Orsay 1st June 2004

Dear Users,

here are the latest news of the SAMBA beamline.

The scientific team around SAMBA has changed with the recruitment of Emiliano FONDA at the begining of February 2004 and the leave of Agnès TRAVERSE. We would like to thank Agnès for the important work she has done for SAMBA during its conception phase and until the approval of the project. You will still have the pleasure to meet her within the team involved in the LURE/ELETTRA convention. Please note that Emiliano FONDA is the new scientific contact for the Physics and Surface Science Communities.

A web page dedicated to SAMBA is now available on the web site of the SOLEIL synchrotron (http://www.synchrotron-soleil.fr/anglais/lignes/SAMBA/SAMBA.htm). We invite you to join us there. We resume in the next part the progress report of the beamline.

All the optics of the beamline have been defined. You will find at the end of this document their synopsis. The collimating and the focusing mirrors with their benders are on the drawing table of the manufacturer. The monochromator dedicated to the sagittal focusing will be chosen in the middle of June after audition of the potential societies. The second monochromator dedicated to Quick-EXAFS is a collaboration with the group of Prof. Frahm from the university of Wuppertal. On SAMBA, the available time scale using the Quick-EXAFS monochromator (cf Frahm, R., Richwin, M, Lützenkirchen-Hecht, D. *Phys. Scripta* **2004**, Proceeding XAFS XII, Malmö) will range from ten ms for XANES to a few hundreds ms for EXAFS.

We are begining now the design of the first experimental hutch. At the end of this letter we report a summary of the proposals made in the Avant Projet Sommaire of the SAMBA beamline for the sample environnements. Then, we remind you briefly our latest acquisitions and those foreseen. We invite you to contact the scientist in charge of your community to define and list your needs if they have changed since the writing of the APS. If your needs or scientific field are different from those in our inventory, you can directly contact the beamline staff.

Thank you for your interest and for your contributions to the project.

Yours sincerely

Stéphanie, Emiliano and Valérie.

Extract from the APS of SAMBA MAI 2002

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3) Sample Environment : from the detection to the samples

a) Detection Modes

Three detection modes must be available on the beam line :
1) The use of ion chambers filled with different gases depending on the required energy is the most versatile system available for the ***transmission mode***. Three ion chambers (I0, I1 and Reference ion Chamber I2) associated to full electronics including three Keithley current amplifiers (working from the pA to nA range) are necessary.

2) The station must be equipped with a multielement solid state detector for ***fluorescence*** experiments. Such a detector with energy discrimination is necessary for dilute systems or thin layer characterisations. The electronics must include semi-automatic gating of the energy range to be selected, high Input Count Rate capability (typically 100 000 cts/s) and dead time correction.

3) Measurements carried out on supported films, bulk materials etc … need to be recorded in ***total electron yield*** detection mode. This includes the development of special detectors. The electronics is the same as for transmission detection (in particular sensitive current amplifiers in the pA range)

b) Acquisition Modes

An acquisition timescale varying from a few seconds (***Quick-EXAFS mode***) to a few minutes (***step by step EXAFS***) must be available on the beam line.

The use of ***quick scanning EXAFS*** is a prerequisite to ***follow dynamical processes*** (phase transition under temperature, hydrolysis-condensation processes in sol-gel chemistry …). Some of the ancillary equipment (e. g. see below differential scanning calorimetry (DSC)) will be fully exploited only if they are coupled with Quick-EXAFS. This option must be planned from the conception of the monochromator system, data acquisition and storage system. Among the 2 possibilities to carry out Quick-scanning of energy, we favourably consider the use of a DC motor feedback servo system to scan the monochromator at constant angular speed.

c) Sample Environments

i) For static working mode

Four kinds of sample environment should be made available for characterisation in the so-called ***static working mode*** :

A. ***Cryostats*** allowing the reduction of the damping effect due to thermal motions, the damage protection of the samples against photon irradiation (e.g. biological samples) and the study of temperature dependent behaviours.
B. ***Controlled atmosphere chamber*** allowing the damage protection against moisture, oxygen …
C. ***Ovens*** with controlled atmosphere, ***thermostated liquid cells, stopped-flow system***, ***high-pressure cells*** and ***preparation chambers*** for surface characterisation… to perform experiments under ***reaction conditions*** and/or to ***perform in-situ preparation*** of materials.
D. A ***goniometer*** allowing versatile orientation of the sample surface for the study of anisotropic behaviours (within 0,1 deg). Such measurements should be possible in the three detection modes available on the beam line.

