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A New Polarized Neutron Reflectometer at KENS

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Abstract

A new polarized neutron reflectometer has been installed at KENS (Neutron Science Laboratory in High Energy Accelerator Research Organization) in Tsukuba. The reflectometer is designed for investigation of surface or interfacial magnetisms of thin films and multilayers. It has an analyzer for polarization analysis and an optional detector bank for off-specular reflectivity measurements which consists of ^{3}He squashed detectors. The overview and the status of the reflectometer are reported.

1. Introduction

The TOP spectrometer at KENS had been built to study mesoscopic magnetism, and was a multipurpose spectrometer which used cold polarized pulsed neutrons [1]. Various experiments, depolarization, polarized neutron reflection and small angle scattering had been done on TOP. During 1996 - 1997 the cold neutron experimental hall at KENS has been rebuilt, and the C1 cold neutron beam port was newly assigned to TOP [2]. However, the C1 port has to be shared with a Small/Wide-angle neutron diffractometer, SWAN, so that TOP could have no room for a detector bank for small angle scattering measurements. Polarized neutron reflection is a powerful tool for the investigation of magnetic thin films and multilayers because using polarized neutrons we can detect weak magnetic moments very accurately [3]. Thus, instead of TOP, a new polarized neutron reflectometer, PORE, has been designed and constructed on the basis of a lot of experience of polarized neutron experiments on TOP.

Neutron reflectivity measurements generally give the information about the depth profile of layer structure including magnetism. In addition to this, off-specular reflection, whose importance many researchers begin to recognize recently, gives the information on surface and interface roughness in thin films and multilayers [4]. The plan of construction of the reflectometer has two stages, Phase 1 (standard measurements of specular reflectivities) and Phase II (optional measurements of off - specular reflectivities). The Phase I has been almost completed. On the other hand the preliminary experiments of phase II is now progressing.

2. Overview

The principal specifications of the reflectometer, PORE, are listed below and the plan view is shown in Fig.1.

Specification

• Beam port	C1 - 1 (50 Hz)
• Moderator	Solid Methane
• L1 (Moderator - Sample)	9.5 (m)
• L2 (Sample - Detector)	1.5 (m)
• Incident Polarized Neutron wavelength	$3 < \lambda < 16$ Å
• Scattering angle	$2° < 2\theta < 22.5°$
• $Q_{//}$ normal to the surface	$0.015 < Q_{//} < 0.7$ ($Å^{-1}$)
(specular reflection) Resolution	$(\Delta Q_{//}) / Q_{// min} > 0.03$
• Q_n parallel to the surface	$0 < Q_n < 0.05$ ($Å^{-1}$)
(off - specular reflection) Resolution	$(\Delta Q_n) / Q_n < 0.02$
• Beam size	30 mm (H) × 0.1 to 10 mm (V)
• Detector	Single ^{3}He gas detector for specular reflection
and	35 squashed ^{3}He gas detectors for off - specular reflection
• External magnetic fields	$\mu_0 H < 1$ (T)
• Temperature	$10 < T < 350$ (K)

The reflectometer utilizes cold neutron wavelength band of 3 - 16 Å from the solid methane cryogenic moderator. The wide band of incident neutrons makes it possible to cover wide range of momentum transfer value, Q, at the fixed incident angle. This is a typical advantage of reflectivity measurement using white beam. Incident unpolarized white neutrons are polarized by a optical device which consists of Fe/Si polarizing supermirrors (OSMIC) [5]. The polarizer also works as the frame overlap mirror. The beam polarizations are slightly changed by the collimation of

