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AMOR – the time-of-flight neutron reflectometer at SINQ/PSI

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Abstract. The apparatus for multioptional reflectometry (AMOR) at SINQ/PSI is a versatile reflectometer operational in the time-of-flight (TOF) mode (in a wavelength range of 0.15 nm $< \lambda <$ 1.3 nm) as well as in the monochromatic (θ–2θ) mode with both polarized and unpolarized neutrons. AMOR is designed to perform reflectometry measurements in horizontal sample-plane geometry which allows studying both solid–liquid and liquid–liquid interfaces. A pulsed cold neutron beam from the end position of the neutron guide is produced by a dual-chopper system (side-by-side) having two windows at 180° and rotatable with a maximum frequency of 200 Hz. In the TOF mode, the chopper frequency, width of the gating window and the chopper–detector distance can be selected independently providing a wide range of q-resolution ($\Delta q/q$ = 1–10%). Remanent FeCoV/Ti : N supermirrors are used as polarizer/analyzer with a polarization efficiency of ∼97%. For the monochromatic wavelength mode, a Ni/Ti multilayer is used as a monochromator, giving ∼50% reflectivity at a wavelength of 0.47 nm. In the present work, a detailed description of the instrument and setting-up of the polarization option is described. Results from some of the recent studies with polarized neutrons and measurements on liquid surfaces are presented.

Keywords. Apparatus for multioptional reflectometry; neutron reflectivity; neutron reflectometer; time-of-flight; polarized neutrons; liquid interfaces.

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1. Introduction

In recent years X-ray and neutron reflectometry have been established as non-destructive microscopic probes for the investigation of stratified structures and hidden interfaces. Due to the unique interaction of neutrons with matter, specular neutron reflection probes the atomic and magnetic scattering length densities perpendicular to the sample surface. It can be very sensitive to electrochemical processes at interfaces as well as to surface and interface magnetism. Neutron scattering length density contrast among the isotopes of an element provides an opportunity for measuring self-interdiffusion in a multilayer stack. Moreover, polymer interfaces and biological model membrane systems can be studied using contrast variation. Growth, wetting, adsorption and adhesion, (self)-interdiffusion, surface magnetism, magnetic excitations, thin film superconductivity, dynamics at interfaces are all research fields applicable to neutron reflectometry.

An extremely flexible state of the art TOF reflectometer, AMOR has become now available at the Swiss neutron source SINQ at PSI [1–3]. AMOR is adaptable to the experimental demands of surface and interface studies in various fields of research. Full polarization analysed neutron reflectivity measurements, measurements of liquid samples using a fully automated Langumir trough and solid–liquid sample cell are recent developments at AMOR.

2. Basic instrument concept

The principal set-up of AMOR allows measurements with polarized or unpolarized neutrons in white beam TOF mode (0.15 nm $< \lambda <$ 1.3 nm) and optionally in unpolarized monochromatic θ–2θ mode. The latter mode is implemented with a thin film Ni/Ti multilayer monochromator consisting of 1500 layers with a bilayer period of 5.2 nm.

The plane of the sample is kept horizontal in order to allow measurements at open liquid surfaces, too. The inclination angle and thereby the accessible q-range is adjusted by tilting a deflection mirror and/or the sample. A flexible software control of the θ–2θ movement around the axes that are not mechanically coupled has been implemented. The standard mode of the instrument is TOF, which has been open for user operation since Oct. 2002. An area detector or two single detector tubes can be operated alternatively with new fast detector read-out electronics. Most optical components are riding on an 8 m optical bench so that the chopper–detector distance can be varied in order to give an optimum sample illumination and resolution (figure 1). In the following sections each of these parameters are described.

3. Chopper system

A cold neutron beam from the end position of the curved neutron guide ($m = 2$, where m corresponds to the critical edge of Ni) 1RNR17 with a flux of 1.38×10^8 n cm^{-2} s^{-1} mA^{-1} (averaged over the spectrum) hits a double chopper system defining the origin of the neutron burst of the instrument. The neutron guide has a cross-section of 5×5 cm^2. The spectral distribution obtained from the liquid D_2 moderator at SINQ offers a wide usable wavelength band which peaks at $\lambda_{\max} = 0.4$ nm. The two chopper discs have two slits at 180°. They are phase coupled with a maximum speed of 6000 rpm giving burst rates of 200 Hz. The distance between the chopper and the detector varies between 3.5 and 10 m. The chopper frequency, width of the gating window and the chopper–detector distance can be selected independently. Hence the resolution can be optimally tuned to the experimental needs in a range of $\Delta q/q = 1$–10%.

