

Neutron Reflectometer with Polarization Analysis (HADAS)



Instrument Parameters

Monochromator:	(002) pyrolytic graphite double monochromator
Wavelength:	$2 \text{ \AA} < \lambda < 6.5 \text{ \AA}$, $\lambda = 4.52 \text{ \AA}$ for reflectometry
Wavelength resolution:	1 %
Collimation:	$0.1 \text{ mrad} < \Delta\Theta < 10 \text{ mrad}$ (unpolarized beam), $0.1 \text{ mrad} < \Delta\Theta < 1.5 \text{ mrad}$ (polarized beam)
Polarization analysis:	Stack of 30 double sided polarizing supermirrors (top $m = 2.5$, bottom $m = 1.8$) set up as divergent collimator
Detector:	2D position-sensitive scintillation detector, 80 mm diameter, resolution $< 1.5 \text{ mm}$, at 1.6 m distance from the sample
Neutron flux at sample:	10^5 unpolarized neutrons /cm ² s with a collimation of $\Delta\Theta = 2 \text{ mrad}$, 2×10^4 polarized neutrons /cm ² s with a collimation of $\Delta\Theta = 1 \text{ mrad}$
Maximal beam size at sample:	50 mm $\times$ 10 mm (unpolarized), 20 mm $\times$ 10 mm (polarized with polarisation analysis)
Special sample environment:	Electromagnet $0.3 \text{ mT} < \mu_0 H < 1 \text{ T}$; He flow cryostat $10 \text{ K} < T < 300 \text{ K}$

Instrument Responsible

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Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	HAD-04-007		
Experiment title:	Magnetization reversal and depth profiling of exchange biased Co/CoO multilayers by polarized neutron reflectivity		
Dates of experiment:	17.06.04	Date of report:	February 21, 2006
Experimental team:	Names: Amitesh Paul, Ulrich Ruecker, Emmanuel Kentzinger, Thomas Brueckel		
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Local contact:	Amitesh Paul		

A reduction of the H_{EB} is seen in most thin-film systems after the first cycle of hysteresis loop for the subsequent reversal of FM magnetization [1]. Usually along with decrease in H_{EB} values a decrease in the coercivity is also noticed. A rearrangement of the AF domain structure has been suggested to be connected with the loss of magnetization in the AF and thereby the magnitude of H_{EB} . A connection of training effects to the microstructure is predicted [2] which was supported by Nowak *et al* [3] in their domain state model with nonmagnetic defects. However, presence [4]/absence [5] of training effect in some epitaxial systems with uniaxial/biaxial magnetic anisotropy rule out the connection of training effect with microstructure. Hoffmann [6] had shown by numerical simulations that the symmetry of magnetic anisotropy in antiferromagnet is crucial to understand training effects. An imbalance between two AF sublattice magnetization could easily explain the training effect in uniaxial as well as biaxial systems. Therefore it also supported the observed presence of training effects in polycrystalline samples which are expected to be more common with multiple equivalent easy magnetization axis. Polycrystallinity on the other hand limits the long range order in the AF layer which is restricted by the presence of grain boundaries and favors domain formation.

In the present report, we investigate the layer-by-layer evolution of the magnetization configuration of the same CoO/Co polycrystalline ML along a full magnetization loop by specular and off-specular Polarized Neutron Reflectometry (PNR) for a trained state and compare it with that of the untrained one. The field cooling axis and the reflectometer axis were the same. The reversal proceeds for both loop branches in a nonuniform mode for both cycles of measurements. All four specular reflectivity channels are measured at 15 different H_a , six of which are shown in Figure 1, together with least-square fits based on an extension of the Parratt formalism to magnetic MLs.

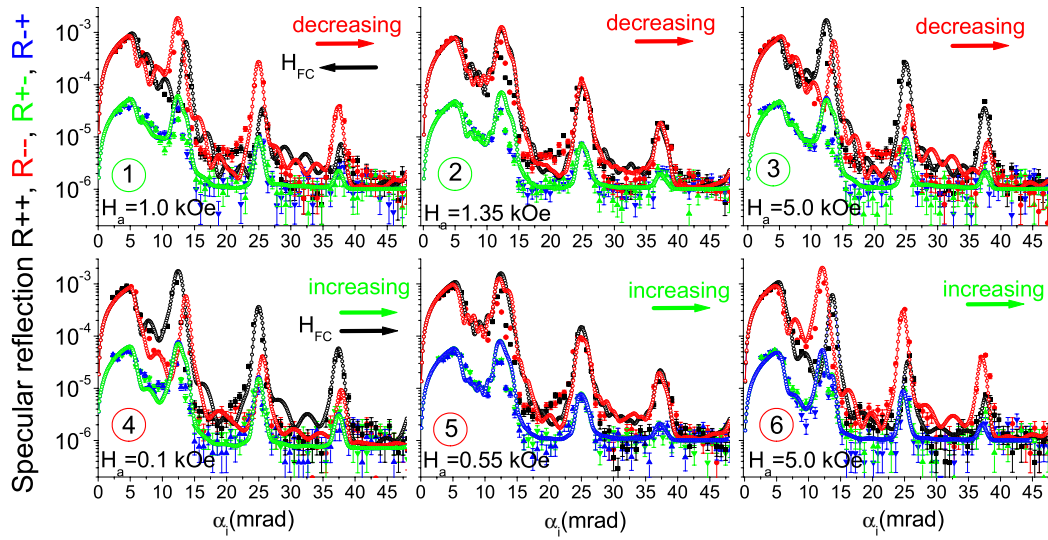


Figure 1. Measured and fitted NSF (R_{++} and R_{--}) and SF (R_{+-} and R_{-+}) reflectivity patterns of *continuous* $\text{SiO}_2/\text{Co}(11.0 \text{ nm})/\text{CoO}(7.0 \text{ nm})]_{\times 20}/\text{Au}(50.0 \text{ nm})$ ML in the *trained state* at different applied fields H_a along increasing and decreasing fields where $H_{FC} = -4.0 \text{ kOe}$.

