Nuclear Instruments and Methods in Physics Research A 632 (2011) 112–123

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research A

journal homepage: www.elsevier.com/locate/nima

The multipurpose time-of-flight neutron reflectometer "Platypus" at Australia's OPAL reactor

M. James [a,b,*], A. Nelson [a], S.A. Holt [a], T. Saerbeck [a], W.A. Hamilton [a], F. Klose [a]

[a] Bragg Institute, Building 87, Australian Nuclear Science and Technology Organisation, Locked Bag 2001, Kirrawee DC, NSW 2232, Australia
[b] School of Chemistry, University of New South Wales, Kensington, NSW 2052, Australia

ARTICLE INFO

Article history:
Received 8 September 2010
Received in revised form
18 November 2010
Accepted 8 December 2010
Available online 17 December 2010

Keywords:
Neutron reflectometry
Cold neutrons
Time-of-flight
Free-liquid surfaces
Polarised neutron reflectometry
Off-specular scattering

ABSTRACT

In this manuscript we describe the major components of the *Platypus* time-of-flight neutron reflectometer at the 20 MW OPAL reactor in Sydney, Australia. *Platypus* is a multipurpose spectrometer for the characterisation of solid thin films, materials adsorbed at the solid–liquid interface and free-liquid surfaces. It also has the capacity to study magnetic thin films using spin-polarised neutrons. *Platypus* utilises a white neutron beam (λ=2–20 Å) that is pulsed using boron-coated disc chopper pairs; thus providing the capacity to tailor the wavelength resolution of the pulses to suit the system under investigation. Supermirror optical components are used to focus, deflect or spin-polarise the broad bandwidth neutron beams, and typical incident spectra are presented for each configuration. A series of neutron reflectivity datasets are presented, indicating the quality and flexibility of this spectrometer. Minimum reflectivity values of $<10^{-7}$ are observed; while maximum thickness values of 325 nm have been measured for single-component films and 483 nm for a multilayer system. Off-specular measurements have also been made to investigate in-plane features as opposed to those normal to the sample surface. Finally, the first published studies conducted using the *Platypus* time-of-flight neutron reflectometer are presented.

1. Introduction

The *Platypus* neutron reflectometer is now operational as part of an international user facility at Australia's 20 MW OPAL research reactor ANSTO in Sydney. Commissioned in 2008, *Platypus* is one of a new class of time-of-flight reflectometers to be built at modern research reactors (e.g. D17 [1] and Figaro [2] at ILL, ROG [3] at TU Delft, and REFSANS [4] at FRMII). The main reason for turning to the time-of-flight method is due to the capacity of such instruments to optimise the interplay between flux and resolution to suit each individual system under investigation. Neutron reflectometry has become one of the key scattering techniques around the world, with typically two or more reflectometers in operation at major international facilities. In most cases, one instrument is dedicated to hard matter systems such as magnetic thin films, requiring a polarised incident neutron beam and polarisation analysis, while the other is dedicated to soft condensed matter (particularly thin-films deposited at the solid/liquid or air/liquid interfaces). Usually these requirements also dictate differences in instrumental geometry, with horizontal scattering geometry typically used for polarised reflectometry and vertical scattering geometry for air/liquid surfaces. Being the only neutron reflectometer at the OPAL facility in the initial suite of seven instruments, *Platypus* was designed to be a flexible, multipurpose instrument to service both hard and soft matter user communities and capable of investigating air/liquid interfaces, solid/liquid and solid/air interfaces, dynamic systems, off-specular, and magnetic thin films [5]. The ability to conduct free-liquid experiments dictated that *Platypus* be configured with a vertical scattering geometry, while acquisition with a wide wavelength bandwidth is essential for kinetic studies of dynamic systems. The off-specular requirement demanded that the detection system was based around a 2-dimensional area detector.

