Current and future capabilities of the neutron reflectometer MIRROR at Oak Ridge National Laboratory's High Flux Isotope Reactor*

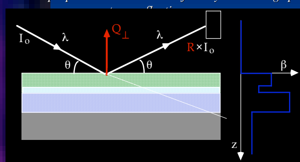
W.A. Hamilton,[a] G.S. Smith,[a] G.B. Taylor,[a] B.M. Larkins,[a] and L. Porcar[b]

[a]HFIR Center for Neutron Scattering, Oak Ridge National Laboratory, Oak Ridge TN 37831-6393, USA
[b]University of Maryland, College Park, MD 20742, and NIST Center for Neutron Research, Gaithersburg, MD 20899, USA

The peripatetic ORNL HFIR Center for Neutron Scattering reflectometer instrument MIRROR has recently been re-installed in an interim beam line position in the reactor beam room. In 2006 an upgraded version of the instrument will move to a high intensity guide hall position fed by the new HFIR cold source. We present some aspects of instrument operation - particularly with respect to data reduction from the instrument's linear reflection plane detector – with examples of ongoing research and analysis, and a brief outline of the expected capabilities of the fully upgraded guide hall instrument.

What is Neutron Reflectometry ?

Reflectometry determines neutron scattering length density β[z] and thus structure perpendicular to an interface by measuring specular

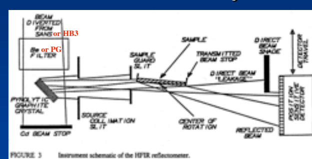


Rayleigh Approximation

$$R[Q] \approx \left| \frac{4\pi}{Q_\perp^2} \int_{-\infty}^{\infty} \frac{d\beta}{dz} \exp[iQ_\perp z] dz \right|^2$$

Typically $\boldsymbol{Q_\perp}$ *~ 0.002-0.3 Å*$^{-1}$
=> depth scales $\boldsymbol{2\pi/Q_\perp}$ *~ 3-3000Å*
Complementary to x-ray reflectometry

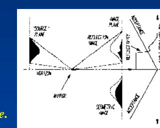
MIRROR Neutron Reflectometer 1992-2000

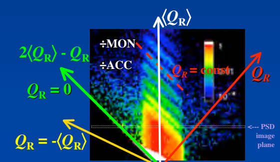


Horizontal scattering plane - vertical reflecting surface
- *complex fluid structure at solid surfaces*
- *semiconductor and metallic multilayers*
- *polymeric segregation and bio-molecular diffusion*
- *protein adsorption and biofilm structure at surfaces*
- *complex fluids under Poiseuille shear*
- *magnetic interface structures …*
- *time evolution of these surface structures …*

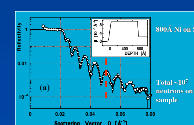
MIRROR capabilities I: Imaging Analysis of NR

- ***Mirror is equipped with a 1-D Position Sensitive Detector in reflection plane. Signal is product of source intensity, sample acceptance and reflectivity.***
- ***High resolution Specular R[Q] can be recovered from data collected across a loosely collimated wide beam. Reduction "automatic" in instrument software.***



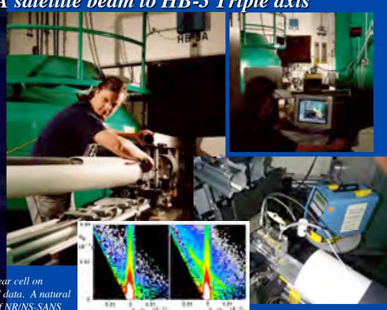


HFIR reflectomer MIRROR: data after normalization for monitor and sample acceptance (30m SANS satellite 4.75Å - 1993)



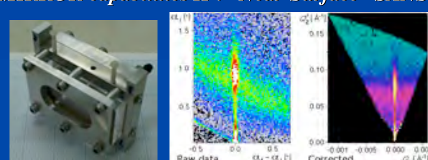
"Neutron Reflectometry as Optical Imaging", W. A. Hamilton, J. B. Hayter, and G. S. Smith, *Journal of Neutron Research* 2, 1 (1994)

MIRROR beam room installation 1995-2000
2.59Å satellite beam to HB-3 Triple axis



Poiseuille shear cell on MIRROR and data. A natural application of NR/NS-SANS

MIRROR capabilities II : "Near-Surface" SANS

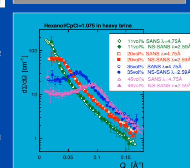


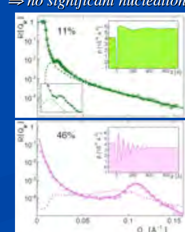
- ***Standard reflection geometry solid-liquid cell measurements: NR gives complex fluid structure within a few thousand Å of interface***
- ***Beam transmitted beam through interface will be small angle scattered within ~100μm and may emerge as off-specular scattering***
- ***Subtracting this NS-SANS signal (correctly) improves NR accuracy …***
- ***Full reduction (as per SANS) allows simultaneous monitoring of bulk phase behavior with NS-SANS while determining surface structure with NR***