This result indicates that the FM layer domains are always parallel or antiparallel to the applied field direction whether they are in the first or subsequent field cycles. Such stable configuration of the FM magnetization is achieved possibly due to the existence of multiple AF-FM interfaces in our ML which therefore satisfies the conditions for perpendicular configuration of the AF sublattices. However, under such a condition, the system is not expected to show any training effect either, as this would indicate the absence of rearrangements of AF magnetization after the first cycle. Our ML on the contrary, do show the existence of training effect as it clearly shows the decrease in H_c value. In case of a broken perpendicular arrangement, we would have expected the FM magnetization reversal only by rotation following the predictions of Hoffmann [6]. Thus we can say that in spite of the initial magnetization rearrangements after the first cycle, the system retained the perpendicular arrangement in the second cycle. Alternatively, we can say that broken perpendicular arrangement has not been instrumental in deciding over the magnetization reversal mechanism in subsequent cycles.

References

- [1] J. Keller *et al.*, Phys. Rev. B **66**, 14431 (2002).
- [2] J. Nogues *et al.*, J. Magn. Magn. Mater. B **192**, 203 (1999).
- [3] U. Nowak *et al.*, Phys. Rev. B **66**, 14430 (2002).
- [4] L. Malkiniski *et al.*, Journ. Appl. Phys **93**, 6835 (2003).
- [5] M. R. Fitzsimmons *et al.*, Phys. Rev. Lett. **84**, 3986 (2000).
- [6] A. Hoffmann, Phys. Rev. Lett. **93**, 97203 (2004).

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Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	HAD-04-010		
Experiment title:	Investigating magnetization reversal in polycrystalline IrMn/CoFe multilayer w.r.t. field cooling and measuring field directions		
Dates of experiment:	12.10.04	Date of report:	February 21, 2006
Experimental team:			
Names:	Amitesh Paul, Ulrich Ruecker, Emmanuel Kentzinger, Thomas Brueckel		
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The shift in hysteresis loop is given by an exchange bias field which is crucial for designing thin film magnetic sensors. The magnetization reversal in such systems is complex and are subject to recent studies. Asymmetric hysteresis loops due to different magnetization reversal process in different branches of the hysteresis loop are common [1, 2, 3, 4]. Theoretically the interpretation of the magnetization reversal was discussed in Ref. [5]. This is governed by an effective field H_{eff} arising from the anisotropy of the FM, the exchange bias field of the AF, and the applied field H_a . H_{eff} and the torque it exerts on the FM magnetization depend on the angle θ between H_a and the AF anisotropy axis or the field cooling axis H_{FC} . Beckmann *et. al* showed that depending on θ the reversal mode is either by coherent rotation (uniform reversal mode) for both loop branches or asymmetric with a nonuniform reversal for the increasing branch. Here nonuniform refer to the reversal of magnetization with no component perpendicular to the H_a direction which is essentially reversal by domain wall motion.

In the present report, we investigate the same polycrystalline IrMn/CoFe sample for different angles, $\theta = 15^\circ$, 45° and 90° . We measure specular and off-specular Polarized Neutron Scattering from the specimen along each full magnetization loop: increasing (negative to positive) and decreasing (positive to negative) field sweeping direction of the H_a for the hysteresis loop with respect to the direction of H_{FC} . For $\theta = 15^\circ$, we observe a crossover from sudden flipping of each layer magnetization to coherently rotating layers along the stack for both loop branches as we perform this layer-by-layer magnetometry. However, for $\theta = 45^\circ$ and 90° the reversal proceeds symmetrically and coherently in an uniform mode. The reversal is accompanied by fluctuations of the in-plane magnetization component perpendicular to H_a as observed earlier [6] irrespective of θ .

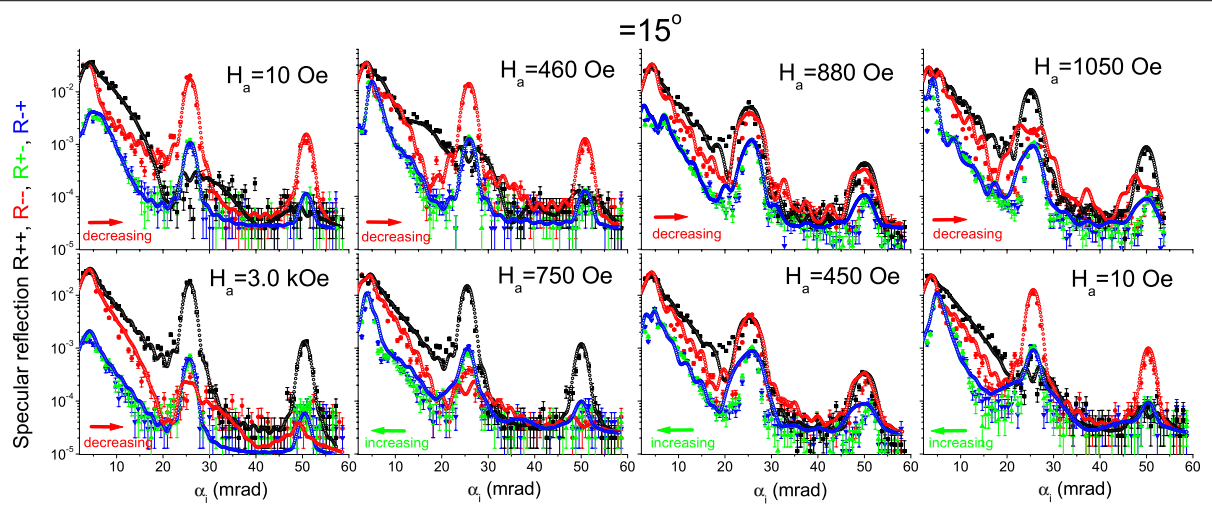


Figure 1. NSF and SF specular reflectivity patterns (solid symbols) along with their fits (open circle) for $[\text{Ir}_{20}\text{Mn}_{80}(6.0 \text{ nm})/\text{Co}_{80}\text{Fe}_{20}(3.0 \text{ nm})]_{10}$ ML with $\theta=15^\circ$ along increasing and decreasing fields.

The details of the fitting procedure have been described earlier [6]. For $\theta=15^\circ$, each layer rotates or flips independently and *sequentially* with field strength, fanning around in different directions in a certain field which shows the layer resolved magnetization direction with respect to the H_a direction. On the decreasing field branch: the bottom layers start flipping first followed by the top layers as we increase the field strength. On the increasing branch: the top layers rotate in different directions while the bottom layers flip back. The flipping and rotation of layers are seen by the presence/absence of SF signals of different intensities as we sweep the field strength on both branches of the loop. These observations are understood in general terms of the torque exerted on the system due to different effective field strengths and the corresponding angles with the magnetization axis. We systematically varied the relative strengths of the exchange and uniaxial anisotropies in a single multilayered system which is particularly possible due to *sequential* switching of layers with increasing N . All reversal modes are accompanied by small scale fluctuations of the perpendicular component of the in-plane magnetization on a length scale comparable to the grain size thus indicating the domain to domain fluctuations in the system. Our measurements show a gradual crossover from layer flipping (domain wall motion) for low torque regimes to coherent rotation for high torque regimes. Our experimental findings finally unravel the mysteries of magnetization reversal of exchange coupled systems and explain earlier observations of asymmetric and symmetric hysteresis loops.