The past few decades has seen neutron reflectometry become an essential tool for the study of nanoscale thin-films and processes that take place at surfaces. While early uses of this technique revolved around studies of surfactants [6], polymers [7], and magnetic thin-films [8,9], (fields of research that still remain popular); more recently, researchers have made use of neutron reflection in studies of nanotoxicology [10,11], cell membrane biology [12–21], protein adsorption [22–27], electrochemistry [28–33] and corrosion studies [34–36], organic photovoltaic systems [37–39], and light-emitting thin films [40–42]. Polarised neutron reflectometry has been widely used to characterize magnetic and superconducting thin-films and multilayers, with recent studies making use of both specular [43–49] and off-specular [50–53] capabilities.

* Corresponding author at: Bragg Institute, Building 87, Australian Nuclear Science and Technology Organisation, Locked Bag 21, Kirrawee DC, NSW 2232, Australia. Tel.: +61 2 9717 9299.
E-mail address: mja@ansto.gov.au (M. James).

0168-9002/$ - see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.nima.2010.12.075

The following paper reports the detailed characteristics and capabilities of the *Platypus* reflectometer along with a diverse range of the initial scientific studies performed using this instrument.

2. Major components of the *Platypus* reflectometer

2.1. Hardware and neutron optical elements

Details of the design and construction of the *Platypus* reflectometer (Fig. 1) have been described previously in Ref. [5]. The following text describes the major components of this spectrometer as well as the key modes of operation. Cold neutrons are generated in the OPAL liquid D_2 cold neutron moderator, with a typical operating temperature of 21 K. Cold neutrons from this moderator are directed through the CG3 curved supermirror guide (radius of curvature 1300 m; 200 mm high × 50 mm wide) within the heavy concrete neutron guide bunker, from OPAL's primary shutters to the secondary shutter at the exit of the bunker. This curved guide removes line-of sight from the cold neutron source at the point where the guide passes through the secondary shutter, such that high energy gamma radiation and fast neutrons (> 0.5 MeV) are trapped within the neutron guide bunker. Following a further 3 m section of straight neutron guide beyond the secondary shutter, the polychromatic neutron beam passes a tertiary shutter and is fed into the *Platypus* via a (20 mm high × 50 mm wide) neutron guide, with nickel coatings on the horizontal surfaces and $m=3$ supermirror coatings on the vertical surfaces.

The white neutron beam is delivered into a B_4C-lined, lead and steel disc chopper bunker, which is designed to protect staff and equipment from thermal neutrons that are scattered from vacuum windows, slits and the chopper discs; as well as γ-rays that are produced by neutrons being adsorbed by the above components. A 400 mm section of guide containing a frame overlap mirror is flanged directly onto the end of the pre-instrument guide. The frame overlap mirror (with a Ni/Ti, $m=1.5$ supermirror coating) reflects neutrons above 20 Å out of the main beam to be adsorbed within the bunker. Neutrons $\leq$ 20 Å are transmitted through the reflective coating and silicon substrate of the frame overlap mirror to the rest of the instrument. The beam passes a coarse collimation slit (s1), an optional beam attenuator and a low efficiency (10^{-6}) neutron beam monitor before entering the disc chopper system. The beam attenuator consists of a 50 mm high × 1 mm wide vertical slit formed from 3 mm thick sintered B_4C plates, which oscillates across the horizontal incident beam with constant velocity and a frequency of 2 Hz. When not required, the attenuator is parked to the side of slit s1, allowing the unattenuated beam to enter the vacuum vessel containing the disc chopper system.

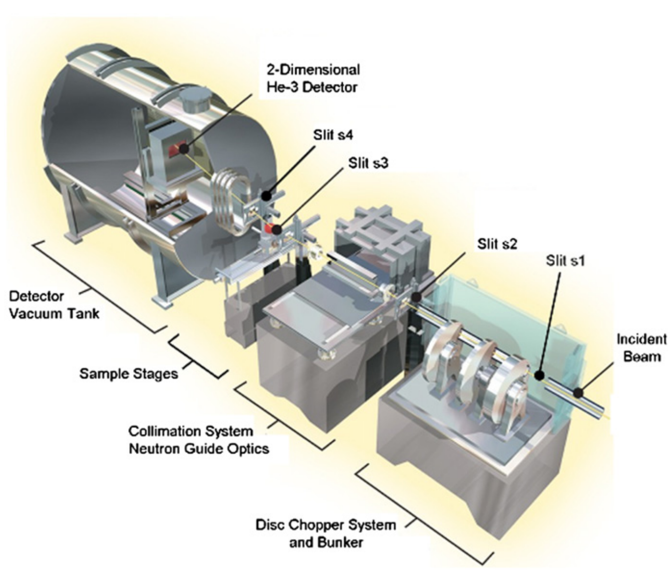

Fig. 1. The *Platypus* time-of-flight neutron reflectometer.