The ***biology community*** must use its own cells which will be adapted to common equipments (cryostats, thermostated devices and pressure device to modulate the pressure between 0 and 2 kbar). Specificities would be cells with a transparent aperture to enable a laser irradiation, that means a tunable laser available on the line and cells equipped with electrodes, for coupled electrochemistry experiments (that means an micro electrochemical system) used to stabilize peculiar states of catalytic cycle of biomimetic compounds (cyclic voltammetry experiments). Note that a special attention should be paid to the development of cells having safety standards (P_2 or P_3 standards) for the study of pathogenic proteins (like prion).

In the field of ***surface science,*** the community needs : *** Present situation in Surface Science**

-an UHV analysis chamber with different detectors (total yield, multielements fluorescence detector and an high luminosity electron spectrometer (Scienta type) for partial yield detection); this last spectrometer will be also used for the resonant electronic spectroscopies measurements in the X-ray range, since the same chamber can be used for the two measurements (possibly on different beamlines). In this chamber, the sample must be heated and cooled down to ≈ 20K and rotated with respect to the polarisation direction (polar and azimutal angles). The rotation in polar angle should be precise to allow reflEXAFS measurements.

-a standard preparation chamber for the studies of metallic thin films with a complete equipment for surfaces and thin films preparation.

-a STM chamber. This chamber could be installed on the beam line in order to perform combined STM and X ray absorption experiments, either to measure EXAFS spectra of a particular dot or to use X-ray for STM elemental analysis. Tests of this method are still necessary.

-different specific preparation chambers which can be shared with other SOLEIL experiments, to prepare thin oxides layer with a oxygen plasma source and to prepare semiconductor model devices. The existence of these specific preparation chambers will offer to the surface community the possibility to study these new materials with several techniques using synchrotron radiation.

Estimated costs: 2 MF for new equipements (Scienta+ STM). These costs are not yet included in the estimated costs of sample environments presented in **Part 4**.

Note that the installation of the ultra-high vacuum chamber (2 m x 2 m x 3 m) to prepare and characterise clean samples requires a sufficient free space at the focus point. This equipment should be installed at the end of the beam line in a large enough area. In addition, a support laboratory and a storage area (see above) have to be installed in the so-called "oreille" room.

ii) For dynamic working mode

Several communities of users have clearly shown an interest to develop equipment suitable for ***dynamic measurements***.

In the field of ***heterogeneous catalysis***, the most valuable information is obtained for samples during the catalytic activity. A consortium of several laboratories in France is interested in developing equipment in order to reproduce, at the beam line, reaction conditions which are as close as possible to those existing in the home laboratory (high temperature 300-500°C, different reaction atmosphere: hydrocarbons, H_2, H_2S, NO_x, CO …

and different pressures:1-40 bars). Basic developments which should be the responsibility of the beam line scientists under the advice of the consortium of users include :

The reaction cell (1bar, 800°C) :

The powder is positioned in a boron nitride sample holder and cover plate. Boron nitride is chemically inert and not harmful for human beings. The small size of the monochromatic beam estimated to a few microns means that it is possible to decrease the size of the sample and thus the thickness of the sample holder so as to obtain higher transmission.

The furnace is equipped with two K type thermocouples : one inserted into the top of the sample mounting block and the second at the bottom to monitor the temperature in the body of the reactor cell. With such an experimental device, the maximum temperature is 700°C. Note that this information should be recorded by the computer which collects the EXAFS data.

In addition, it is usually very difficult to perform structural investigations in systems with concentrations of active components below 1%. Thus, a fluorescence reactive cell method has to be build in developed in order to study very diluted systems.

A special regulation device :

A device with various gas cylinders (such for example H_2, N_2+O_2, CO, NO, H_2S, Ar and other gases) and flow controllers with large flow rate ranges must be developed to reproduce the catalytic conditions as closely as possible. It would be of great interest to have the possibility to modify the nature as well as the rate of gas flow via the computer which controls the experiment. This configuration allows perfect timing between the beginning of the chemical reaction and the data acquisition procedure.