References

- [1] W.-T. Lee *et al.*, Phys. Rev. B **65**, 224417 (2002).
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- [4] F. Radu *et al.*, Phys. Rev. B **67**, 134409 (2003).
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Experimental Report of Neutron Scattering Experiments at the FRJ-2 Reactor

Proposal number:	HAD-04-014		
Experiment title:	Magnetization reversal of a combination of narrow and wide magnetic stripes		
Dates of experiment:	17.-24.11.2004 10.-14.03.2005	Date of report:	19.06.2006
Experimental team: Names	Addresses		
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Local Contact:	Ulrich Rücker		

Experimental report text body

We performed polarized neutron reflectivity measurements on a periodic array consisting of alternating wide and narrow Fe bar. The bars have a width of 0.9 and 1.9 μm , a length of 30 μm and a height of 90 nm. They are arranged in lines as can be see in Fig. 1. The distance between the lines of bars is 10 μm and the distance between adjacent bars is 1.6 μm .

Longitudinal MOKE hysteresis loops (Fig. 2) were performed at different angles of sample rotation with respect to the applied field. Measurements at easy axis (field applied along the ridge of the bars) and at 45°

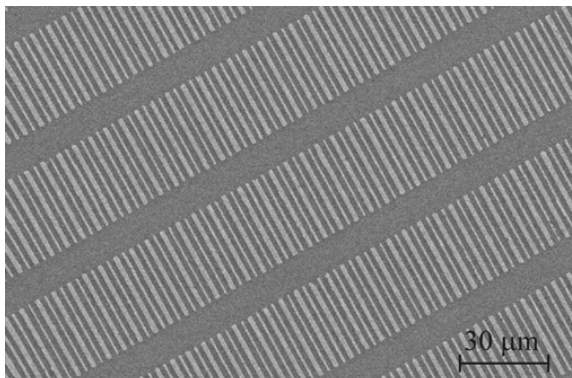


Fig. 1: Scanning electron microscopy image of the two dimensional array of lines of alternating wide and narrow Fe bars.

represent stepped hysteresis loops respective of two different coercive fields of the wide and narrow stripes. We performed PNR measurements at the instrument ADAM (ILL) in the easy axis configuration using a pencil detector and a PSD. Measurements with a sample rotation of 45° were only performed with a pencil detector. The aim pf the present experiment was to repeat these measurements by using a PSD. In Fig. 3 we show a scan performed along the total reflection edge with a varying angle of incidence α_i and a constant exit angle α_f . In this scan reflections from both lateral periods can be observed: at small angles α_i the period of about 40 μm with respect to the array of lines of bars and at higher angles α_i the period of the unit cell of the wide and narrow bars of 6 μm

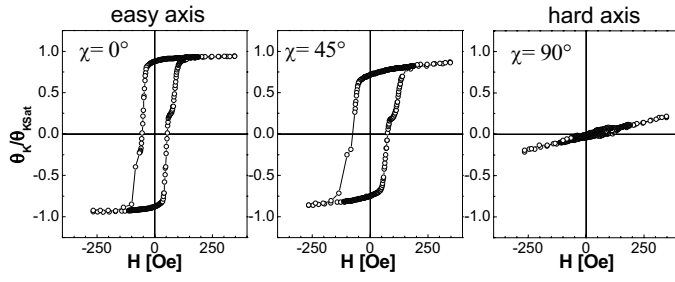


Fig. 2 MOKE hysteresis loops performed at different angles of sample rotation with respect to the applied field.

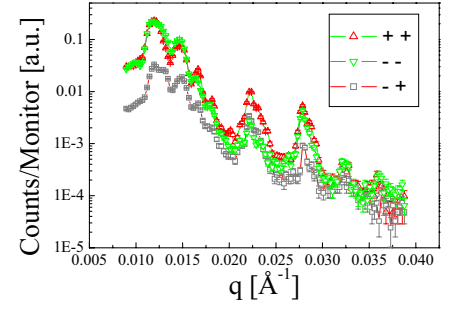


Fig. 3: Polarized neutron reflectivity measurement performed along the total reflection edge at $\alpha_f = \text{const.}$

In the HADAS experiment we planned to measure polarized intensity maps at different magnetic fields in the 45° configuration. In Fig. 4 measurements performed at two different fields are presented. The measurements at 49.5 Oe (left maps) are performed at a field below the coercive fields of both types of stripes. It shows specular reflectivity, off-specular Bragg diffraction from the lateral period and some off-specular diffuse scattering. The second-order Bragg reflections have a higher intensity than the first order one. This is due to structure factor reasons. The measurements at 94 Oe (right maps) are performed in the antiparallel state of both types of stripes. In this case antiparallel alignment changes the magnetic structure factor resulting in higher intensity at the first-order Bragg reflections. Additionally; Bragg diffraction from the large period of the lines of bars of $40 \mu\text{m}$ can be observed close to the specular line. There can also be observed some changes with changing field.

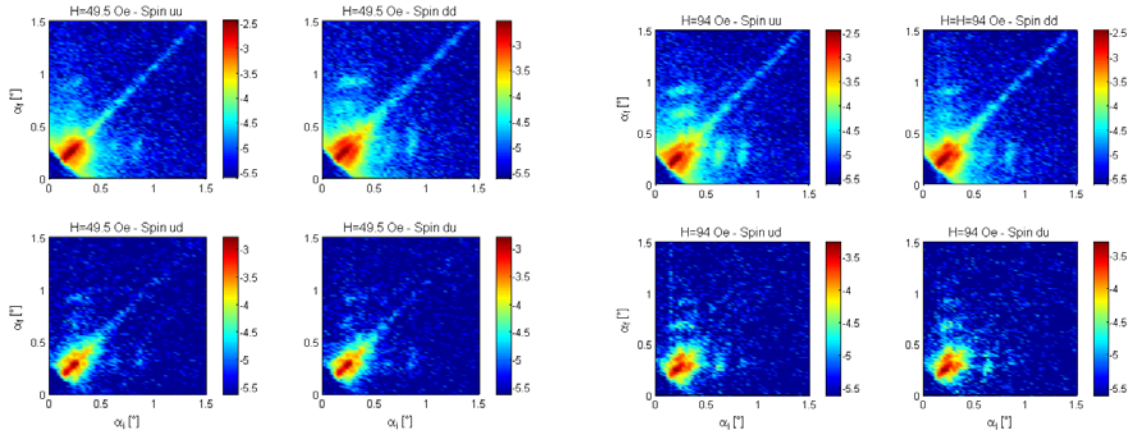


Fig.4: PNR maps performed at two different magnetic fields.

