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Bio-REF: Neutron reflectometer, dedicated to the study of bio-related thin film systems

Kwangpyo Hong [ab*], Kyehong Lee [ab*], Jimyung Ryu [ab*], Jeonghun Han [a], Sangwon Kim [a], Cheongsoo Kim [a], Jumi Lee[bc] and Kwanwoo Shin[bc]*

[a]*Korea Atomic Energy Research Institute, Daejeon 305-353, Korea*

[b]*Sogang-HANARO Joint Center for Biological Interfaces, Daejeon, Korea*

[c]*Department of Chemistry and Institute of Biological Interfaces., Sogang University, 35 Baekbeom-ro, Mapo-gu, 121-742, Seoul, Korea*

[*]*Corresponding authors: kphong1@kaeri.re.kr and kwshin@sogang.ac.kr*

1. Introduction

With the development of biomimetic and molecular electronic devices technology, the study on the surfaces and interfaces of polymer and biological membranes attracted more and more interest in recent years. As a complementary method for other technique of surface study [1], neutron scattering can offer available means of examining the phenomenon at both of surfaces and interfaces due to its higher penetration resolution [2]. Moreover, the neutron scattering technique accesses to the nanometer range with sub-angstrom accuracy [3], therefore, it can be used to investigate the larger intermolecular structure in a complex of several molecules. The HANARO neutron reflectometer with horizontal sample geometry has recently been installed at a cold neutron port at the Korean HANARO research reactor at Korea Atomic Energy Research Institute (KAERI).

2. Methods and Results

2.1 Neutron optics

The reflectometer was installed into cold neutron research facility to be specialized in bio. A schematic overview of the bio-REF is shown in Fig. 1. A neutron guide (CG4B guide; 950 mm high x 500 mm wide) of which surface is coated with super mirror (with a Ni/Ti, m=2) transports cold neutron to instruments [4]. The curved guide removes high energy gamma radiation and fast neutron 1 m out of HANARO's shutter within the heavy concrete neutron bunker. The Neutron beam pass through a fixed monochromator, plasced 0.1 m from the beam cut in CG4B guide, to the reflectomoeter.

The monochromator is made by an assembled group of 4 pieces of highly oriented pyrolytic graphite (HOPG) crystals (75 mm × 20 mm × 2 mm). Each piece is spread for mosaic in angle of 0.4° ± 0.1° to obtain the maximum neutron flux. The monochromators are comprised of aligned HOPG monochromators, tilt stage, rotation stage, translation stage (X, Y axis), optical bench and shutter (Fig 2). The aligned HOPG monochromators are mounted on a tilt stage that provides $\pm 20^{\circ}$ of motion to adjust flatness. Since it adjusts horizontal axis of beam, scattering angle to monochromator was set by using rotation stage located below the tilt stage. Rotation stage is mounted on a translation stage for guiding beam to shutter.

Monochromatic neutrons from the crystal exit the monochromatic shield via a beam channel in the 45° take off beam path. A liquid nitrogen-cooled Be filter can be used to restrict the transmission of neutron beam with the wavelength below a certain threshold. Because there are many lattice planes with different d_{hkl} values, Be crystals is used to remove the higher order harmonics.

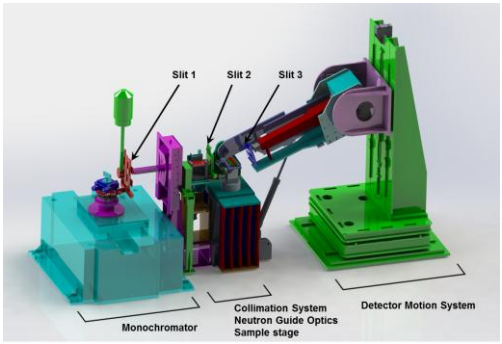

Fig. 1. The layout of bio-REF reflectometer installed at the HANARO

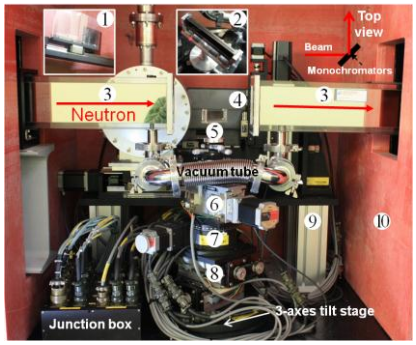

Fig. 2. Monochromator assembly located within the guide cut of the cold neutron guide. (1) beam attenuator, (2) top view of the monochromator, (3) neutron guide, (4) slit 1, (5) monochromator, (6) tilt stage, (7) rotation stage, (8) translation stage, (9) optic bench and (10) biological concrete shielding.

