A multipurpose ultra-high vacuum-compatible chamber for \textit{in situ} X-ray surface scattering studies over a wide range of temperature and pressure environment conditions

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Abstract. A low/high temperature (60-1000K) and pressure (10$^{-10}$–3x10$^{-3}$ mbar) “baby chamber”, specially adapted to the grazing-incidence X-ray scattering station, has been designed, developed and installed at the Spanish CRG BM25 SpLine beamline at European Synchrotron Radiation Facility. The chamber has a cylindrical form with 100 mm of diameter, built on a 360° beryllium nipple of 150 mm height. The UHV equipment and a turbo pump are located on the upper part of the chamber to leave a wide solid angle for exploring reciprocal space. The chamber features 4 CF16 and 5 CF40 ports for electrical feed through and leak valves, ion gun, etc. The heat exchanger is a customized compact LN2 (or LHe) continuous flow cryostat. The sample is mounted on a Mo support on the heat exchanger, which has in the back side a BORALECTRIC® Heater Elements. Experiments of surfaces/interfaces/multilayer materials, thin films or single crystals in a huge variety of environments can be performed, also \textit{in situ} studies of growth or evolution of the samples. Data measurement can be collected with a punctual and a bi-dimensional detector, being possible to simultaneously use them.

1. Introduction

Advanced X-ray techniques available at synchrotron radiation sources are playing a key role for the atomic structure characterization. Grazing incidence X-ray diffraction (GI-XRD) techniques allow the studies on surface, thin film and interface materials in a non-destructive way$^{[1-3]}$. Due to the weak surface signal from these materials, the control of the X-ray penetration depth through the incident angle is essential for atomic structure surface studies. At the same time, advanced instrumentation to perform X-ray scattering techniques is improving and more sophisticated systems are developed with the aim of studying real materials at real conditions. Sensitive detectors and high flux of photons are necessary to perform the experiments, together with the constantly improved environment set-ups$^{[4-6]}$.

In this article, we present a portable chamber developed to measure surface and near-surface layers by GI-XRD under different environment conditions, being the main goal the incorporation of a cooling/heating system to control the sample temperature in a wide working range. Thus, the sample
characterization can be performed in a wide temperature range (60-1000K) and sample environment possibilities. Due to the portability character, it can be mounted in many other end-stations (for example, those with X-ray absorption set-ups). Moreover, an appropriate preparation of the chamber allows the incorporation of ultra-high vacuum (UHV) compatible components, such as evaporators, leak valves, ion guns, etc. The chamber has a cylindrical form, built on a 360° beryllium nipple, which allows a complete atomic structural characterization in GI-conditions, as well as in situ studies to follow the growth or the evolution of the samples. Besides, the experimental work conditions at the six circle multipurpose diffractometer available at SpLine-BM25B allow the use of two detectors simultaneously with the portable chamber: a scintillation point detector and a two-dimensional charge-coupled device (2D-CCD).

2. Description of the portable UHV chamber

Real image of the versatile UHV/high pressure “baby chamber” (~10^{-10} to ~10^{-3} mbar) is shown in Figure 1. The main body of the “baby chamber” is made of stainless steel. The chamber is assembled of three separated parts: lower body part which contains the continuous flow cryostat and the sample-holder heating support, the 360° double CF ended Be-nipple and the upper part with the auxiliary CF ports.
Figure 1. Real image of the portable chamber mounted on the Six Circle diffractometer at the BM25-SpLine beamline (ESRF).
The UHV/high pressure “baby chamber” has a cylindrical form, built on a 360° Be- window pipe. The Be tube has double CF100 endings, with 0.5 mm thickness, 100 mm of diameter and 150 mm long, allowing a maximal vertical scattering angle bigger than 70 degrees. These conditions leave enough solid angle to explore a maximum reciprocal space requested to accomplish GI-XRD and X-ray Reflectivity experiments. Depending on the experimental needs, some chamber configurations can be adopted. Eight flanges are located in the lower part, six CF16-UHV flanges are foreseen for electrical feedthrough necessary for sample heating and/or for conductivity measurements. Two other special flanges are used for continuous liquid coolant circulation. The upper part is formed of a multi flange cover plate with 5 UHV CF40 flanges. These CF40 flanges are foreseen for auxiliary components such as capillary gas-inlet, viewports, leak valves, ion gun, turbo pump, etc. The upper flanges pointing to the sample: one in the centre and four distributed a 90° to each other around the central one. These 4 flanges are forming a tilt angle of 14.5° respect to the sample surface normal and the flange-sample distances are 258 mm. In the central flange, there is usually allocated a turbo-molecular pump and the others are used for introducing other equipments. But if the experimental setup requests the central flange, the turbo pump can be interchanged with other systems easily. The complete “baby chamber” dimension once the equipments are mounted is of about 70 cm in height, weighting around 20 kg.