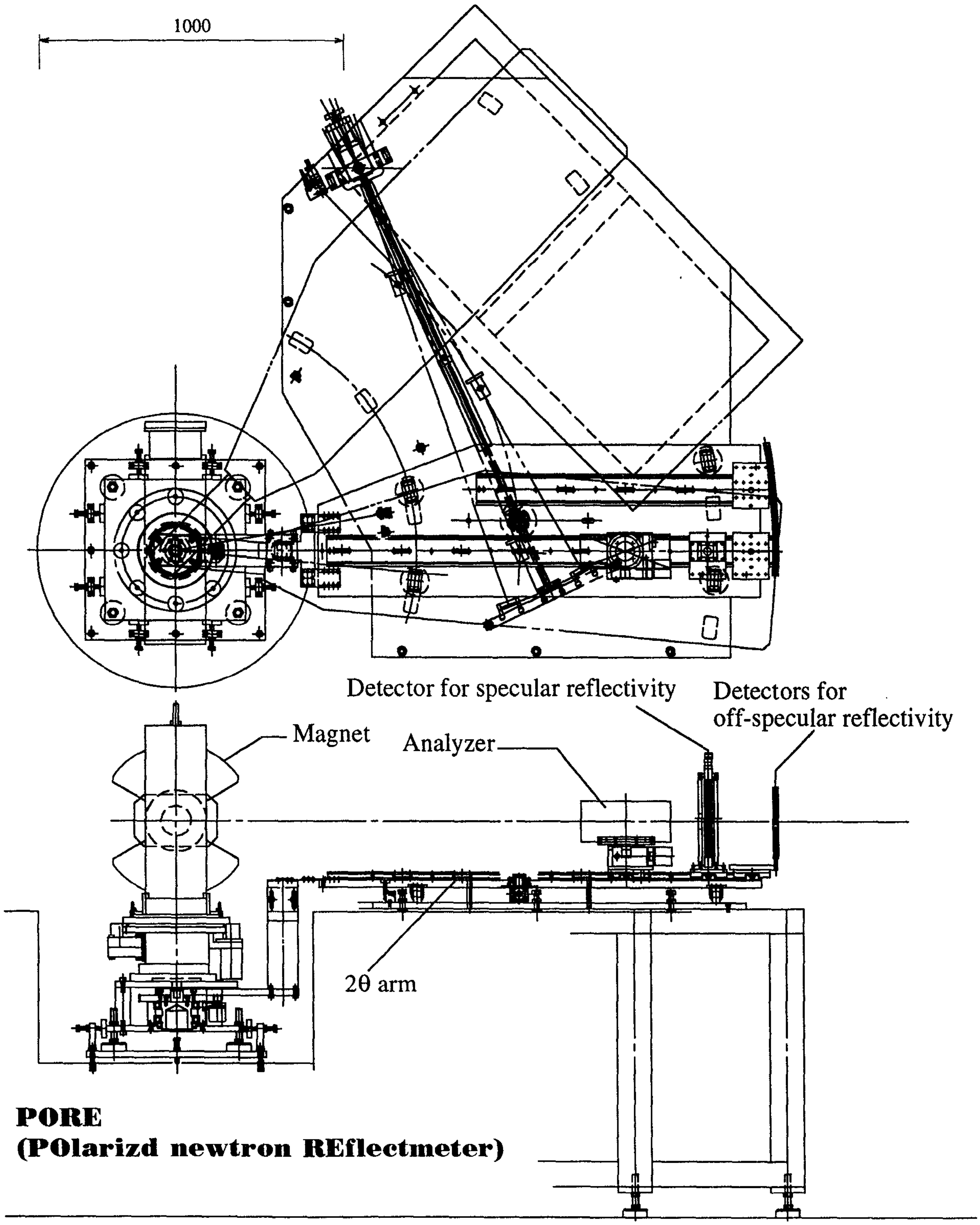

Figure 1 A plan view of the new reflectometer, PORE at KENS.
A polarizer, a flipper and a detector bank for off-specular measuremenst are not shown in this figure.

incident beam, however, keep constant value (> 92 %) for neutrons within the band. The direction of polarization can be reversed by a two coil flipper with almost perfect flipping efficiency before samples. In the normal operation neutron spins are flipped every 4 seconds. The polarized neutrons are collimated by two pairs of horizontal slits and collimator assembled by SUS306, B4C and Cd. At the sample position, beam is horizontally separated into two beams for PORE and SWAN, respectively, whose separation is only 40mm. Samples are vertically set to the sample position on a θ gonio stage from the geometry of beam sharing with SWAN. Therefore the scattering plane is always in the horizontal plane.

