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A new neutron reflectometer at Australia's HIFAR research reactor

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Abstract

A new neutron reflectometer has been built at Australia's 10 MW HIFAR research reactor. The X172 reflectometer operates in a monochromatic, angular dispersive mode collecting reflectivity data as a function of angle. The incident neutron beam is monochromated by a pair of pyrolytic graphite crystals ($\lambda = 2.43$ Å) before being collimated using a pair of motorised sintered B_4C slits. Detection of the reflected neutron beam is via a 10-atmosphere, helium-3, linear position sensitive detector. Examples of data collected using the X172 reflectometer at air-solid and solid-liquid interfaces are given. Neutron reflectivity values as low as 10^{-5} have been measured on this instrument.

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1. Introduction

1.1. Neutron reflectometry as a probe of surfaces and interfaces

Over the past 20 years, neutron reflectometry (NR) has emerged as a powerful technique to probe the structure of surfaces, thin-films or buried interfaces, as well as processes occurring at surfaces and interfaces such as adsorption, corrosion, adhesion and interdiffusion. Neutron reflectivity is now being used for studies of surface chemistry, surface magnetism, solid films and biological systems (see Table 1).

Specular neutron reflectometry probes variation in the neutron scattering length density (SLD) normal to a flat surface at depths of up to several thousand angstroms, with a resolution of a few angstroms. The neutron SLD is determined by the composition and the density of the film of interest. Beyond the critical angle (θ_c), below which total external reflection occurs, some of the neutrons are

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Table 1
Systems typically studied using neutron reflectivity

Soft-matter systems	Hard-matter systems	Magnetic systems
Phase separation in polymer films	Photosensitive films	Multilayer materials (e.g. GMR's)
Interlayer diffusion and roughness	Non-linear optical systems	Magnetic monolayers
Inorganic templating at interfaces	Catalytic surfaces	Depth-dependent domain imaging
Complex fluids under flow	Electrochemistry	Superconducting thin films
Vesicles and gels	Polymer coatings	Superparamagnetic monolayers
Reaction kinetics	Self-assembled monolayers	Exchange-biased interfaces
Surfactants at interfaces	Langmuir–Blodgett films	Magnetic tunnel junctions
Biometric membranes		Hard/soft magnetic multilayers
Protein adsorption		
Complex fluids		

absorbed by the sample, some are transmitted through the sample and the remainder are reflected. Reflectivity (R) is measured by dividing the number of neutrons reflected from a thin-film sample (I) by the number incident upon its surface (I_0), typically as a function of the angle of incidence (θ) of the neutron beam.

When neutrons are reflected from a thin-film sample supported on a substrate they do so from both the air/sample interface as well as the sample/substrate interface. Provided that the interfaces are sufficiently sharp, Kiessig fringes appear in the reflectivity profile and the spacing between the minima of successive fringes is inversely proportional to the film thickness. In addition, if a surface is not entirely smooth its local roughness will alter the specular reflectivity in a manner similar to that of a diffuse interface.

The success of neutron reflectivity arises from the basic properties of the neutron. With wavelengths of the order of an angstrom, neutron reflectometry has a resolution of a fraction of a nanometer, so that information is gained at the molecular level. Neutron scattering lengths vary randomly from element to element, as opposed to X-ray scattering lengths that increase monotonically with atomic number. This makes them particularly useful for the study of soft-matter systems since neutrons are strongly scattered by light atoms such as H, C, O and N which form the basis of organic and biological materials. Moreover, the nuclei of different isotopes of the same element scatter neutrons with different intensity. The most common example of use of this in NR is the variation in neutron scattering length afforded by hydrogen and deuterium such that one can isotopically label different components in a sample. Generally, the technique of selectively deuterating part or all of a sample is known as contrast variation and is instrumental in the application of neutron reflectometry to colloidal, polymeric, biological and other soft-condensed matter systems.

Unlike X-rays, neutrons are highly penetrating and can be used to study buried interfaces that are not easily accessible to other techniques. Neutrons can penetrate materials such as silicon, quartz, and sapphire with little attenuation. These materials can act as both substrates for samples and windows for solid/liquid, liquid/liquid and electrolytic cells. Similarly neutrons can penetrate materials such as aluminium and alumina that are used in the construction of closed-cycle refrigerators, cryostats and furnaces.

Further information relating to the experimental basis of the neutron reflectometry technique and data analysis may be found in one of several reviews [1–5].

2. Description of the X172 neutron reflectometer

Staff at Australia's 10 MW HIFAR research reactor have recently completed the construction of the new X172 neutron reflectometer (Fig. 1). The reflectometer operates in a monochromatic,