Unfortunately, we had to stop our experiment after measuring at 94 Oe because of enhanced background due to another experiment. This enhanced background already can be seen in the Spin up-down map taken at 94 Oe. Unfortunately, also the measurements performed in March 2005 were not successful.



Experimental Report of Neutron Scattering Experiments at the FRJ-2 Reactor

Proposal number:	HAD-04-017		
Experiment title:	Polarized neutron reflectometry on discontinuous metal insulator multilayers		
Dates of experiment:	24.01.2005 to 07.02.2005	Date of report:	06.06.2006
Experimental team: Names	Addresses		
Prof. Dr. W. Kleemann S. Bedanta, M.Sc. Dr. O. Petravic	Angewandte Physik, Universität Duisburg-Essen, 47048 Duisburg Angewandte Physik, Universität Duisburg-Essen, 47048 Duisburg Angewandte Physik, Universität Duisburg-Essen, 47048 Duisburg		
Local Contact:	Dr. E. Kentzinger, Dr. A. Paul, Dr. U. Rücker		

Experimental report text body

Introduction :

Polarized neutron reflectivity (PNR) studies have been performed on granular multilayers $[\text{Co}_{80}\text{Fe}_{20}(t_n)/\text{Al}_2\text{O}_3(3\text{nm})]_9$ for two different nominal thicknesses $t_n = 1.7$ nm and 1.8 nm (percolating ferromagnet). The measurements were done at several temperatures, $T = 150, 300$ and 350 K with the new cryostat in the field range of $0.47 \leq \mu_0 H \leq 12$ mT.

Motivation :

Interacting ferromagnetic nanoparticle systems $[\text{Co}_{80}\text{Fe}_{20}(t_n)/\text{Al}_2\text{O}_3(3\text{nm})]_m$ where m represents the number of bilayers, show different kinds of collective behavior depending on the nominal thickness of CoFe layers t_n . For $t_n < 1$ nm one finds superspin glass (SSG) behavior [1], while at higher coverages ($1 \text{ nm} < t_n < 1.5 \text{ nm}$) the interactions between the superspins give rise to a “superferromagnetic” (SFM) state with magnetic percolation, but particulate structure [2,3]. Above $t_n = 1.4$ nm percolation occurs, and the system behaves like a bulk ferromagnet (FM). The samples have an in-plane uniaxial anisotropy induced by the preparation method. The magneto-optic Kerr effect in polar geometry reveals absence of the polar component of magnetization in the sample, *i.e.* the sample magnetization lies in the film plane. SQUID measurements performed on samples with $t_n = 1.7$ nm and 1.8 nm show steps in the hysteresis. The possibility of layer-by-layer magnetization reversal in this sample is studied by PNR on these two samples.

Results and Discussions :

In this report we will discuss the results obtained from the *percolating ferromagnetic sample with $t_n = 1.8$ nm*. Fig. 1 (a) shows PNR data taken at positive saturation (12 mT). The FM state of the sample can be recognized by the splitting of the reflectivity edges where $R^{++}(\theta)$, is shifted to higher angles in comparison to $R^{--}(\theta)$ [3]. The first order structural superlattice Bragg peak at $2\theta = 4.6^\circ$ is dominant in the R^{--} channel. Its position corresponds to a bilayer thickness $d \approx 5.6$ nm in agreement with X-ray results (data not shown here). The presence of a weak signal in the two SF channels R^{+-} and R^{-+} is primarily due to the finite flipping ratio of 18 corresponding to the limited efficiency of 94 % for the polarization analysis. The oscillatory behavior (Kiessig fringes) of R^{++} and R^{--} is due to the multilayer structure of the sample. The PNR data shown in Fig. 1(c) were taken at the weak guiding field (0.47 mT) of the neutrons after negatively saturating the sample. They closely refer to the negative remanence as indicated by the low angle data close to the plateau of total

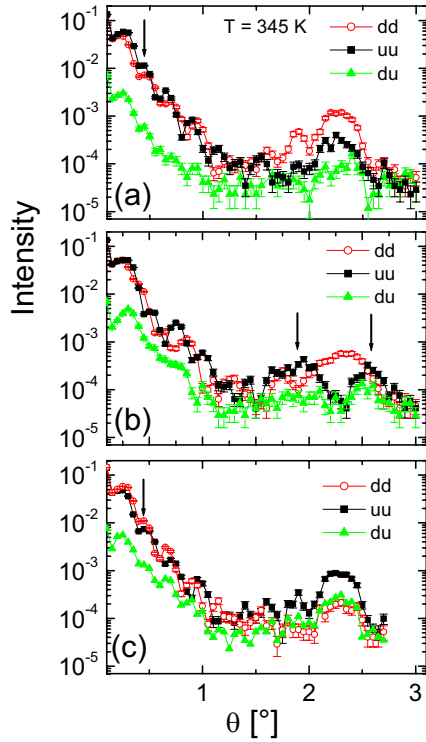


Fig.1. Spin-polarized neutron reflectivities R^{++} in red (dark gray), R^- in black and R^{+-} in green (light gray) vs. angle of incidence, θ , measured at $T = 350$ K and $\mu_0 H = 12$ mT (saturation) (a), close to the coercive field $\mu_0 H = 1.95$ mT (b), and close to remanence in the guiding field of the neutrons, $\mu_0 H = 0.47$ mT, after negative saturation (c) of ferromagnetic $[\text{Co}_{80}\text{Fe}_{20}(1.8\text{nm})/\text{Al}_2\text{O}_3(3\text{nm})]_9$.