The reflection angle can be changed from -5 to 45 (deg.) by moving a 2θ arm driven by a stepping motor. The reflected neutrons from the samples are detected by a time analyzer and single 1"-^{3}He detector for the specular reflection measurements, and by 35 squashed ^{3}He detectors for the off-specular ones. Deficient 15 detectors are going to be added to the bank in the near future. The data are stored by a hard disk on a personal computer (Power Macintosh 7600/200, Apple) which controls the slits, polarizer, analyzer, incident angle and reflection angle by GPIB. The computer also controls the data taking system using a package software, LabVIEW, sold by NATIONAL INSTRUMENTS [6]. During measurements intensities of incident neutrons are monitored by a fission neutron monitor.

3. Examples

3.1 Phase I (Specular reflectivity)

Figure 2 shows the unpolarized neutron specular reflectivities of 2 - vinylpyridine-styrene-d8-2-vinylpyridine triblock copolymer (PDP) [7] measured by PORE. In the same figure data of the same sample taken by the CRISP spectrometer are plotted by open circle. CRISP is a neutron reflectometer which uses a wavelength band, from 0.5 to 6.5 Å, at Rutherford Appleton Laboratory pulsed neutron source ISIS in UK. ISIS is the world's brightest spallation neutron source, and the power of injected proton beam to neutron target is 160 kW . On the other hand, the power of KENS is only 3 kW. Although the power is not directly proportional to luminosity of neutron beam, intensity ratio is estimated to be at least 20.

In both measurements, the wide Q range was covered by changing the incident angle, ie. 0.6 and 1.5 (deg.), however, the covered Q ranges were not the same between these two spectrometers because of the difference of utilized wavelength band. Incident angle of 5 (deg.) was also used on PORE to cover higher Q region. In order to compare the data with the same instrumental resolution, $(\Delta\theta/\theta)$ was kept the same value, 0.07 between two spectrometers. It took 1.5 hours to collect the data on CRISP and 5 hours on PORE typically. Reflectivities in the Qregion higher than 0.3 Å^{-1} have been buried under background noise on PORE. The figure shows that PORE is capable of measuring reflectivities down to values at least in the region of

10^{-5} with the same accuracy of CRISP.