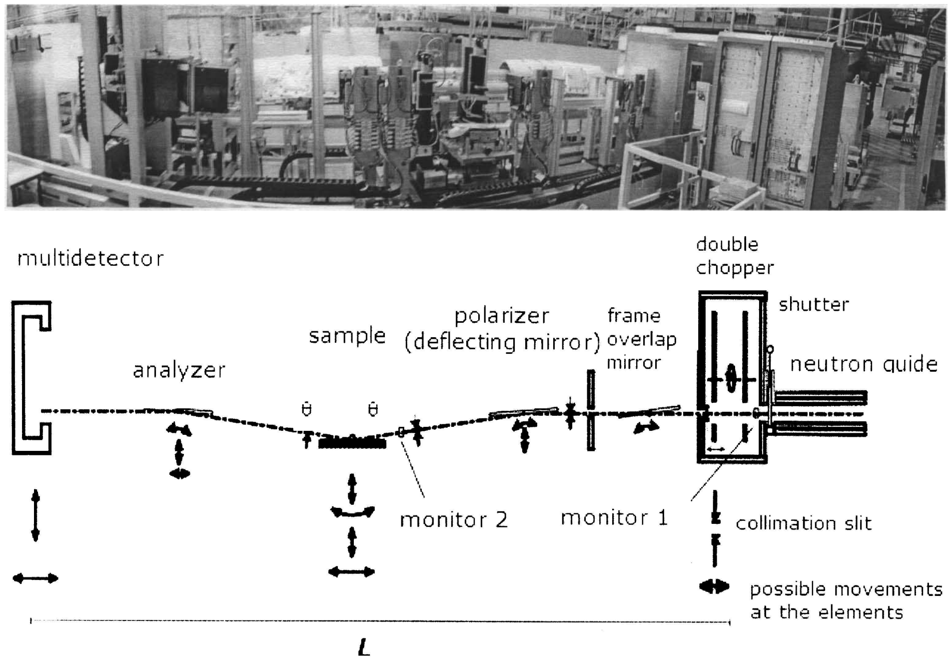

Figure 1. Reflectometer AMOR at SINQ/PSI and principal layout.

4. Optics

A frame overlap mirror consisting of a Si based Ni/Ti supermirror ($m = 2$) positioned behind the chopper system eliminates undesired neutrons with wavelengths larger than 1.3 nm. Following this position is a station that can be equipped by either a deflecting mirror of a highly oriented pyrrolytic graphite (HOPG) double monochromator to give a monochromatic beam of $\lambda = 0.4735$ nm or by a remanent polarizing FeCoV/Ti : N supermirror ($m = 2.6$) that can operate for a broad wavelength band of polarized neutrons. The performance of the polarizing mirror is shown in figure 2.

The sample stage to align the sample horizontally (vertical scattering geometry) is equipped with two rotation angles and a translation in z. Samples of size up to 150×500 mm^2 can be measured at ambient atmosphere when placed on a standard sample mount. Behind the sample stage, a reflecting mirror analogue to the deflecting polarizing mirror can be installed for the polarization analysis of the reflected beam. Between the frame overlap mirror and the sample, and the sample and the detector, various diaphragms can be placed in order to define the beam. To fulfil the requirements of experimental stability and vibration control, all devices are installed on the 8 m long solid optical bench, where they can be moved along the beam direction (see figure 1).