Example above: "Local membrane ordering of sponge phases at a solid-solution interface", W.A. Hamilton, L. Porcar, P.D. Butler and G.G. Warr, *Journal of Chemical Physics* **116**, 8533 (2002)* [and *Virtual Journal of Biological Physics Research* **3** (2002) http://www.vjbio.org]. See also "Using Neutron Reflectometry and reflection geometry "Near-Surface" SANS to investigate surfactant micelle organization at a solid-solution interface", W. A. Hamilton, L. Porcar, and L.J. Magid, *Physica B* **357**, 88-93 (2005)

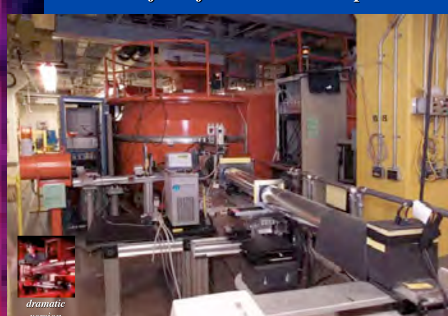
Surface constraint and isotropic phases

Surfactant sponge sample.
NR (below) ⇒ surface layering with increasing concentration.
NS-SANS is equivalent to bulk SANS ⇒ no significant nucleation.



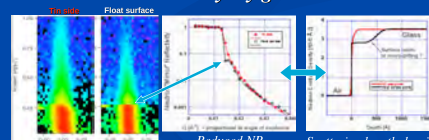


MIRROR beam room re-installation October 2004-present
4.25Å beam from future HB-2D Triple axis drum



dramatic version

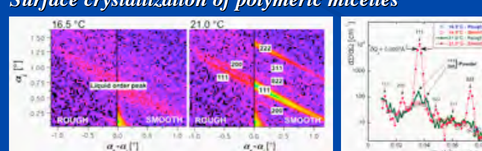
Two sides to some everyday glass



"Raw" data · *Reduced NR vs* Q_R · *Scattering length density vs depth*

Made on the surface of molten tin, common "Float" glass is is a simple technological wonder with a surface roughness of only about 10Å rms. However, the float surface (often) shows a very abrupt fall in reflectivity from the critical angle than the tin contact underside probably due to nanoscale pitting - with Larry Anovitz UT/ORNL-CSD

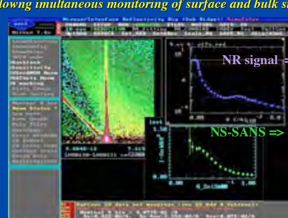
Surface crystallization of polymeric micelles



Poloxamers are tri-block copolymer surfactants that form clear thermoreversible micellar gels in water. The data at left show the difference in crystalline surface ordering of the gelation on rough and smooth quartz surfaces for the common F127 poloxamer. Rough surface scattering is simply the powder pattern of the strongly surface aligned fcc smooth surface pattern, but the transition is displaced in temperature.

Future of MIRROR NR program

Current software offers the unique capability of integrated reduction of NR and NS-SANS allowng imultaneous monitoring of surface and bulk structure



Upgraded instrument(s) will provide users with state of the art NR measurements with data collection taking full advantage of NR "imaging" in new CNS/SPICE format

Full Upgrades 2006 …
MIRROR to primary beam in Guide Hall (CG-4 #1)

Effective FLUX* × 10
Background / 10

SPEED × 10
Sensitivity × 100

Down strean on guide CG-4:
USANS/Interferometer (proposed)
US(Brookhaven)-JAPAN Cold 3X
NP Weak Interaction Station

**Cold guide estimate ~same flux at 5Å as at 2.59Å in beam room (RMM)*
Imaging NR: Shorter instrument for same resolution - Acceptance ×2 and $(1/r^2) \times 4$

**** "Mirror and Interface Reflectivity Rig - Oak Ridge" - JBH***
WAH would like to gratefully acknowledge his debt to the late John Bingley Hayter (1945-2001).
Thanks also to Dan Glandon, Scott Moore, Mohana Yethiraj and Herb Mook of ORNL for their fabrication, design and support of various aspects this work.
The construction and operation of the MIRROR reflectometer at the High Flux Isotope Reactor at Oak Ridge National Laboratory and research carried out using the instrument is supported by the U.S. Department of Energy under contract DE-AC05-00OR22725 with the Oak Ridge National Laboratory managed by UT-Battelle, LLC.