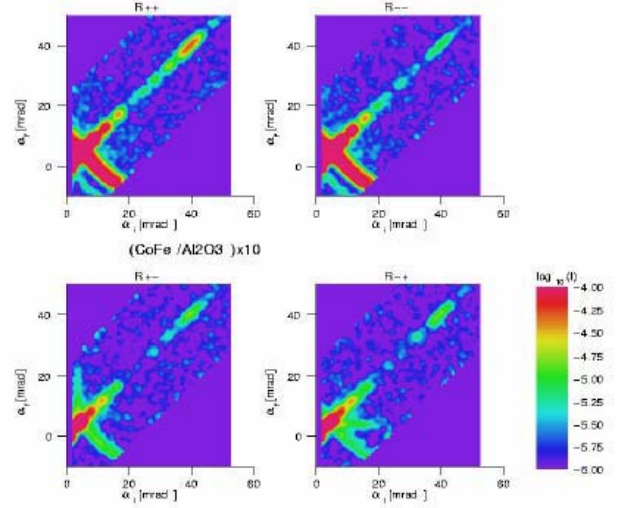


Fig.2. Off-specular scattering of polarized neutrons measured at the remanence of $\mu_0 H = 0.47$ mT, after negative saturation of a ferromagnetic $[\text{Co}_{80}\text{Fe}_{20}(1.8\text{nm})/\text{Al}_2\text{O}_3(3\text{nm})]_9$ sample.

Interestingly measuring the neutron reflectivity at the coercive field the sample exhibits two enhanced satellites (arrows) near the 1st Bragg peak ($\theta = 2.3^\circ$) as shown in Fig. 1 (b). Analysing this specular reflectivity we found that this is probably due to an oscillating magnetization from FeCo layer to FeCo layer with a period of eight bilayers along the multilayer stack [5]. Presence of weak spin-flip intensity hints a transverse component of magnetization in the sample which may arise from the domain walls or from a transverse component of the net magnetization in the sample. We also observed off-specular scattering from this sample arising due to domains as shown in Fig. 2. Further analysis of the diffuse scattering will reveal the mean domain size in the sample. It has been found by the help of micromagnetic simulations that competition between long and short-ranged dipolar interactions apparently gives rise to this modulated magnetization depth profile in magnetic multilayers. [6]

Similar results were obtained for the DMIM sample with $t_n = 1.7$ nm. The data analysis is in progress.

Conclusions and acknowledgements :

In the ferromagnetic samples an oscillatory magnetization depth profile is found. Off-specular scattering was observed from which one can calculate the mean domain size in the sample.

We thank Dr. A. Paul, Dr. Emmanuel Kentzinger and Dr. Ulrich Rücker for their support at HADAS and we gratefully acknowledge Forschungszentrum Jülich for the financial support for the work at Jülich.

Reference:

1. X. Chen et al., Phys. Rev. B **72** (2005) 214436.
2. W. Kleemann et al., Phys. Rev. B **63** (2001) 134423.
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Experimental Report of Neutron Scattering Experiments at the FRJ-2 Reactor

Proposal number:	HAD-04-018, HAD-04-019		
Experiment title:	Test of a transmission polarizer device for KWS-2 – Part I and Part II		
Dates of experiment:	26.11. – 28.11.04 21.01. – 24.01.05	Date of report:	12.06.2006
Experimental team: Names	Addresses		
E. Kentzinger A. Ioffe U. Rücker	Institut für Festkörperforschung, Forschungszentrum Jülich		
Local Contact:	E. Kentzinger		

Experimental report text body

This experiment was entering in the project of equipping KWS-2 with polarized neutrons until its move to FRM-II.

A first try has been performed, using the spin down neutrons transmitted by the polarizer of NSE that is sharing the same neutron guide as KWS-2. This experiment was rather unsuccessful: a flipping ratio of only 2-to-3 was obtained at the KWS-2 sample position. We explain this by the bending of the KWS-2 guide and by the fact that the NSE-polarizer does not polarize the whole incoming divergence of the neutron guide.

A second idea that we had was to build a polarizer device based on transmission polarizers already tested on HADAS (see report HAD-03-005). Those transmission polarizers do not produce any small angle scattering in the -- and spin flip channels if they lie in a strong enough field ($\mu_0 H > 0.4$ Tesla). However, some ++ small angle scattering, due to interfacial roughness, is still present (see Fig. 1a).

In order to remove this small angle scattering, we have installed, 0.5 mm behind this polarizer, an identical transmission polarizer that would reflect this ++ scattering and bring it above the horizon. This idea has been tested and the measurement performed on such a device is shown in Fig. 1b. We see a strong reduction of the ++ small angle scattering around $\alpha_i = 5$ mrad and around the primary beam. Some small angle scattering intensity is still seen around $\alpha_F = 5$ mrad. We explain this by the fact that, due to the limited space at sample position on the HADAS instrument, we were not able to install a long enough ‘analyzer’ supermirror and that those small angle scattered neutrons therefore did not meet it.

Practically, the polarized SANS and GISANS measurements on KWS-2 were performed using only one single polarizing supermirror. As a matter of fact, the tests on KWS-2 surprisingly revealed that, in that case, the level of GISANS was smaller than using a two supermirrors set-up..

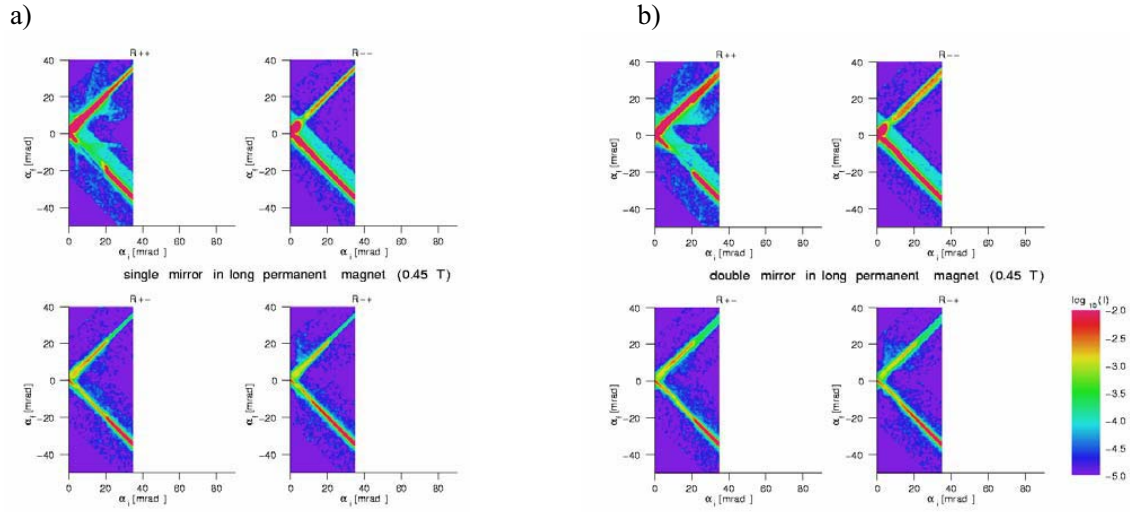


Fig.1: a) Reflectivity ($\alpha_F=\alpha_I$), off-specular scattering ($\alpha_F>0$), transmission ($\alpha_F=-\alpha_I$) and small angle scattering ($\alpha_F<0$) measured on a single transmission polarizer at an applied field of 0.45 Tesla. α_I and α_F are respectively the angles of incidence and exit of the neutrons with respect to the sample surface. The large background intensity just above the primary beam does not come from our sample but from diffuse scattering from the HADAS polarizer.

b) Same as before but with a second ‘analyzing’ supermirror placed 0.5 mm behind the first polarizer. ($s_1=s_2=0.3$ mm).

Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	HAD-05-001		
Experiment title:	Magnetic field distribution in $\text{YBa}_2\text{Cu}_3\text{O}_7$ / $\text{La}_{2/3}\text{Ca}_{1/3}\text{MnO}_3$ multilayers		
Dates of experiment:	5.4. - 14.4.2005	Date of report:	June 1, 2005
Experimental team:			
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Temperature dependent neutron reflectivity measurements showed an unexpected behaviour of the magnetic field distribution of superlattices consisting of $\text{YBa}_2\text{Cu}_3\text{O}_7$ and $\text{La}_{2/3}\text{Ca}_{1/3}\text{MnO}_3$ [1]. To get a better insight in this field distribution, measurements at different temperatures with polarized neutrons with spin analysis were performed on HADAS.

The sample was first cooled to 5 K in an applied magnetic field of 100 Oe. By keeping the applied magnetic field constant, off-specular measurements of all spin channels were performed at 5 K, 90 K, 130 K, 160 K, 200 K and 300 K.

At 5 K, which is well below the transition temperature of the superconductor ($T_c \approx 80$ K), a Bragg sheet at the first Bragg peak was found in the $(--)$ channel. In the same channel Yoneda-like wings with weak intensity at the first Bragg peak were also observed (Fig. 1) [2]. While the Bragg sheet indicates a correlation vertical to the layers, the Yoneda-like wings indicate a correlation in the plane of the layers. The Yoneda-like wings were found to be more pronounced in the two spin flip channels (Fig. 1). This means, those wings are caused by a magnetic correlation in the plane of the layers with some magnetic moments pointing out of the direction of the applied magnetic field.

When heating the sample to 90 K, both features remain with only slight changes.

At 130 K, the Bragg sheet in the $(--)$ channel becomes slightly smaller and the Yoneda-like wings decrease in the spin flip channels while they increase in the $(--)$ channel. One part of this transition between 90 K and 130 K may be induced by a structural phase transition of the substrate SrTiO_3 at ~ 108 K. However there is clear evidence for a change in the magnetic field distribution, as the intensity of the Yoneda-like wings in the spin flip channel decreases.

The most significant change in the coherence, however, occurs between 90 K and 130 K. The length of the Bragg sheet in the $(--)$ channel and the Yoneda-like wings in the spin flip channel decrease significantly. At 200 K, the Bragg sheet and the Yoneda-like wings disappear in all channels. As there is no Bragg sheet and there are no Yoneda-like wings visible at roomtemperature (which is above the ferromagnetic transition temperature $T_{Curie} \approx 240$ K), all correlations found must be of magnetic origin. Due to the magnetic correlation vertical to the layers, it seems that the ferromagnetic layers somehow couple at low temperatures.

This correlation remains to temperatures much higher than the superconducting transition temperature. Therefore it is not sure, whether this interaction is caused by superconductivity or not.

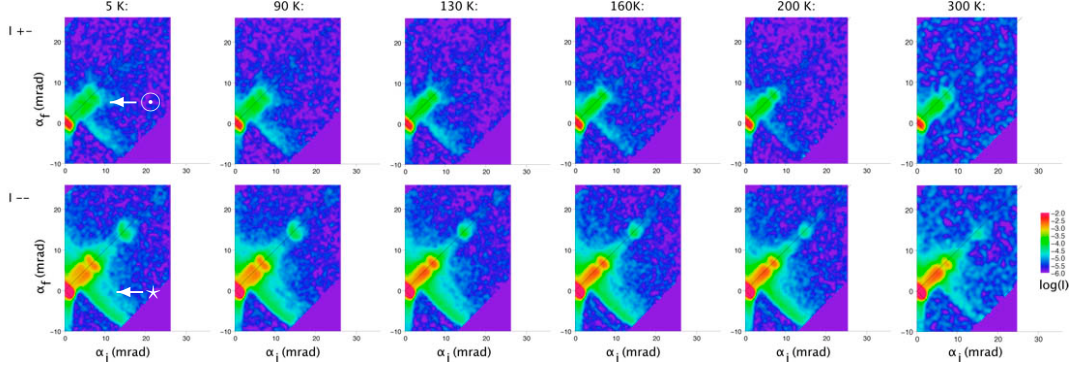


Figure 1: Off-specular and specular reflectivities of the spin flip channel $(+-)$ and the non spin flip channel $(--)$ in an applied magnetic field of 100 Oe at 5 K, 90 K, 130 K, 160 K, 200 K and 300 K. The Bragg sheet ($\star$) at the first Bragg peak in the $(--)$ channel and the Yoneda-like wings ($\odot$) at the first Bragg peak in the spin flip channel decrease the most between 130 K and 160 K.

To study out the influence of the applied magnetic field on this correlations, the sample was cooled from roomtemperature to 5 K in applied magnetic fields of 11 Oe, 100 Oe and of 4000 Oe (Fig. 2). We found that an applied field of 100 Oe results in a good alignment of the magnetic moments. Only then a magnetic correlation is obtained. In a magnetic field of 4000 Oe the magnetic correlation is different compared to in a field of 100 Oe. The vertical correlation length is increased and the Yoneda-like wings at the first Bragg peak are only visible in the $(--)$ channel, which means that the magnetic moments of the ferromagnet are aligned exactly along the applied field.