Security of the experimental set-up :

Due to the possible use of toxic gases, special attention has to be paid to the safety of the experiment. Several specific gas detectors must be located close to the experiments and must be linked to the regulation device in order to evacuate automatically the reactive gas in case of a gas leak. Also, the output gas has to be removed from the experiment. Thus, a system devoted to this operation has to be present on the beam line.

The activity and selectivity of the catalytic reaction could be determined by analysing the exhaust gases through gas chromatograph/mass spectrometer/catharometer. This further development should be the responsibility of the concerned community.

In the field of ***high pressure and high temperature***, the community, who has already at LURE an access to the EXAFS dispersive station, will develop experiments using the voluminous Paris-Edimburgh (PE) cells (see the description in the x-ray diffraction under extreme conditions APS) on the beam line proposed here. The PE cells allow the access to higher temperatures (about 2000°C) than the diamond anvil cells used on the dispersive station (limited typically at 700°C). The usual pressure for the PE cells used in the conditions of an X-ray absorption experiment is about 6 Gpa. The use of such PE cells is compatible with fluorescence experiments that offers the characterisation of minor elements in compounds (impurity in oxides, magnetic dopants in semi-conductors, minor elements in metallic alloys…). Note that such experiments are not possible at LURE on the dispersive set-up and will be probably not straightforward on the dispersive set-up at SOLEIL. Furthermore, the PE cells allow the study of materials with high atomic numbers which is not possible with the diamond anvil cells (due to the presence of diamond Bragg reflections not easily removed by rotation of the cell at high photon energy). Then, clearly new fields in the study of materials under high pressure and high temperature should be available on the beam line with PE cells. The experiments will take benefit of possibility of recording both EXAFS and diffraction on the same set-up. Therefore the pressure can be measured from the XRD patterns of internal standard. This possibility is discussed on the part devoted to combined experiments in this section.

For ***biological issues***, dynamic measurements are required to study for example the reactivity of pharmaceutical molecules. In this case a cell with a system assuring the circulation of the solution during kinetics experiments (like a stopped-flow system) or during HPLC experiments should be available.

The different sample environments must be set on a motorised stage allowing easy sample manipulation in x, y and z directions, and also tilt and rocking when required. This table must be able to support large, heavy (until 100 kg) pieces of experimental equipment. It must also be removable to give place to the preparation chamber for surface science.

iii) Equipment already available

A continuous He-flow cryostat with the sample being into the exchange gas (He) has been recently purchased in the framework of the "Option 1" program at LURE. With this cryostat, measurements in the three detection modes can be performed at temperatures ranging from 4K to room temperature.

Other possible transferable equipment from LURE to SOLEIL:
Thermostated liquid cells (-30°C<T<100°C) with adjustable optical path length for transmission
Liquid Nitrogen Cryostat for transmission and fluorescence experiments with automation of the flow of Liquid N_2 into the cryostat

For surface experiments, 230 kEuros of equipment including UHV elements, pumping, surface preparation and characterization devices, electronics could be transferred from LURE.

d) Combined Experiments

The optics and detection of the beam line are optimised for absorption measurements. Nevertheless, it is very convenient in Materials Science to have simultaneously an access to ***different kinds of information for the same material***. Indeed simultaneous experiments on a sample offer great advantages with respect to separate experiments, not only to spare time but also to rid oneself of errors due to differences in sample environment, thermal history, age, temperature and sample preparation. Even more important is the possibility to resolve ambiguities in the understanding of phase transitions mechanisms by allowing accurate determination of the order of occurrence of the events by the different techniques. Note that most of the combinations presented hereafter require Quick scanning EXAFS mode to minimise the recording time and to access to time resolved studies like phase transition under temperature variation (glass transition, gelification, crystallisation …).

