diffraction techniques (Rubio-Zazo et al. 2005b and 2007). The experimental set-up includes a heavy 2S+3D diffractometer, a UHV chamber and an electrostatic analyzer able to handle with high kinetic energy electrons. The UHV system consists on a cylindrical vessel with 27 flanges, 19 of them pointing to the sample position, five turbomolecular pumps, five dry pumps for the turbomolecular's pre-vacuum, an ionic pump and a titanium sublimation pump surrounded by a cryogenic panel. The sample surface is mounted vertically. The X-rays enter and leave the vacuum system through Be-windows, welded onto the stainless-steel chamber, which are strong enough to hold vacuum. A 200° in-plane access and a 50° out-of-plane access are allowed by the exit Be-window. A wide portion of the available reciprocal space is therefore achievable. The sample moves on the diffractometer coupled into the vacuum by a rotating feedthrough, which is composed of a differentially pumped rotating seal and a bellow that enables the adjustment of the vessel respect to the diffractometer. A specially designed mini-LEED mounted on a 63 mm flange is incorporated on the UHV system so that pre-characterisation of the ordered surface could be done. An ion bombardment gun, evaporation and gas leakage sources are also mounted on the UHV chamber that allows the possibility of doing X-ray diffraction and HAXPES experiments during growth deposition. Helium and nitrogen cooling system are incorporated so that low temperature experiments could also be performed. The UHV chamber has also MBE evaporation sources, an electron gun, a UV discharge lamp and a load-look port. In Figure 9 a picture of the UHV experimental set-up is displayed. The UHV chamber is mounted on a 2+3 diffractometer in horizontal geometry from which it can be detached without breaking the vacuum, allowing other users to use the diffractometer.

The analyzer is an electrostatic cylinder-sector, with a compact geometry and high transmission due to second order focusing. It is based on a cylinder sector with 90° deflection and 300mm slit-to-slit distance and an entrance lens with 50mm sample distance. This gives a very compact design of the analyser that is easily integrated into a

multipurpose experiment with different techniques (Escher at al. 2008 and Rubio-Zazo et al 2008). The analyzer is capable to handle kinetic energies both up to 15 keV and down to a few eV with the same analyzer setup and power supply. It is mounted on a motorised table in order to scan the sample surface without moving the sample position. In that way, the counting rate is maximized without disturbing the diffraction experiment. Even more, the sample-lens distance is fixed to 50 mm so that the previous solid angle is left free.

The experimental set-up is located at  $\sim$ 57 meters from the source, i.e., at  $\sim$ 27 meters from the monochromator. Such a large distance imposes the necessity of high stability at the monochromator. A slight movement of the monochromator will be greatly enhanced at the sample position. However, as the sample-monochromator distance is nearly the same as the monochromator-source distance, the source beam size is recovered at the sample position.

Both tools SXD and HAXPES can be operated either simultaneously or independently to each other. This offers a unique opportunity to obtain, on a sample and under identical experimental condition, electronic, geometrical and chemical information.



Figure 9. Photograph of the HAXPES and SXRD experimental set-up installed at Branch B. It consists of a huge 2S+3D diffractometer, a UHV chamber with many surface science facilities and a high kinetic energy analyzer.

Representative HAXPES Cu and Au valence band obtained at the SpLine HAXPES-SXD set-up measured on a 21 nm thick Au film growth on a Cu polycrystalline sample are shown in Figure 10-Left. The spectra were obtained with a photon energy of hv = 7.5 keV ( $E_{kin} = 7.36 - 7.50 \text{ keV}$ ), hv = 9.0 keV ( $E_{kin} = 8.86 - 9.00 \text{ keV}$ ), hv = 1.0 keV ( $E_{kin} = 10.86 - 11.00 \text{ keV}$ ), hv = 13 keV ( $E_{kin} = 12.86 - 13.00 \text{ keV}$ ) and hv = 15 keV ( $E_{kin} = 14.86 - 15.00 \text{ keV}$ ), respectively. They are multiplied from bottom to upper by a factor of 1, 1.3, 3.5, 8.0 and 14.25, respectivitly. Note the absolute and relative cross section differences in the spectra. Using the advantage of tuneable X-ray radiation the photoelectron kinetic energy, i.e. the information depth can be changed. Figure 11-Right shows the experimentally obtained photoemission peak intensities ratio of Cu 3s substarte signal and Au 5s overlayer signal as a function of their kinetic energy. The normalized signal is only a function of the EAL and of the atomic density N(z) functions of the corresponding measured element, i.e. Cu and Au.