The disc chopper system (EADS Astrium GmbH) consists of four boron-10 coated carbon fibre discs, separated by neutron guide elements (Fig. 2). At any one time, a pair of boron-10 coated, aluminium discs rotating at 20 Hz are used to shape the neutron pulses. Full specifications of the disc chopper system are given in Table 1. The first disc (indicated by the red dashed line in Fig. 2) is the master and T_0 chopper disc that triggers the frame acquisition on the *Platypus* detector, and operates in combination with the second (green), third (blue) or the fourth (purple) slave discs. By utilising different master/slave disc pair combinations, *Platypus* is able to generate three distinct resolution settings: high- ($\Delta\lambda/\lambda=1.2\%$), medium- ($\Delta\lambda/\lambda=4.3\%$), and low-resolution ($\Delta\lambda/\lambda=8.4\%$). When not required to shape the neutron pulse, the two other slave discs are parked in a stationary, open position.

The neutron pulses are then delivered to *Platypus* collimation system, which consists of a pair of high-precision beam-defining slits (2835 mm apart) that encompass a vacuum chamber containing various neutron optical elements to focus or deflect the neutron beam towards the sample. The slit package (s2) immediately before the collimation vacuum chamber is mounted centrally with respect to the incident neutron beam, while the slit package immediately before the sample stage (s3) is mounted on a motorised vertical translation stage (Fig. 3(a)). Each slit package is capable of defining a beam from fully

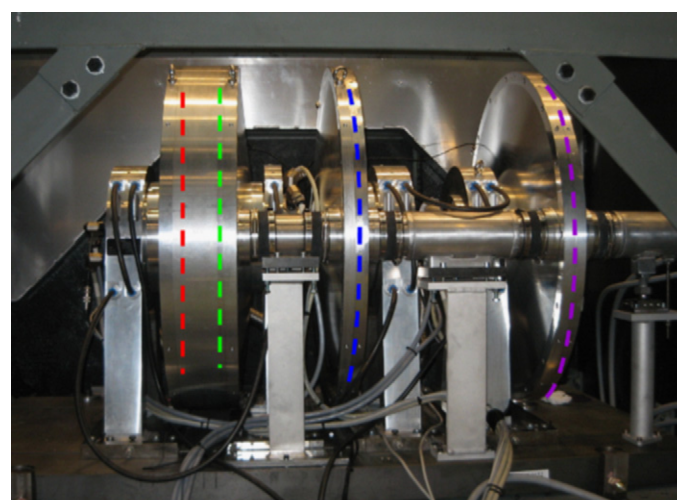

Fig. 2. The *Platypus* disc chopper system. Disc pairs 1 and 2, 1 and 3, or 1 and 4 are used to generate neutron pulses of wavelength resolution ($\Delta\lambda/\lambda$) 1.5%, 4% or 9%, respectively. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

Table 1
Specifications of EADS Astrium GmbH disc chopper system.

Specifications				
Disc outer diameter (mm)	700			
Window radial length (mm)	60			
Maximum speed (Hz)	83.33			
Relative phase variation (°)	< 0.1			
Disc absorber material	boron-10			
	Disc 1	Disc 2	Disc 3	Disc 4
Disc cut-out angle (°)	60	10	25	60
Distance from master disc (mm)		103	359	808
$\Delta\lambda/\lambda$[a] (for $D=7545$ mm)[b]		0.012	0.043	0.084

[a] Gaussian FWHM, calculated using the method of Van Well and Fredrikze [58].
[b] Standard instrument configuration. D can vary from 7520 to 8610 mm.