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Neutron Reflectometer with Polarization Option at the Budapest Neutron Centre

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Introduction

The ever increasing need for product advancement and miniaturization keeps thin film assemblies, membranes, magnetic and non-magnetic multilayer and patterned heterostructures in the limelight of materials science and technological development. A number of thin film and surface characterization methods have emerged recently to meet the new challenges. The increased interest in magnetic thin film analytical instruments – mainly triggered by the discovery of the giant magnetoresistance and related phenomena [1] – resulted in a boom of Polarized Neutron Reflectometry studies as well as in construction of a number of new neutron reflectometers with polarization option. This article reports on the design, construction and operation parameters and first example uses of the "Grazing Incidence Neutron Apparatus" (GINA) a recently installed neutron reflectometer at the Budapest Neutron Centre (BNC) in Hungary.

General overview of the instrument

The GINA reflectometer is a constant-energy angle-dispersive, vertical-sample instrument [2]. The setup is displayed in Figure 1 and the operation parameters are summarized in Table 1. The focusing graphite monochromator MONO provides neutrons with wavelengths within the range of 3.2Å and $\Delta\lambda/\lambda \approx 1\%$. The polarized neutron beam is produced by using a magnetized supermirror (SM) and an adiabatic radio-frequency (RF) spin flipper [3] (SF1). The beam scattered on the sample may undergo spin analysis by an identical setup of a spin flipper and a spin analyzer (P2), and finally it is detected by a two-dimensional position sensitive neutron detector (DET). The incident intensity is monitored by a low efficiency (~0.1% at $\lambda = 4.6$Å beam intensity monitor (IM). The components of the reflectometer are mounted on two heavy-load optical benches. The first one supports the beam shutter (BS), the IM, the beryllium filter (BF), the slit (S1) and the supermirror (SM) polarizer (P1) , the adiabatic RF spin flipper (SF1) and the slit (S2). The downstream end of the bench is fixed to the central sample tower ST and supports the various sample environment components (electromagnet, cryostat, etc.). The incident angle on the sample surface is set by the major (θ) goniometer of ST. The second bench – the 2θ-arm of the reflectometer – supports the slit (S3), the spin flipper (SF2), the spin analyzer (P2), and the detector along with its electronics and dedicated control PC mounted underneath. The slit (S4) in front of the detector is optionally used when data collection is restricted to specularly scattered neutrons. The 2θ-motion is driven by a wheel running on the marble surface while the corresponding air pads are activated. The wavelength may be changed by manually rotating the entire GINA setup around the turntable under the monochromator while air pads are activated and both arms float over the marble floor. At present, the available wavelengths are restricted to 3.2, 3.9,

Table 1. Operation parameters of the GINA reflectometer.

Parameter	Range
Wavelength	3.9 ÷ 5.7 Å in five steps
Present wavelength	4.6 Å
Max. scattering angle	$\geq\theta = 35°$
Angular resolution ($\Delta\theta$)	0.003°
$\Delta\lambda/\lambda$	≈1%
Background level	0.01 cps cm^{-2}
Detector	2D PSD, 200 × 200 mm^2
Detector spatial resolution	1.6 × 1.6 mm^2
Neutron flux at the monochromator position	$4 \times 10^5 n \times cm^{-2} \times s^{-1}$
Background reflectivity	$<7 \times 10^{-5}$
Overall polarization efficiency	0.895

4.6, 5.2 and 5.7 Å by the respective channels through the cylindrical concrete shielding (SH) around the monochromator assembly.

The monochromator MONO is located in a gap of the curved Ni/Ti SM guide 19 m downstream the cold source, and comprises five highly oriented pyrolytic graphite crystals on small motorized 2-circle cradles for horizontal alignment and vertical focusing. Vertical focusing of the beam to the sample position doubled the intensity reflected by a 20 × 20 mm^2 sample at grazing incidence as compared to the non-focused case of parallel graphite crystals. Higher harmonics intensity is efficiently filtered by a Be block. The transmission of the filter is 41% and 87% for $\lambda = 4.6$Å without and with liquid nitrogen cooling, respectively.