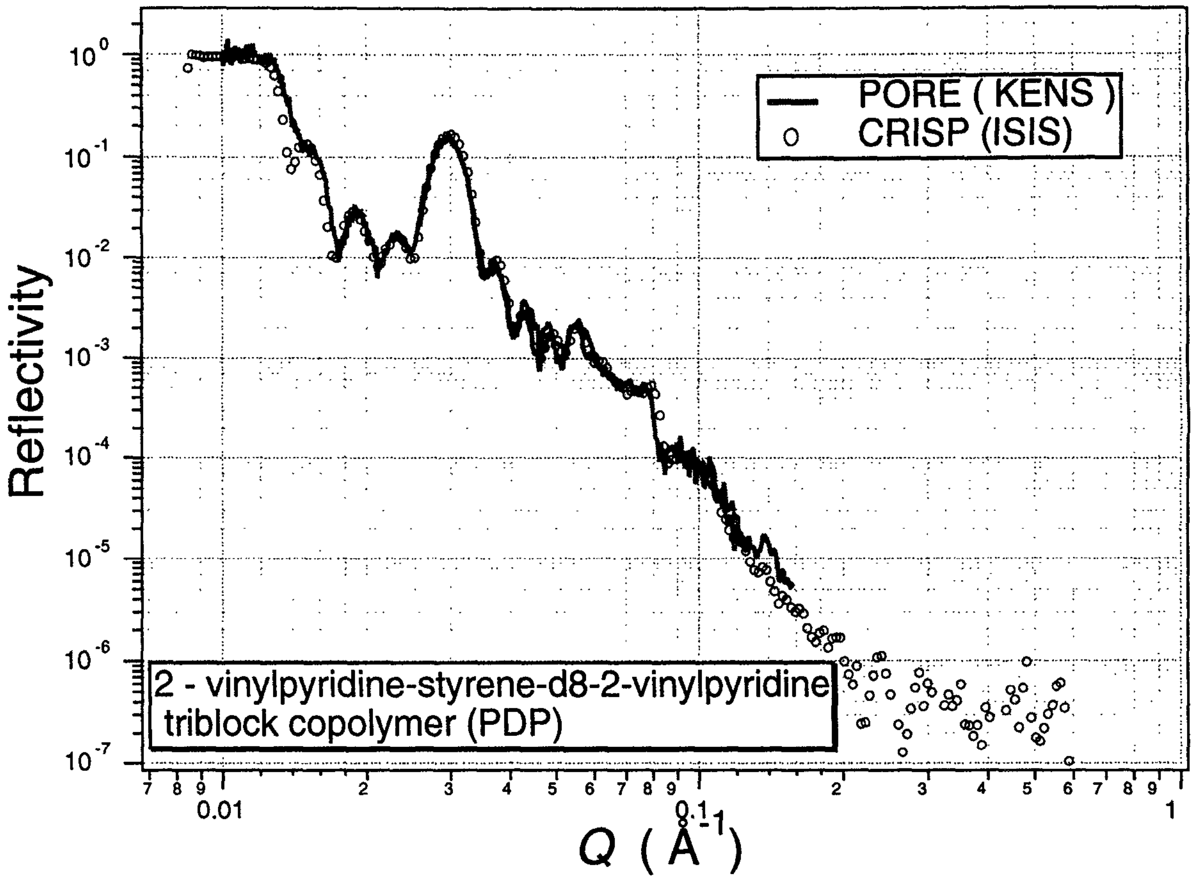

Figure 2 Reflectivities of a PDP triblock copolymer film on Si.

Polarized neutron specular reflectivity curves of natural Ni mirror with thickness of 2000 Å are displayed in Fig. 3 (a) and (b) with error bars. An external magnetic field of 40 Oe was applied in the mirror to maintain the polarization of neutrons. The total reflection of neutrons appeared in the lower Q region and clear fringes can be seen in the vicinity of the total reflection. In Fig. 3 (a), "Spin Flipper Off" means that spins of incident polarized neutrons are parallel to the magnetization of Ni mirror, and "On" in Fig. 3 (b) corresponds to antiparallel. The weak magnetic moments of Ni make the difference between these two data. The difference can be more clearly seen by flipping ratio, $I_{\mathrm{off}} / I_{\mathrm{on}}$ (Fig. 4). Here I_{on} is the intensity in the case of flipper "ON" and I_{off} is that in the case of "Off". This figure shows the flipping ratio is very sensitive to weak magnetic moments.

