JOURNAL OF SYNCHROTRON RADIATION

ISSN 1600-5775

Characterization of a Pt mirror to be used to deflect synchrotron radiation beam onto Langmuir monolayers

Carlos E. P. Vieira,[a,b] Antonio A. M. Gasperini,[b] Pedro P. S. Freitas,[b] Rafael G. Oliveira[c] and Leide P. Cavalcanti[b]*

[a]Instituto de Ciência e Tecnologia, Universidade Federal de São Paulo, São José dos Campos, São Paulo 12231-280, Brazil, [b]Brazilian Synchrotron Light Laboratory, CNPEM, CP 6192, Campinas, SP 13083-970, Brazil, and [c]Departamento de Quimica Biológica – CIQUIBIC (CONICET), Facultad de Ciencias Químicas, Universidad Nacional de Córdoba, Argentina. *Correspondence e-mail: leide.cavalcanti@gmail.com

Received 7 October 2014
Accepted 22 January 2015

Edited by P. A. Pianetta, SLAC National Accelerator Laboratory, USA

Keywords: Langmuir monolayer; diffraction; grazing incidence; deflector mirror.

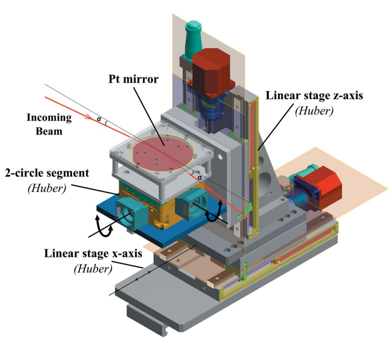
A homemade mirror for X-rays has been built to prepare a diffraction beamline for liquid surface diffraction and scattering measurements. This simple approach is in operation at the XRD2 bending-magnet beamline at the Brazilian Synchrotron Light Laboratory.

1. Introduction

Liquid surfaces and interfaces such as Langmuir monolayers have been intensively studied in different domains such as pharmaceutics, electronics, food and paintings (Daillant & Alba, 2000; Cavalcanti *et al.*, 2006). Surface-sensitive techniques such as grazing-incidence diffraction (GID) and scattering (GISAXS) are powerful tools for investigating the molecular structure on liquid surfaces using a synchrotron radiation source (Daillant *et al.*, 1996; Murphy *et al.*, 2014). As a first approach, we present here a simple way to implement these techniques at the Brazilian Synchrotron (LNLS), using a single Pt mirror to deflect the beam onto a six-circle diffractometer already in operation.

2. Experiment

The Pt mirror was inserted into the optics of the XRD2 beamline, LNLS, which is a bending-magnet source (D10A) with photon energy from 5 to 15 keV and total flux of the order of 10^{10} photons s^{-1} (100 mA)$^{-1}$ at 8 keV. The existent optics consists of a single cylindrical Rh-coated mirror to focus the beam vertically and a double-crystal monochromator focusing the beam horizontally at the sample position, in the centre of the goniometer. We used Si scatterless slits (Xenocs) to define the beam. The detector is a Pilatus 100k from Dectris mounted on the six-circle goniometer vertical arm. The deflection of the beam to study liquid surfaces is of the order of a few tenths of a degree. The Pt material was chosen due to its good reflectivity at 8 keV, the maximum intensity for this bending-magnet beamline at the 1.37 GeV source. One homemade Pt mirror was produced onto a 3-inch-diameter quartz substrate. Using the DC-sputtering technique, a buffer chrome layer ($\sim$100 Å) was deposited before the Pt layer of about 600 Å. A holder was designed to fasten the Pt mirror on top of two linear stages (5301 model from Huber) and two-circle segments (5203.10 model from Huber) (Fig. 1).