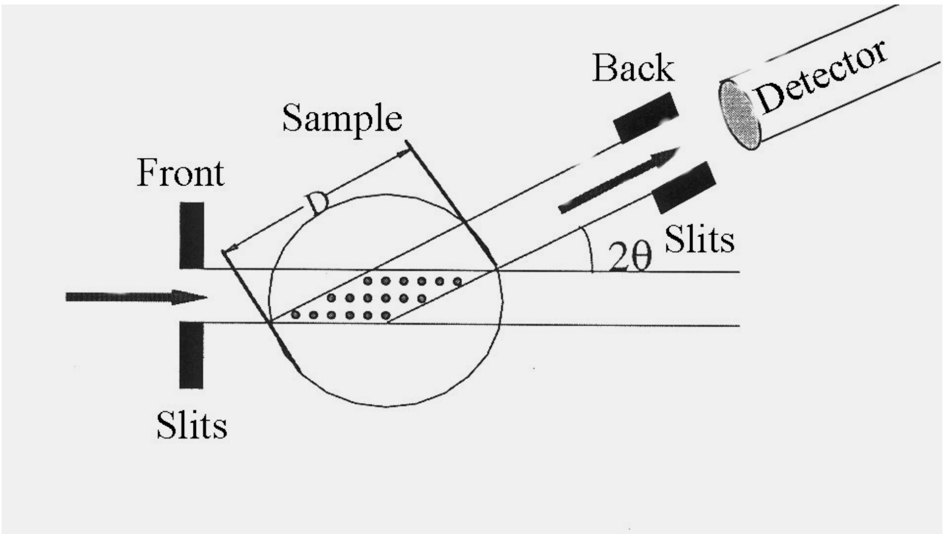
Different combinations will be available at SOLEIL thanks to a transfer from LURE :

a) Combination of ***absorption spectroscopy with thermodynamic information*** obtained from differential scanning calorimetry (DSC) experiments. We have recently purchased a Setaram DSC111 instrument (Option 1) allowing accurate determination of enthalpy of transition in the temperature range -196°C to 600°C.

b) Combination of ***absorption spectroscopy with electronic information*** obtained from UV-Visible spectroscopic experiments. We have recently purchased a Cary 50 UV-Visible Spectrometer (Varian) combined with optical fibers and allowing simultaneous recordings of UV spectra and EXAFS spectra.

For catalysis experiments, it would be useful to develop the possibility to ***combine EXAFS and Infrared or Raman Spectroscopy*** in order to access at the same time to the local order around a given atom and to the adsorption state of gases (e.g. CO) at the surface of the catalyst. A part of this development, in particular the design of reactor cell optimised for such a combination, should be the responsibility of the users' community but the purchase of the spectrometers could be included in the budget of the beam line equipment.

Finally we would like to offer to the community the possibility of recording



(simultaneously or not with the X-ray absorption spectrum) an X-ray powder diffraction pattern. This option is clearly a prerequisite of the high pressure and high temperature community for the use of Paris-Edimburg cells. More generally, the ***combination of XAS and XRD*** should be available during the dynamic characterisation of a reaction itself, that means for a sample surrounded by special cell (oven, cryostat etc…). A flexible set-up should be designed for such a combination. We can imagine to adapt a set of CdTe diodes like on the BM29 beam line (ESRF) in order to record by energy scanning x-ray diffraction recording (ESXRD) overlapped parts of XRD patterns. The principle of ESXRD is quite simple. The diffraction pattern is obtained at fixed angles as a function of the energy of the incident photons. This is quite similar to the energy dispersive set-up which is generally used with a white beam. The main advantage of the energy scanning set-up is the resolution determined by the angular acceptance of the slits system mounted in front of the detectors (10^{-3}) and the energy resolution of the monochromator which is smaller (10^{-4}). In case of energy dispersive set-up, the resolution is given by the energy resolution of the detector (10^{-2}). With 6 detectors at different angles, a large d spacing range can be covered with a limited energy scanning (< 10 keV). The sample is mounted in a quite complex assembly and the main problem for x-ray diffraction experiments is to avoid the signal coming from the environment of the sample. The energy dispersive set-up is well adapted to solve this problem because the slits system which fixes the angle of diffraction acts also as a Soller slit (Figure 9).

Figure 9 : Principle of combination of XAS and ESXRD

For other purposes we can use an image plate or CCD device as position sensitive detectors. Note that the possibility of probing both the short and long range ordered structures in a given sample by use of the combination of XAS and XRD is clearly proposed in order to optimize the XAS experiments. Structural studies which require high quality XRD data are not the scope of the beam line presented in this document but rather of the transferred H10 beam line.