Polarized neutrons are produced by an Fe-Co/Si magnetic SM placed in an in-plane vertical magnetic field of 30 mT in transmission geometry (P1 in Figure 1). Spin analysis of the specularly reflected beam is performed by a single magnetic SM analyzer (P2) of identical construction with P1. In order to decrease the scattering of neutrons by the beam-line components, adiabatic RF spin flippers [3] are installed. The flipper coil is placed in a longitudinal gradient field of 20–40 mT/m, with a center field of 5.6 mT produced by two iron plates energized by permanent magnets upstream and shunted downstream. The flipper coil is part of a resonant circuit, with typical values of effective RF current and bandwidth of 4 A and 4.5 kHz at the resonance frequency of 166 kHz.

Fine definition of the beam is maintained by the four slits with cadmium blades. The blades are operated with a precision of 0.05 mm. Slit S1 defines the beam on the polarizer P1 to decrease the divergence thus increasing the polarization ratio. Slit S2 decreases the beam divergence on the sample and absorbs the neutrons scattered by the polarizer. With these optical elements, the setup exhibits a relative Q-resolution of 10% to 2% for the available Q-range of 0.005 to ~0.25$Å^{-1}$.

Scattered neutrons are registered by a delay line type multi-wire proportional chamber with active area of 200 × 200 mm^2 and spatial resolution of 1.6 mm (FWHM).

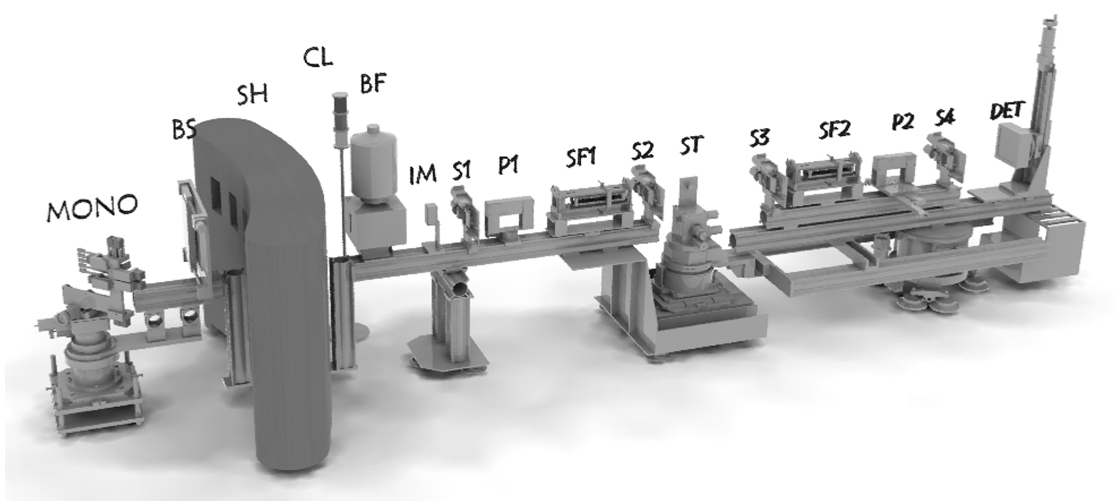

Figure 1. The layout of the GINA neutron reflectometer. The MONO assembly is mounted behind the concrete shielding (SH) on a turntable connected to the first optical bench supporting the beam shutter (BS), (latter monitored by semaphore control light [CL]), the beryllium filter (BF), the intensity monitor detector (IM), slit S1, polarizer P1, adiabatic RF spin flipper (SF1) and slit S2. The bench is connected to the sample tower (ST). The second optical bench is connected to the turntable underneath ST and supports the slit (S3), the second spin flipper (SF2), the spin analyzer P2 and optional slit S4 in front of the detector (DET).