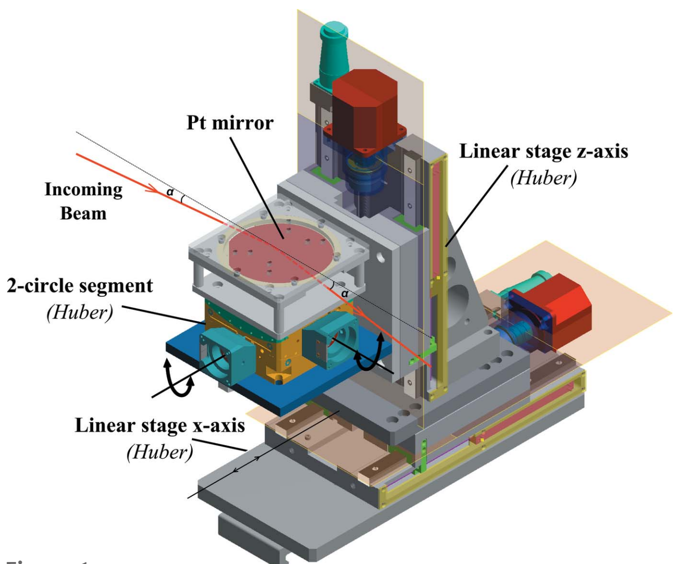
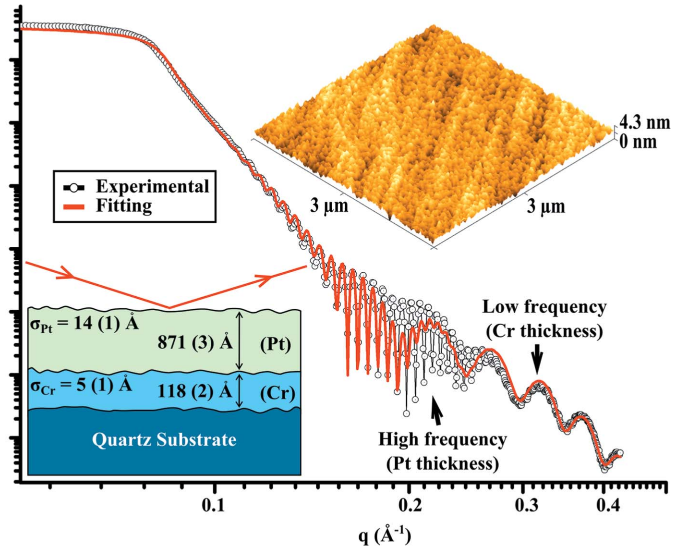
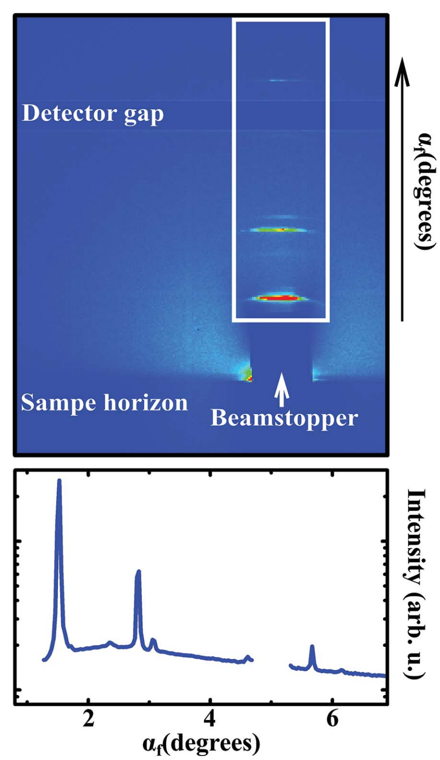


Figure 1
Mirror holder: three-dimensional representation. The incoming beam hits the Rh mirror (first element of the optics) with an angle of 3.6 mrad, keeps this direction after passing through a double-crystal monochromator, and reaches the Pt mirror where it is deflected downward to the liquid surface.



(*a*)



(*b*)

Figure 2
(*a*) X-ray reflectivity from the Pt mirror and three-dimensional topography by AFM. (*b*) Vertical scattering in the incidence plane from myelin lipid supported film placed in the horizontal using a Pilatus 300k detector.

Roughness measurements were performed using a portable atomic force microscope (Nanosurf Easyscan2). The root-mean-square (RMS) roughness value measured by atomic force microscopy (AFM) was around 16 (1) Å for Pt/quartz, which was satisfactory for this first approach compared with the RMS surface roughness of $\leq$3 Å for the commercial Rh mirror manufactured by Carl Zeiss placed before the monochromator of the same beamline. Placing the Pt mirror on the sample position, at the center of the goniometer, we measured its X-ray reflectivity. The data were analyzed using *GenX* (Björck & Andersson, 2007) software for thin films. We made a simulation based on elements of the mirror: platinum coating, chromium buffer and quartz substrate. Then the experimental data were fitted to obtain the parameters of thickness and roughness as shown in Fig. 2. The roughness of 14 (1) Å matches with the result of AFM described above.

Myelin lipid film deposited on a solid substrate was placed in the horizontal simulating the geometry condition of an experiment with a Langmuir monolayer. The myelin lipid in chloroform/ethanol solution was spread onto a Si substrate. Fig. 2(*b*) shows a diffraction pattern of the periodicity along the lipid bilayer. This is a work in progress to complement some measurements performed with neutron diffraction at Institute Laue Langevin, France, under humid/wet conditions. Lipid bilayer stackings and Langmuir monolayer measurements can help us to elaborate structural phase diagrams of biomembranes and biomimetic materials.

3. Conclusion

The Pt mirror deflector is in operation for an incidence angle of around 0.2° with the horizontal at 8 keV at beamline XRD2. The intensity of the reflectivity was about 75% of the incoming beam for such conditions. The maximum incidence angle would be approximately 0.6° on the monolayer at 8 keV and using the Pt mirror, whose critical angle for total external reflection is 0.58°.

Acknowledgements

The authors thank Douglas Araujo and Henrique Canova for technical help at the beamline, Renan Geraldes for the technical drawings of the mirror holder, and Carlos Costa for helping with AFM measurements. The experiments were performed at beamline XRD2 at LNLS. CEPV acknowledges a grant from CAPES. AAMG, RGO and LPC acknowledge a grant from FAPESP. RGO is career investigator of CONICET.

References

Björck, M. & Andersson, G. (2007). *J. Appl. Cryst.* **40**, 1174–1178.

Cavalcanti, L. P., Konovalov, O. & Torriani, I. L. (2006). *Eur. Biophys. J.* **35**, 431–438.

Daillant, J. & Alba, M. (2000). *Rep. Prog. Phys.* **63**, 1725–1777.

Daillant, J., Quinn, K., Gourier, C. & Rieutord, F. (1996). *Faraday Trans.* **92**, 505.

Murphy, B. M., Greve, M., Runge, B., Koops, C. T., Elsen, A., Stettner, J., Seeck, O. H. & Magnussen, O. M. (2014). *J. Synchrotron Rad.* **21**, 45–56.