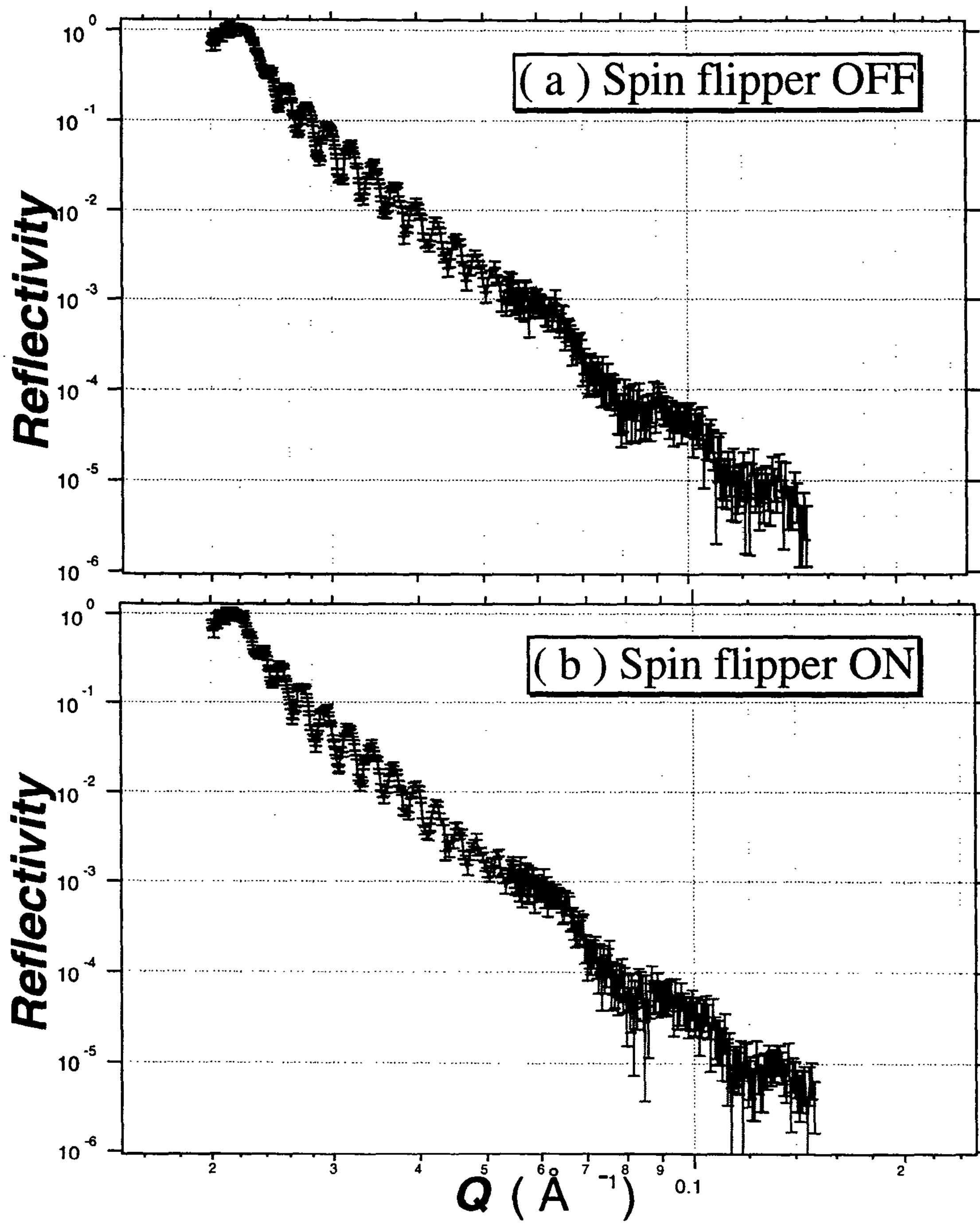

Figure 3: Polarized neutron reflectivities of a Ni mirror with thickness of 2000 Å, (a) spin flipper OFF and (b) ON