The detector is filled with a ^{3}He / CF_4 gas mixture of 2.5/3 bar partial pressures and is encased in a boron-containing shielding for background suppression. A DASY TDC module (produced by ESRF, Grenoble) is installed in a slot of the PC dedicated exclusively to the detector data-acquisition and mounted on the 2θ-arm of the reflectometer. If no spin analysis is required, for further background suppression, an evacuated flight tube is mounted along the entire length of the 2θ-arm. Encasing the analyzer P2 and flipper SF2 in a vacuum vessel is a plan for the near future.

Measurement control

The GINA hardware and its control software are designed for maximum flexibility and remote controllability. In its full configuration, GINA comprises more than 30 stepping motors. All motions are remotely controllable. Certain critical motions, such as θ- and 2θ-angles and precision slit positions are absolute or relative encoder-controlled. Hardware control is maintained via a custom made unit built around a USB multi-function data acquisition module to control the air compressor, the air pads, the temperature of the Be-filter, the beam shutter and control lights, the beam intensity monitor and various modular DC power supplies. The high voltage power supplies, the linear amplifiers, the discriminators and the ratemeters are of NIM standard. The control PC directly communicates with the detector PC via Ethernet and with the indexer modules of the motion control units as well as with the temperature controller via RS232 under the supervision of GINASoft [4] written in LabView for MS Windows. The program user interface is highly configurable. It enables remote control of the experiments including alignments as well as control of polarization and the sample environment (flipper current and frequency, sample temperature, magnet current, etc.). Two-dimensional detector pictures and reduced reflectivity data are efficiently viewed and manipulated during the data acquisition. Collected data as well as extended log information are saved in a clearly structured database format. Human control is facilitated by a web camera. Using remote desktop option, most operations can be performed remotely via internet from outside the experimental hall or even from a distant continent.

Figure 2. Partial view of the GINA reflectometer. Components are marked as explained in Figure 1 apart from the sample environment with the electromagnet (MAG) with mounted closed-cycle cryostat. The magnet weight is partially relieved by the frame FR.