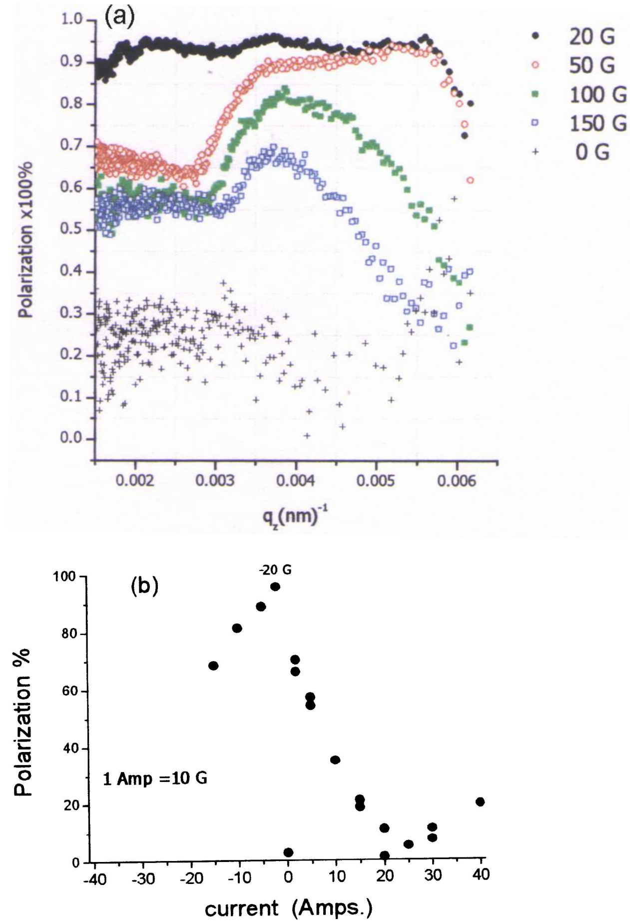

Figure 2. Variation of polarization as a function of q_z at various applied magnetic field at the polarizer at an angle of 0.5° (**a**) and variation of polarization at $q_z = 0.0036\ \text{nm}^{-1}$ (**b**).

5. Detectors

Two ^{3}He single detector tubes and one ^{3}He two-dimensional multi-wire position sensitive detector (PSD) are installed in AMOR. The EMBL 2D-PSD has an active area of 172 × 190 mm^2 with less than 2 mm spatial resolution. The obtained data either in monochromatic or in TOF mode are stored in ASCII or HDF5/NEXUS file format, respectively.

6. Sample environment

The flexibility of the instrument allows for large sample environments, such as cryomagnets, evaporation chambers, furnaces or Langmuir troughs. Currently a 1.8 T electromagnet with a horizontal field operational at room temperature, two

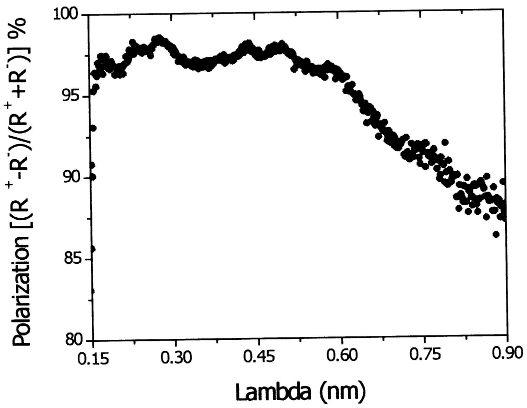

Figure 3. Variation of polarization as a function of reflected wavelength band at an incident angle of 0.5°.

Helmholtz coils for a small horizontal field up to 150 G applicable around larger sample stages, a 2 T cryomagnet with a horizontal field and a temperature range between 1.5 and 300 K, and a Langmuir trough for measurements at the air–liquid interphase, are provided as special sample environments on AMOR. A sample cell for measurements at the solid–liquid interface and a dedicated two-zone-heating vacuum furnace in the temperature range of 300–1100 K are also available.

7. Polarization set-up

AMOR is operational for full polarization analysed neutron reflectometry measurements since June 2003. Remanent FeCoV/Ti : N supermirrors [4] are used as polarizer/analyzer with a polarization efficiency of ∼97%. The polarization state of the neutron beam (either up $|+\rangle$ or down $|-\rangle$) is defined by applying a saturation field (+350G or −350G, respectively) for a short period of time.

During the setting-up of the polarizer it has been found that the polarization of the reflected beam from the polarizer is very sensitive to: (i) the angle of the polarizer towards the incoming beam and (ii) the guide field required to keep the polarization. Depending on the incoming wavelength band from the choppers (tuned with chopper speed, phase and distance between them), the polarizer has to be kept at a specific angle so as to reflect the complete incoming wavelength band. The guide field required to keep the polarization has been determined by performing detailed measurements of polarization by varying the magnetic field (guide field) around the polarizer. From figure 2 it can be seen that a field higher (or lower) than ∼20 G starts depolarization of the beam as the thin layers of the supermirror starts switching with the field. Figure 3 shows the variation of polarization as a function of reflected wavelengths at an angle of 0.5°. Up to a wavelength of 0.6 nm polarization remains almost constant to a value of ∼97% and for the higher wavelengths the polarization decreases down to 87% for a wavelength of 0.9 nm.