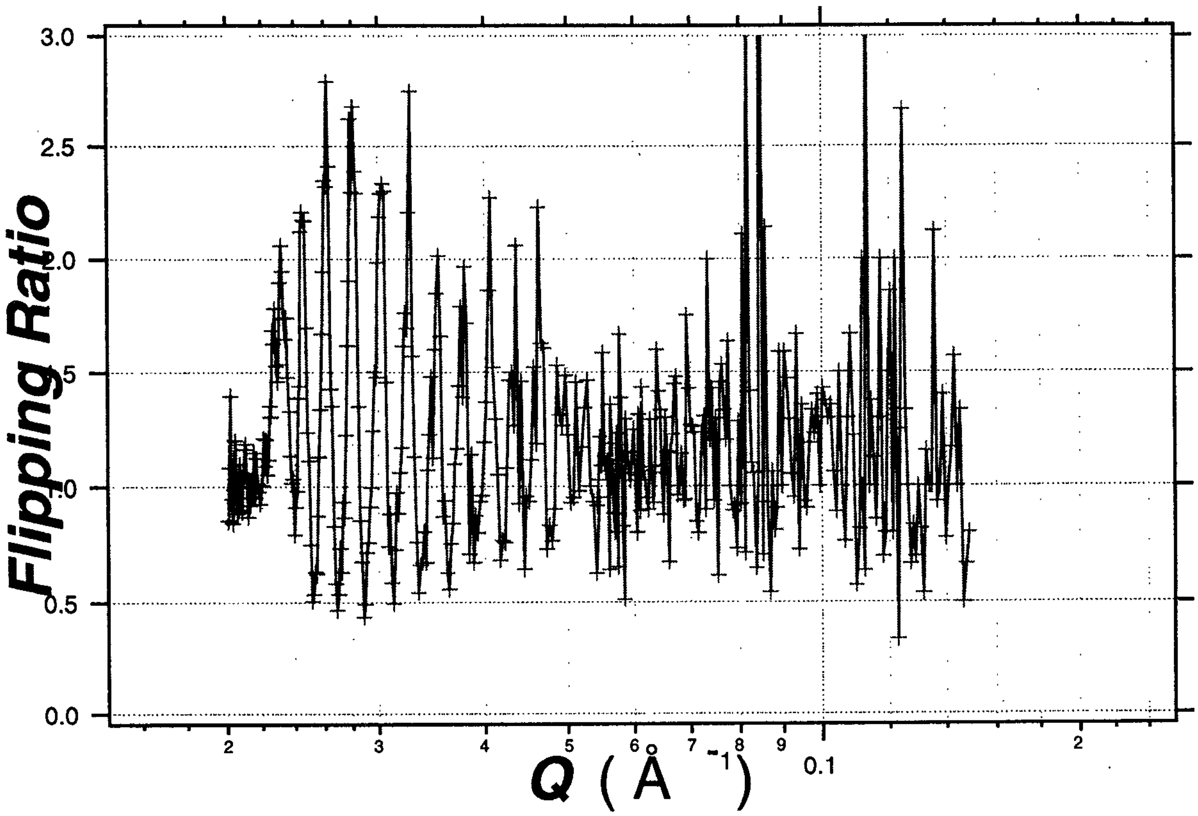

Figure 4: Flipping ratio deduced from Fig. 3

3.2 Phase II (Off - specular reflectivity)

Setup for the off-specular reflectivity measurements is now final stage. Almost all components have been installed and we started in doing preliminary measurements. Figure 5 shows one of the results of off-specular reflection of a Ni/Ti multilayer [Ni(55Å)/Ti(44Å)] × 15 on MgO [8] with flipper bing on (a) and off (b) under the field of 40 Oe. This contour map of intensities of reflected neutrons was collected by the optional detector bank for off-specular reflection measurement in which the 35 squashed detector with 4 mm in width by 100 mm in hight are lined up on arc.

In the contour map the horizontal axis is the number of TOF channel corresponding to wavelength of neutrons, and the vertical axis is the number of detectors corresponding to reflection angle. In this case the detector 0 corresponds to specular condition, ie. the incident angle of 1.5 (deg.) is equal to the reflection angle, and the reflection angle increases with increasing the detector number. Intensities under the condition that incident angles are different from reflection angles are that of off-specular reflection. Bragg peak from Ni/Ti bilayer with thickness of 99 Å