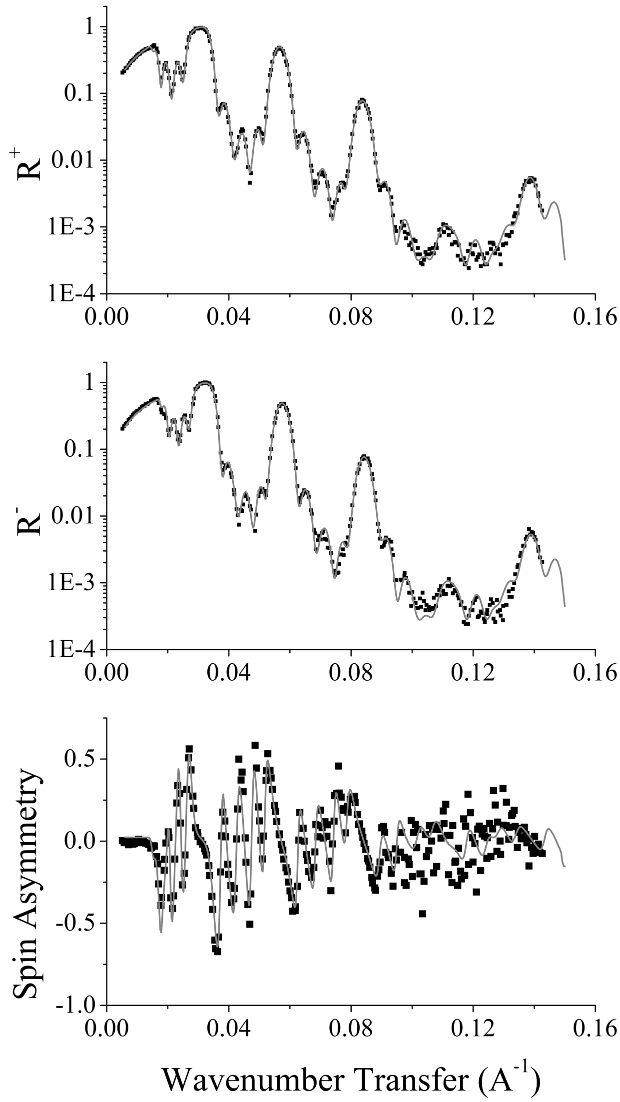

Figure 3. Measured R^+, R^- specular polarized reflectivities and the derived spin asymmetry *(R^+-R^-)/(R^++R^-)* on MgO(001)/[^{nat}Ni(15nm)/^{62}Ni(5nm)]$_5$. The statistical errors for most data points are smaller than the symbol size.

Sample environment

GINA is dedicated to reflectometry of magnetic heterostructures, for studies requiring different environmental parameters, such as (low) temperature and (occasionally high) external magnetic fields. Consequently, the sample mounting depends on the sample environment. For room-temperature reflectivity measurements, the sample is held in position by vacuum. Two cradles and two perpendicular translators position the sample in the vertical plane and set the sample surface orientation. A closed-cycle cryostat (12 to 300 K range) can be mounted on the sample tower ST with (*cf*. Figure 2) or without the electromagnet. At GINA an air-cooled electromagnet is available, which generates magnetic fields up to 0.55 T for the pole distance of 40 mm that accommodates the 1.5" diameter cryostat housing. The optional water-cooled air core coil pair provides fields up to approximately 35 mT.

Example reflectogram

The example in Figure 3 highlights the polarized specular reflectivity performance of GINA on a sample of a 20 × 20 mm^2 isotope-periodic multilayer prepared with a MgO(001)/[^{nat}Ni(15nm)/^{62}Ni(5nm)]$_5$ nominal layer structure by molecular beam epitaxy (MBE) [5]. Spin-dependent neutron reflectivities were recorded at room temperature. The sample was magnetized from the initial nonmagnetized (as prepared) state by 50 mT in-plane external magnetic field. The total data collection time was 56 hours. The measured R^+, R^- and the spin asymmetry are displayed in Figure 3. The *simultaneous* fit using FitSuite program [6] to the measured R^+ and R^- (solid lines in Figure 3) reveals (17.5 ± 0.5) nm and (5.35 ± 0.5) nm thickness for ^{nat}Ni and ^{62}Ni layers, respectively. The magnitude of the fitted scattering length densities SLD(^{nat}Ni) = (9.13 ± 0.5)·10^{-6} Å^{-2} and SLD(^{62}Ni) = (−7.01 ± 1)·10^{-6} Å^{-2}) show only minor deviations from their known bulk values. The magnetizations of the ^{nat}Ni and ^{62}Ni layers were constrained-identical and the fit provided (0.44 ± 0.12)T. This value amounts 66% of the known room temperature saturation magnetization M_S of the bulk Ni. According to the magnetooptical Kerr-loops the magnetization at 50 mT (the value of external field in the reflectivity measurement) is 74% of the saturation magnetization of the same sample. The lower value of 66% given by the reflectivity fit can be explained by a somewhat lower magnetization in the virgin magnetization state of the sample during the reflectivity experiment.

Summary

GINA is a versatile instrument: a dance-floor-type, vertical sample, constant energy angle-dispersive neutron reflectometer. Reflectivity ranges above four orders of magnitude have been measured. Further developments including an environmental cell for membrane studies, a supermirror fan analyzer and further background suppression elements will be installed within the next two years. The GINA reflectometer is open for Hungarian and international users throughout the year. Information concerning proposal submissions can be found at www.bnc.hu.

Acknowledgments

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