Figure 10. (Left) Representative HAXPES spectra measured at the SpLine set-up. The extended valence band region of a Au layer (21 nm) in-situ deposited on a Cu substrate is shown for different excitation energies. It could be seen that at high electron kinetic energies photoemission signal from the Cu buried substrate is detected. (Right) Ratio of the Cu 3S and Au 5S photoemission signal as a function of the photon energy, i.e., electron kinetic energy. It could be seen that for high excitation energies the contribution from the buried substrate arises.

A typical Surface X-Ray Diffraction spectra is shown in Figure 11. The 10-CTR is shown for a system formed of a SrTiO3 (53 nm)/La0.7Ca0.3MnO3(5 nm)/SrTiO3 multilayer. The diffraction signal from the manganite layer could be observed at half-integer L values evidencing the double-STO lattice parameter. The interference fringes arising from the manganite finite layer thickness and from the total layer thickness can be also observed. This experimental station is operational since May 2006. It shares experimental hutch and optics with the Grazing incidence and Single Crystal X-Ray Diffraction station of the BM25-SpLine branch B infrastructure. Hence the use of this service is coordinated with the use of the beamtime on the GID/SCD station service. Both services cannot be offered simultaneously.



Figure 11. Representative Surface X-Ray Diffraction measurements obtained at the SpLine setup. The 10-CTR is shown for a multilayer sample of SrTiO3 (53 nm)/La0.7Ca0.3MnO3(5 nm)/SrTiO3. The spectra displays a manganite diffraction peak at half-integer L values, a manganite peak overlapped to the substrate peak at integer L values, the fringes from the manganite thickness layer and the fringes from the total multilayer thickness.

# **Conclusions and outlook**

The BM25 CRG X-ray beamline SpLine at the European Synchrotron Radiation Facility contributes to increase long term research projects using hard X-ray SR and provides competitive facilities to the Spanish Scientific Community interested in the use of hard X-ray radiation. The beamline covers a broad range of interests crossing very different research areas: physics, chemistry, material sciences, geology, mineralogy, biology, environmental sciences, and culturage heritage.

The excellent performance and flexibility makes this beamline a unique and powerful facility for Absorption and diffraction experiments. The beamline has been designed to give the Spanish Scientific Community access to a third generation synchrotron radiation facility to perform X-ray absorption and diffraction experiments in a broad energy range.

By using the advantages of a wide Front End the beamline is split into two branches. Branch A allocated on the soft edge enables the performance of X-Ray absorption Spectroscopy and High Resolution Powder Diffraction measurements. branch B allocated on the hard edge has facilities for Single crystal diffraction including surfaces, interfaces, superlattices and thin films, Surface X-Ray Diffraction and Hard X-Ray Photoelectron Spectroscopy measurements

#### Acknowledgements

We thank the SpLine staff for their precious help in the development, construction and operation of beamline BM25 SpLine. We want also to thank the different institutions who help in the beamline construction project: Material Science Institute of Madrid ICMM (Instituto de Ciencia de Materiales de Madrid: (www.icmm.csic.es); CIEMAT, Centro de Investigaciones Energéticas, Medio-Ambientales y Tecnológicas (www.ciemat.es); The Universidad Autónoma de Madrid (www.uam.es); The Instituto Eduardo Torroja (www.ietcc.csic.es); The ICMA, Instituto de Ciencia de Materiales de Aragón (www.ietma.csic.unizar.es) and The Universidad de Oviedo (www.uniovi.es) in cooperation with the Universidad de La Coruña (www.udc.es). Financial support was provided through Spanish ministry of Education and Science (MEC) Grants nos. FAP-2001-2166 and MAT1999-0241-C01.

#### References

de Andrés, A., Rubio, J., Castro, G.R., Taboada, S., Martínez, J.L., Colino J.M. (2003). Applied Physics Letters, Vol 83-4.

- Escher, M., Merkel, M., Rubio-Zuazo, J. and Castro, G.R. (2006). Proceedings of Recent Trends in Charged Particle Optics and Surface Physics Instrumentation, Brno, Czech Republic
- Rubio-Zuazo, J., de Andrés, A., Taboada, S., Prieto, C., Martínez J. L., and Castro, G. R. (2005-a) Physica B 357- 1-2, 159-164
- Rubio-Zuazo, .J. and Castro, G.R. (2005-b). Nuclear Instruments and Methods in Physics research, Section A, 547 64
- Rubio-Zuazo, J. and Castro, G.R. (2007). Reviews on Advanced Materials Science, 15 pp. 79-86.

Rubio-Zuazo, J., Escher, M., Merkel, M. and Castro, G.R. (2008). Journal of Physics: Conferences Series, Vol 100 pp. 072032