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REFSANS: Reflectometer and evanescent wave small angle neutron spectrometer

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Abstract: The horizontal reflectometer REFSANS, operated by GEMS, Helmholtz-Zentrum Geesthacht, was designed to enable specular reflectometry as well as grazing incidence neutron scattering studies of both solid samples and liquid-air interfaces.

1 Introduction

By using a polychromatic incident neutron beam and time-of-flight (TOF) wavelength resolution, REF-SANS gives simple access to a large Q range. Typical reflectometry curves are recorded using three incident angles to cover the $0 - 2 \text{ nm}^{-1} \text{ Q}_z$ domain. In the case of GISANS, the TOF mode provides direct information about the full penetration curve from a single incident angle.

The instrument versatility relies on the one hand on the fact that the wavelength resolution can be tuned between 0.2 and 10 %, on the other hand on the possibility to independently control the horizontal and vertical divergence by means of a complex optic. These two characteristics make it possible to optimally perform reflectometry and GISANS. One can easily switch between these two configurations for a given sample and thereby fully investigate its structure without having to alter externally applied fields or constraints (temperature, chemical environment).

For reflectometry, a horizontally smeared out beam of up to 80 mm width is used in order to maximise intensity. For GISANS, up to 13 point beams are impinging on the sample and point focused on the 2D position sensitive detector placed at a distance of 9 m. This setup allows to resolve lateral structures with dimensions up to several micrometer. In all other cases the detector can be placed at any distance





Figure 1: Instrument REFSANS (Copyright by W. Schürmann, TUM).

between 1.5 m and 12 m from the sample, thereby making it easy to control the explored angular range and optimise the resolution/ background intensity trade-off.

2 Typical Applications

The TOF reflectometry and GISANS techniques can be used to characterise thin films in general. Reflectometry provides information about the structure along the sample's normal, while GISANS gathers information about the in-plane correlations. Typical reflectometry experiments include:

- Characterisation of polymer thin film structure and their swelling behaviour in presence of various vapors
- Biological systems such as solid or liquid supported membranes (e.g determination of the morphology and localisation of proteins at interfaces)
- Metallic multilayers (e.g magnetically active films)
- Coatings

GISANS complements these measurements and has been successfully applied to polymer thin films (lateral correlations e.g in dewetted systems, detection and identification of polymer lamellae in immiscible blends or semicristalline systems), composites, nanopatterned metallic surfaces for which Bragg truncation rods have been reconstructed.

3 Sample Environment

The optimal sample size is 70 x 70 mm². Various environments are available:

- Simple sample changer for three substrates
- Vibration controlled Langmuir trough for liquid-air interfaces studies
- Magnetic fields up to 7 Tesla
- Cryostats

A heavy load Huber goniometer (max. load 200 kg) is normally used to carry the experimental set-up, but it can easily be removed and replaced by custom equipments.





Figure 2: Schematic drawing of REFSANS.

4 Technical Data

4.1 Primary beam

- Neutron guide NL2b
- Astrium choppers with wavelength resolution to be chosen in the range 0.2 10% for wavelengths in the range 2 20 Å. Rotation speed up to 6000 rpm
- Collimation: 2 vertical adjustable slits (0 12 mm) separated by 8.68 m
- For reflectometry, the horizontal divergence is maximized by use of supermirrors (m = 2 3)

4.2 Flux at sample

Typical values ($\Delta Q / Q = 3\%$):

- $1 \cdot 10^4$ n s⁻¹ (incident angle 0.2°)
- $3 \cdot 10^6$ n s⁻¹ (at 2.5°) in the wavelength range 2 to 6 Å for a 60 x 60 mm² sample.

4.3 Accessible Q-range

• Reflectometry:

 Q_z up to 0.3 Å⁻¹ for reflectivities down to the 10⁻⁷ range.

• GISANS:

 $Q_y = 9.5 \cdot 10^{-5}$ Å-1 to 0.18 Å⁻¹ (corresponding to distances from 6 µm down to 3.5 nm)

4.4 Detector

• High performance 2D 500 x 500 mm² multiwire ³He detector (pixel size 2.7 mm, efficiency 80 % at 7 Å, gamma sensitivity $< 10^{-6}$) positioned between 1.5 m and 12 m from the sample. The detector is installed in a liftable vacuum tube in order to reach exit angles up to 6 degrees at the maximum distance.

4.5 TOF analysis

• The data are acquired in list mode, each neutron arrival time and impact position being stored for later analysis. This makes it possible to perform various rebinnings in order to tune the resolution/ intensity trade-off.