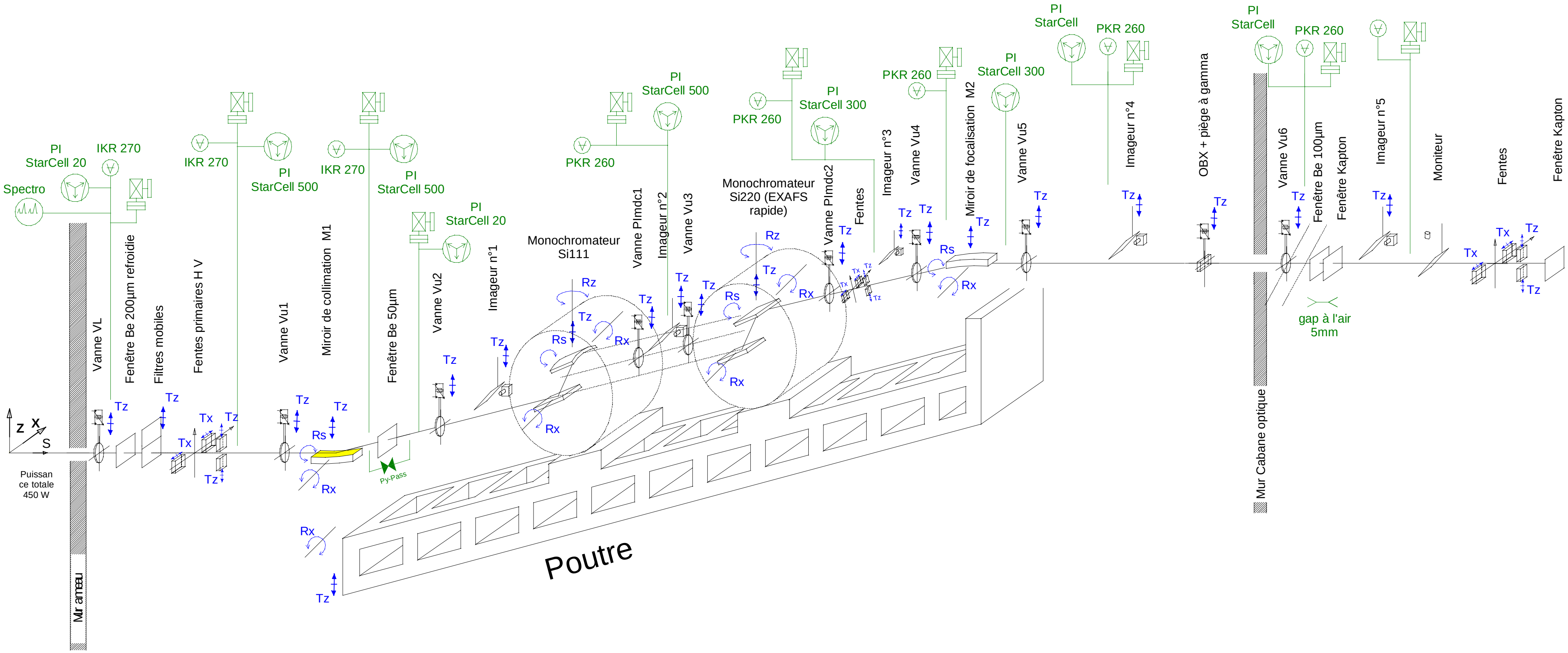
*** Present situation in Surface Science**

The manpower is at present insufficient to be involved in a real development of the SEXAFS equipment. This equipment will be transferred as it is to SOLEIL. Human resources will be allocated for the development on this part starting from the commissioning of SAMBA. This could be done better and much more quickly with the help of the community which is friendly invited to contact Emiliano FONDA.

NEWS and NOTE

We have acquired a RAMAN spectrometer (KOSI RAMAN HL5R diode laser 785 nm) which has been already described in our last letter.

For fluorescence measurements, a drift silicon detector is presently under test in the workshop of Detectors Group of SOLEIL. Depending on the results of these tests and if the claimed high counting rate will be met, we could decide to acquire one for the beamline.



Synopsis for SAMBA beamline