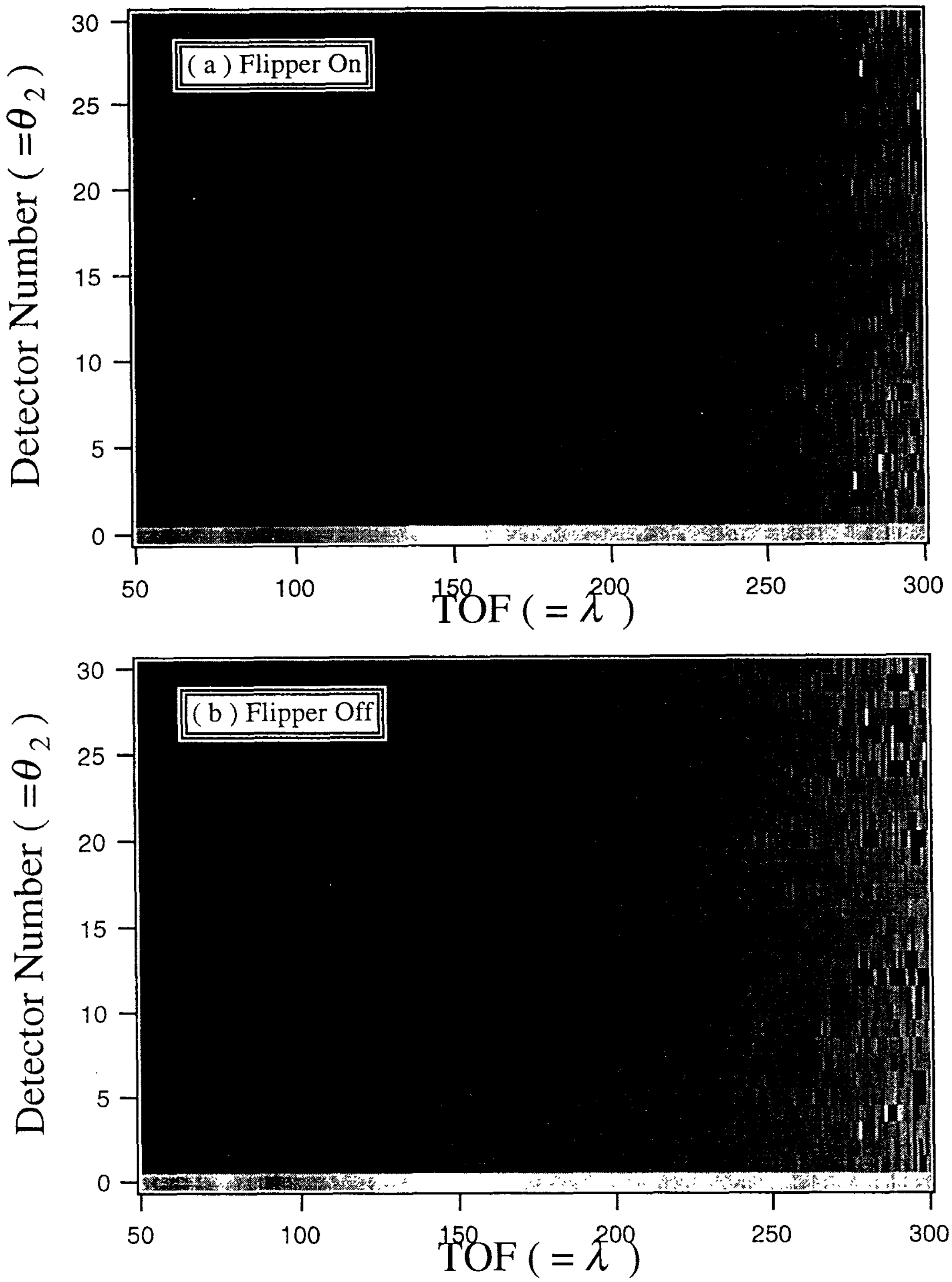

Figure 5 Contour map of intensities in gray scale. The vertical axis corresponds to reflection angle and the horizontal one to wavelength of neutrons. Detector 0 holds specular condition.

appears at around position (150, 0) both in Fig. 5 (a) and (b). Clear two streaks starting at point (150, 0) are observed in these two figures in the different way although a weak magnetic field was applied. One of the streaks which is not parallel to the vertical axis expresses the correlated interfacial roughness from the bottom to the topmost layer [4]. This indicates that the PORE reflectometer is very promising tools for the investigation of surface and interface roughness with magnetic origin with appropriate theory.

4. Concluding Remarks

The new polarized neutron reflectometer, PORE, at KENS works excellently for the specular reflection measurements and is ready to accept user proposals. However, the background in reflectivity is not well below 10^{-6}. If reflectivities down to 10^{-6} have to be measured, it is necessary to reduce the background noise by much better way. The off-specular reflection measurement is expected to be open to users within this year.

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References

[1] S. Itoh, T. Watanabe and Y. Endoh, Proc. of ICANS-XI ,Tsukuba (1990) 792.

[2] KENS REPORT - XI (1997) .

[3] H. Zabel and A. Schreyer, Neutron News **9** Issue 2 (1998) 18.

[4] S. K. Sinha, Physica B **173** (1991) 25.

[5] M. Takeda, K. Kurahashi, Y. Endoh and S. Itoh, Supplement A to J. Phys. Soc. Jpn. **65** (1996) 234.

[6] S.Satoh and M.Furusaka, Proc of DANEF'97, Kubuna (1997) 92.

[7] Y. Matsushita, J. Suzuki, N. Takabayashi, N. Torikai, M. Nomura and I. Noda, Macromolecules **31** (1998) 2378.

[8] P Böni, I. S. Anderson, R. Hauert, P. Ruterana, K. Solt, B. Farnoux, G. J. Herdman, J. Penfold and O. Schärpf, Proc. of ICANS-XI ,Tsukuba (1990) 910.