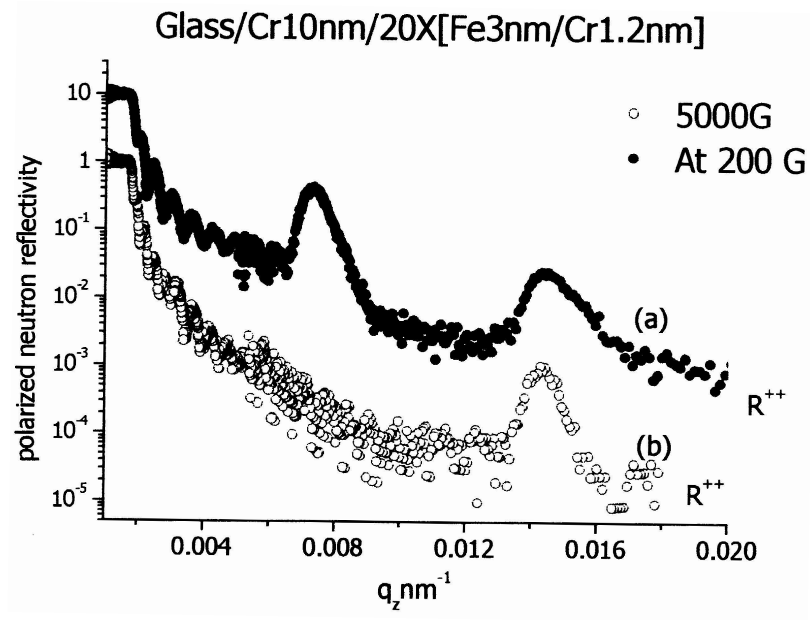

Figure 4. Polarized neutron reflectivity of Fe/Cr multilayer at a saturation field of 5000 G and a remanent field of 200 G. At remanence the magnetic peak due to AF coupling can be seen clearly at double periodicity around $q_z = 0.007\ \text{nm}^{-1}$. The upper curve has been shifted by a factor of 10 for clarity.

8. Recent results

Figures 4 and 5 show representative results of polarized neutron reflectivity on Fe/Cr multilayers and that of liquid D_2O respectively. In the case of Fe/Cr multilayer, the measurements have been done at a saturation field of 5000 G and at a remanence field of 200 G. It is well-known that Fe/Cr multilayers exhibit strong anti-ferromagnetic (AF) coupling [5], when exposed to the saturation field where all the spins are aligned ferromagnetically. As can be seen in figure 4b, the AF peak, which is clearly observed at remanence, disappears completely at saturation field.

Figure 5 shows the reflectivity of a D_2O surface measured at the Langmuir trough. As can be seen, the scattering length density from the current measurement matches well with the theoretical values over four orders in dynamic range, within a reasonable time of 2–3 h for the measurement.

9. Conclusions

As can be seen from the present work, the neutron reflectometer AMOR offers extremely flexible opportunities to measure all kinds of multilayered structures,

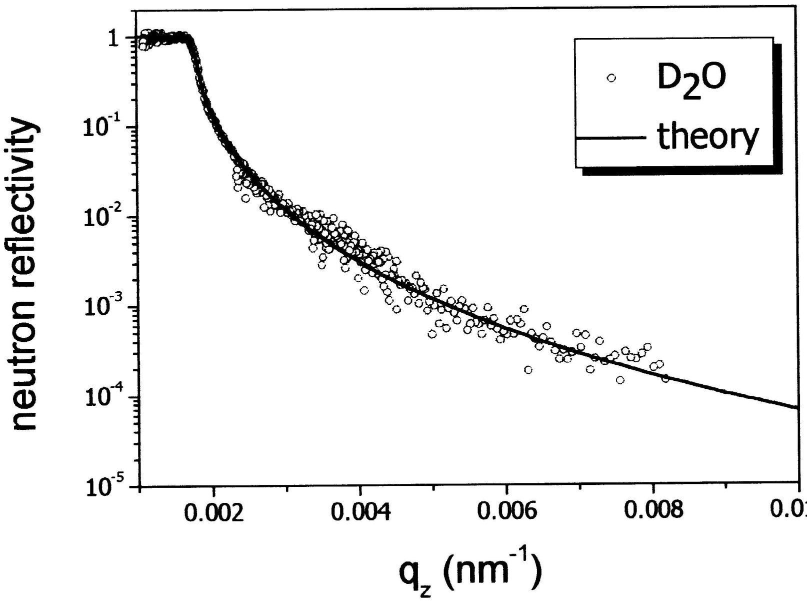

Figure 5. Reflectivity of liquid D_2O at air/water interface at room temperature.

magnetic or non-magnetic, to study various phenomena, e.g. adsorption, wetting and de-wetting, diffusion etc. With the available wavelength range of 0.15 nm $< \lambda <$ 1.3 nm, and with TOF, q range up to 0.02 nm^{-1} can be obtained relatively fast with only 2-3 angular settings. As AMOR is designed to perform reflectometry measurements in horizontal sample-plane geometry, it allows studying both solid–liquid and liquid–liquid interfaces. With the feasibility of selecting the chopper frequency, width of the gating window and the chopper–detector distance a wide range of q-resolution $\Delta q/q$ = 1–10% can be selected according to experimental requirements.

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