2.1. Cryostat (lower part)

Three different functionalities have been considered: continuous flow cryostat control, sample temperature control and sample electrical characterization. The mode of operation is based on a continuous flow cryostat system, with components consisting of the cryostat, storage Dewar, coolant transfer tube, temperature controller and gas flow controller at the outlet pipe. The liquid coolant is drawn from the separate cryogen storage Dewar, which is maintained at over pressure and circulated via a flexible transfer tube through to the cryostat heat-exchanger. The coolant flow can be regulated by a flow valve on the outlet tube. Both, the coolant outlet pipe and the transfer tube are flexible to allow rotating the cryostat and the baby chamber with respect to the Dewar, and consequently to positioning the sample respect the incoming X-ray beam, making the GI-XRD experiments perfectly available.

All components are made of stainless steel, with exception of the main heat exchanger which for thermal reason is manufactured out of copper. The cryostat incorporates an extended surface heat exchanger (four expansion chamber) at the cold Cu-block tip which provides efficient heat transfer between the coolant and the sample mount. Figure 2 shows a cut through the cryostat on which it is possible to identify most of the components. Temperature control of the continuous flow cryostat is achieved by a combination of manual coolant liquid (LN2 or LHe) flow control and power dissipated by an electrical heater (BORALECTRIC® Heater Elements) in contact with the heat exchanger, regulated using a temperature controller. The sample temperature has been experimentally tested, obtaining a range between 60 and 1000 K or 100-1000 K, depending on the coolant used. The sample is fixed on metal plate in thermal contact with heat exchanger.
2.2. Sample Holder

The sample is fixed to a Mo (or Inconnel) plate in hat form, which is in contact with a ceramic heater (BORALECTRIC® Heater Elements) in the back side. The sample is fixed by means of four clamps, of different dimension adapted for many sample dimensions. The contact between the Mo (or Inconnel) plate and the cryostat heat exchanger is done via four sapphire balls, which provide the indispensable thermal contact due to its excellent thermal conductivity keeping the necessary electrical isolation. These features keep the possibility to polarize the sample; besides, two electrical multi pin feedthrough are used to provide the necessary electrical contacts for different sample characterization techniques. In addition, two thermocouples are used to control the temperature, one type K (chrome-nickel and aluminum-nickel) attached to the sample holder plate and one N-type (nicrosil-nisil) to the sample.

As it is mandatory in XRD the sample must be rigidly fixed to the diffractometer, therefore the whole system is mounted on the diffractometer through a rigid support system constituted of a special adapted fixation plate which allocates four M16 screws that are joined to a solid cross arm bracket fixed to the lower body part (Figure 1).

3. Outlooks

This portable chamber has been designed to carry out a large variety of X-ray scattering experiments with a point and/or bi-dimensional detectors. The setup has been designed for standard GI-XRD and Grazing-incidence small-angle X-ray scattering (GISAXS) taking advantage of 2D-CCD detector. However, Be-reflections produced by the direct in-coming and out-coming x-ray beam could constituted an important interference problem, as the intense scattering of the X-ray incidence beam with the Be-window produce a signal that can overlap the real sample reflections. Thus, this effect could fake the sample signal and it has to be taken into account and minimized as much as possible.

In case of the presence of faked reflections, the measurements can be carried out with the point detector because the two pairs of slits that are located on the diffractometer arm avoid the problem. However, this solution presents a drawback due to the long acquisition time required for GISAXS experiments. In order to reduce the diffuse scattering produce at the Be-windows we have developed a motorized slits system which block the diffuse scattering produce by the in-coming beam and include
a support for a beam stopper inside the chamber (Figure 3 see also image in set). The motorized slits are synchronically rotated with the baby chamber (opposite directions) during the data collection, to keep the slits in the right position when the sample (chamber) is rotated.

Figure 3. Schematic general scheme of the monitored slits sited in the interior of the chamber

3. Conclusions
A multipurpose portable vacuum-compatible chamber designed to carry out GI-XRD has been described. Experiments of surfaces/interfaces/multilayer materials, thin films, single crystals or compacted powder pellets in a huge variety of environments can be performed, as well as experiments to in situ follow the growth or evolution of the sample. Their temperature can go from 60 to 1000K, as the cryosystem can work with liquid He and N$_2$. The pressure range goes from 3 bars to $\sim 10^{-10}$ mbar.

4. Acknowledgements
The authors are grateful to the SpLine staff for their assistance. The financial support of the CSIC and MINECO (PI201060E013 and MAT2011-2378) is also acknowledged.

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