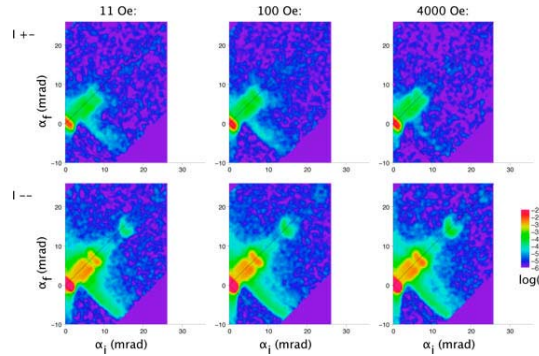


Figure 2: Off-specular and specular reflectivities of the spin flip channel $(+-)$ and the non spin flip channel $(--)$ at 5 K in an applied magnetic field of 11 Oe, 100 Oe and 4000 Oe.

References:

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Experimental Report of Neutron Scattering Experiments at the FRJ-2 Reactor

Proposal number:	HAD-05-002		
Experiment title:	Off-specular scattering from magnetic neutron wave-guides structures		
Dates of experiment:	24.05.2005-28.05.2005	Date of report:	31.03.2006
Experimental team: Names	Addresses		
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Frédéric OTT	Laboratoire Léon Brillouin UMR12, CEA/Saclay, bât. 563 91191 Gif-sur-Yvette Cedex, France Tel: +33 (0)1 69 08 61 21, FAX : +33 (0)1 69 08 82 61 e-mail: fott@cea.fr		
Local Contact:	Amitesh PAUL		

Experimental report text body

This investigation is the part of the project on development of neutron wave-guides (NWG). A NWG consists of a thin film of low optical index (Ti or Al) with thickness of 10-100 nm sandwiched between 2 layers of high optical index. The neutron wave can channel inside the guiding layer with low optical potential. The practical application of NWG is producing of highly collimated narrow (nm) beam which goes out from the edge of a sample while keeping a significant neutron flux. In such structures the interaction of the neutron beam is strongly enhanced when resonant states (or guided states) are created in the neutron wave-guide [1]. Several experiments have already been performed with non-magnetic neutron wave-guides [2-5]. Magnetic materials (permalloy, Fe, Ni) can be used for changing of an upper layer transparency and increasing of neutron beam intensity on the exit of a neutron wave-guide.

On reflectometer HADAS (neutron wavelength is $\lambda=0.47$ nm) we have investigated magnetic neutron waveguides Py10(20nm)/Ti70(10nm)/Py(50nm)/glass. On Fig. 1 a,b calculation of neutron wave function of spin states up and down inside the wave-guide Py20nm/Ti70/Py50/glass is shown. For each incidence conditions corresponding to a propagation mode in the wave-guide, there is an enhanced scattering in the wave-guide at the interfaces and this leads to dips in the total reflection region (Fig. 2). Another contribution in dips at the total reflection is wave-guiding effect which can be considered as additional absorption. Simultaneously, for each of the resonance conditions, the scattering by the interfaces is strongly enhanced and a significant off-specular scattering is observed (Fig. 3 a,b). Thus even small interfacial roughness effects are easily observed. For our study these "roughness" effects are parasitic effects. In magnetized state in 100 Oe magnetic field there is only non spin-flip off-specular reflection due to structural lateral roughness correlations.

In low magnetic field 2 Oe demagnetized state of wave-guide structure PyTiPy has been investigated. Additional spin-flip off-specular reflection due to magnetic domains in lateral direction has been observed.

This work has been done under contract Polarized Neutron Technology EUR 634505925-08 A-LABNA-01-06-08. This experiment has been supported by NMI3 program for users.

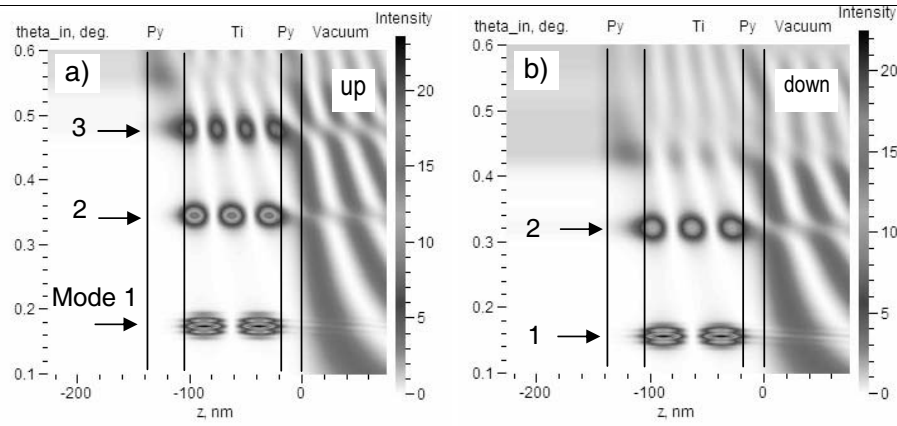


Fig. 1. Calculation of neutron wave-function $|\psi|^2$ for spin up (a) and down (b) inside wave-guide structure Py04nm/Py17/Ti82/Py48/glass.

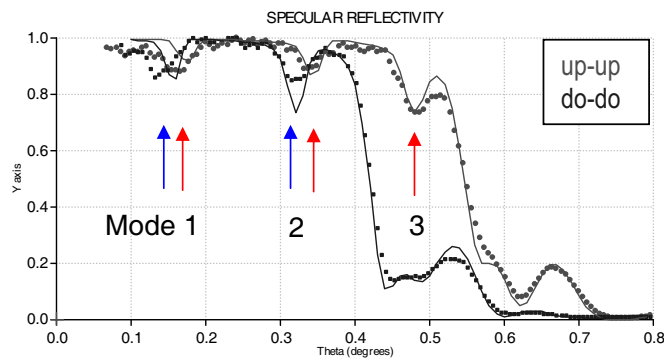


Fig. 2. Specular reflectivities up-up (red) and down-down (blue) from structure Py20nm/Ti70/Py50/glass.

