Progress with Hot Commissioning on the SPATZ Neutron Reflectometer

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Neutron reflectometry provides information on the composition, thickness and interfacial roughness of thin films with the precision of a few atoms. These experiments may be carried out at air/solid, solid/solid and solid/liquid interfaces under a wide range of conditions. SPATZ was formerly the V18 BioRef reflectometer at the BER-II Reactor at HZB in Berlin, Germany, and was transferred to ANSTO in February 2017 under a donation agreement. The progress with the hot commissioning is described here.

Specifications

Time-of-Flight instrument located on the cold-neutron guide CG2B. A flexible four chopper system defines the incident beam.

Wavelength range: 2 - 20 Å

Wavelength resolution: 1 – 11% (selected through the chopper configuration)

Q resolution: 1.5 – 17 % (combination of chopper and collimation settings)

Minimum Specular Reflectivity: 10^{-7}

Sample geometry: Vertical

Off Specular Capability: Yes

Q-range: ~0.006 – 2.0 $Å^{-1}$

Maximum flux at sample position: 10^8 n cm^2 s^{-1}

Detectors: 2D position sensitive ^{3}He detector

Sample environments: Solid-liquid cells, *In situ* Fourier Transform Infra-Red (FTIR) spectrometer, HPLC pump for solvent exchange, Water baths for temperature control, and much more…

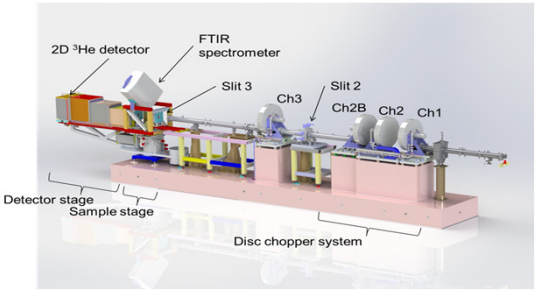

Figure 1: Layout of the major components (no shielding shown) of the Spatz time-of-flight reflectometer with vertical sample geometry.

Neutron spectrum

The neutron spectrum has been measured under different chopper configurations. The useable wavelength at 25 Hz is 2.5 to 20 Å.

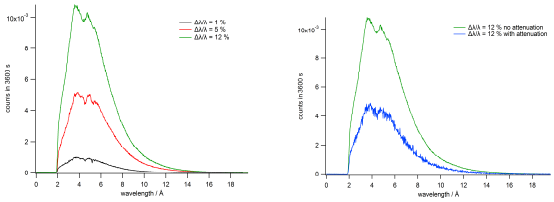

Figure 2: a) Spectrum of SPATZ under different chopper configurations. b) spectrum with oscillating attenuator.

Wavelength calibration

Bragg mirror (25 bilayers of Ni and Ti) has been used to calibrate the wavelengths under different chopper configurations and confirmed by measuring the position of the critical edge of different substrates

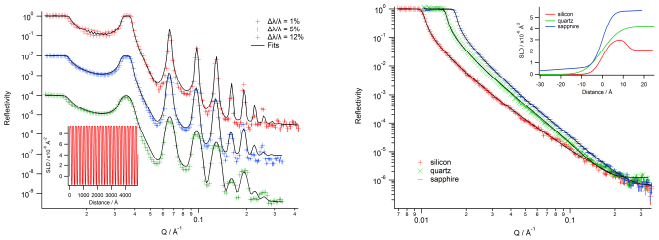

Figure 3: a) Reflectivity of the Bragg mirror at different chopper settings (inset is the corresponding real-space SLD profile from the Δλ/λ=1% fit). b) Reflectivity profiles from silicon, quartz and sapphire (inset is corresponding SLD profiles)

Polymer test sample

Polystyrene films were spin-coated onto a silicon wafer and measured by neutron reflectometry and ATR-FT-IR.

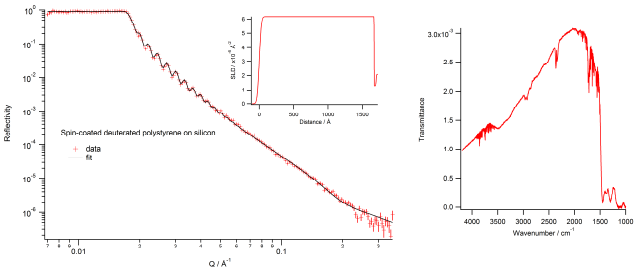

Figure 4: a) Neutron reflectivity of deuterated polystyrene on silicon in air (inset: corresponding SLD profile), b) ATR-FT-IR spectrum of hydrogenous polystyrene in air (un corrected).

Solid-liquid cells

Solid-liquid cells have been tested and isotopic solvent contrast changes can be controlled through the use of a HPLC pump.

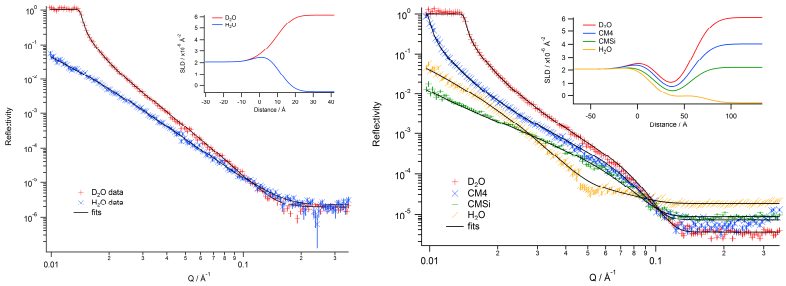

Figure 5: a) Silicon wafer under a D2O and H2O contrast. b) Phospholipid POPC bilayer on silicon under different isotopic solvent contrasts. Insets: corresponding real-space SLD profiles.

Current status

Spatz is a neutron reflectometer capable of examining surfaces, thin films, buried interfaces, multi-layered structures and processes that occur at surfaces and interfaces. This can be done under a wide range of different sample environments, including the dedicated infra-red spectrometer for *in situ* ATR-FT-IR spectroscopy that can be conducted simultaneously with neutron reflectometry measurements. So far to date a few user experiments have been completed and the number of user experiments completed each round will continue to grow.

SPATZ is now in the ACNS User Program and is accepting proposals for experiments.