2.2 Sample stage

The sample stage is comprised of the top and bottom z-translation to change the height of samples, rotation stage and chi stage and align solid and liquid/solid

samples with regard to the beam. The sample translation stage has the load capacity of 300 kg. Yet, 2-circle and z-axis translation stage is currently installed for the theta-2theta measurements (Fig. 1). Distances from the monochromator to the sample, and the sample to the detector and from the sample are fixed to 2000 mm, allowing the high-resolution measurements.

2.3 Detector and readout system

The detector bank has been designed to select two different types of detectors for each specific purpose: while TD is used for narrow beams and has a low background, the 2D PSD is used to measure about wide beams. Currently, a tube type detector (TD) has been installed for the high-resolution, and high-Q measurement. The detector is comprised of elevation to do move vertically and rotation to do keep distance from the sample. The motion along the z-axis can be manually, the motion along the x-axis (rotation) can be followed by z-axis automatically. (Fig. 4)

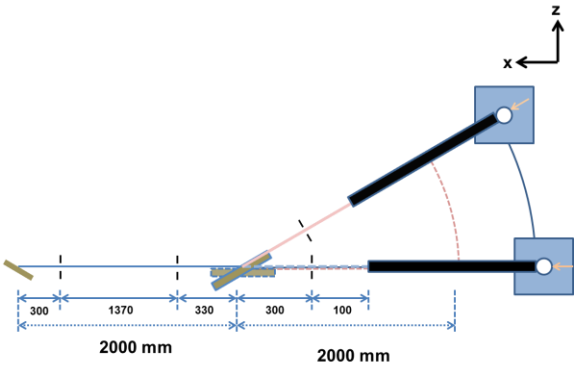

Fig. 3. Theta-2theta geometry for the extended Q-range measurement, maintaining the horizontal sample geometry.

The bio-REF is a suitable instrument for measure reflection at air/solid and liquid/solid interfaces. A deuterated poly-styrene (d-PS) on Si substrate was used as the sample of measurements to check the feasibility of the reflectometer. The film was spun-coated on a Si wafer slab with diameter of 76.2mm. The approximate thickness of the film was 22 nm. It should be noted that the neutron reflectivity profile of the d-PS thin film was collected with highly extended wave-vector transfer (Q) range from 0.003 up to 0.5 $Å^{-1}$, clearly proving its capability as the state-of the art performance.

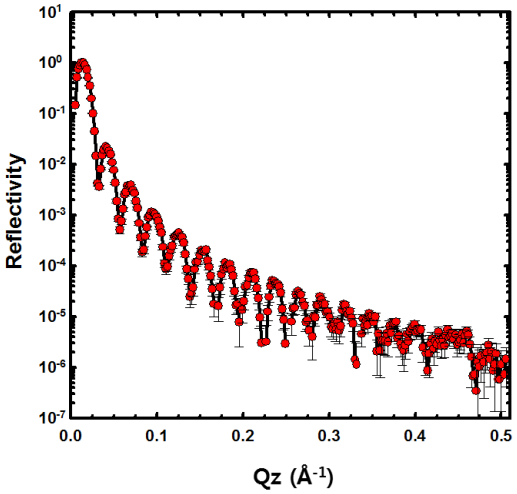

Fig. 4. Neutron reflectivity for d-PS film for the test of feasibility of thin films, collected by the HANARO Bio-REF reflectometer as a function of wave-vector transfer, Q.

3. Conclusions

The new Bio-REF established to Cold Neutron Guide Hall in 2013, which is dedicated to the characterization of the bio-related systems, supported lipid layers, polymer brushes at the solid/air, and the liquid/solid interfaces. The wavelength from the cold source was used at 4.744 Å and the available Q ranges is up to 0.5 $Å^{-1}$. The performance and quality of the Bio-REF are considered as one of the state of arts in this field, and will provide new opportunities for the researchers in polymer and biological science communities.

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