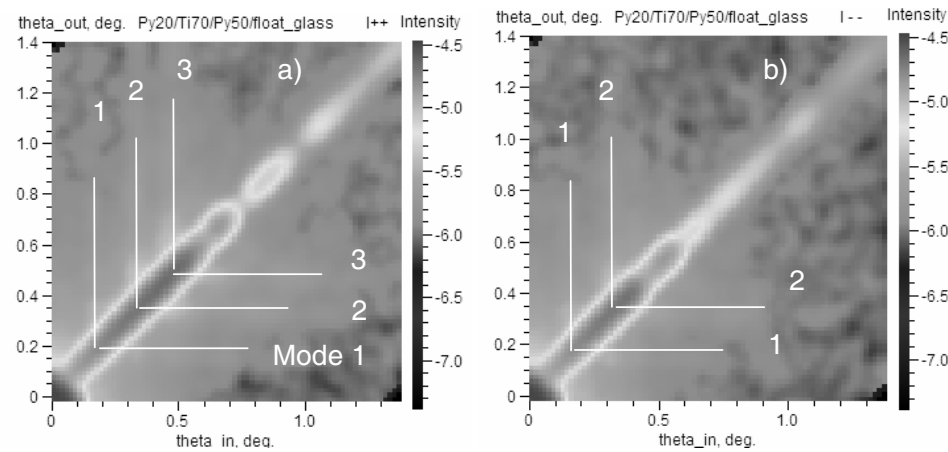


Fig. 3. Off-specular reflection from magnetized structure PyTiPy in 100 Oe for spin up-up (a) and down-down (b).

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Experimental Report of Neutron Scattering Experiments at the FRJ-2 Reactor

Proposal number:	HAD-05-003		
Experiment title:	Test of a 2D-CASCADE detector with high efficiency with reflectometry experiments		
Dates of experiment:	20.04. - 24.04.05	Date of report:	24.05.06
Experimental team: Names	Addresses		
Dr. Werner Hinderer Dr. Martin Klein Dr. Christian Schmidt	Universität Heidelberg Physikalisches Institut Philosophenweg 12 69120 Heidelberg Tel.: +49-6221-549376		
Local Contact:	U. Rücker, E. Kentzinger		

Experimental report text body

The aim of this test session was to continue the evaluation of the CASCADE detector technology and to gain references by performing reflectometry with a glass plate covered with Nickel.

The first prototype of a 2D-CASCADE detector was successfully tested during the operation of the FRJ-2 reactor at the experimental test site EKN in November 2004. For this experiment, an extended and improved version of the 2D-CASCADE detector was characterized and used for reflectometry.

In figure 1 the reflection profile of a Nickel metallic film coated on glass is shown. The data points show the measured specular reflected neutron intensity (normalized to the incident neutron intensity). They were acquired with the scintillation detector of the HADAS instrument. From this profile one can determine the thickness of the metallic film. The fit (red curve) yields a thickness of $(830 \pm 1) \text{ \AA}$.

With the same setup, the measurement was repeated with the 2D-CASCADE detector.

The operation of the HADAS reflectometer by our group and the integration of the CASCADE detector into the HADAS environment took more time as expected. Thus there was not enough time to measure the reflection profile at larger angles as we did with the scintillation detector. The result is shown in figure 2. For the Nickel film one gets a thickness of $(855 \pm 4) \text{ \AA}$. A matching result for the two thickness determinations may be expected if more periodic oscillations would have been measured.

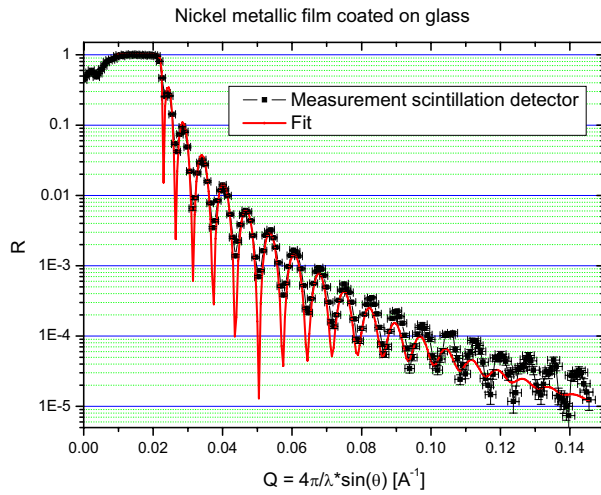


Figure 1: Curve of reflection from a Nickel surface measured with the scintillation detector at the HADAS instrument.

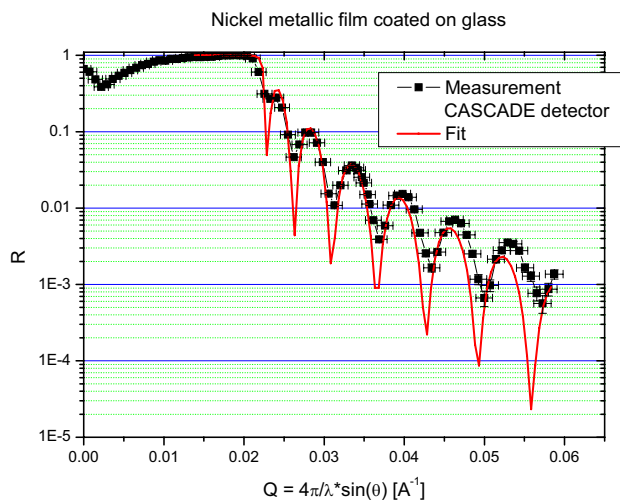


Figure 2: Curve of reflection from a Nickel surface measured with the 2D-CASCADE detector.

This beamtime showed that the 2D-CASCADE detector can be integrated into existing neutron instruments. The comparison between the 2D-CASCADE detector and the scintillation detector of the HADAS instrument demonstrates the correct working of the 2D-CASCADE detector.

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Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	HAD-05-004		
Experiment title:	Investigating structural evolution in Co/Cu multilayers with number of bilayers		
Dates of experiment:	14.04.05	Date of report:	February 22, 2006
Experimental team:			
Names:	Amitesh Paul, Ulrich Ruecker, Emmanuel Kentzinger, Thomas Brueckel		
Address:	Institut für Festkörperforschung Forschungszentrum Jülich GmbH D-52425 Jülich, Germany Deutschland		
Local contact:	Amitesh Paul		

Physical properties of magnetic multilayer (MLs) such as interlayer coupling [1] and giant magnetoresistance (GMR) [2] are not only closely related to the structure of the multilayers, *i.e.* interface roughness and/or the evolution of the roughness along the ML stack,[3] but depend also on the details of the magnetic domain structure.[4] Recent investigations by Langridge *et al.*[4] and Paul *et al.*[5] have demonstrated the usefulness of neutron techniques in determining the field dependence of the antiferromagnetically (AF) coupled domain structure.

Recently, Borchers *et al.*[6] have shown the dependence of GMR on the vertical correlation of domains in a coupled ML at the coercive field. The loss of GMR was attributed to the vanishing of the vertical correlation. On the other hand, vertical correlation of domains was shown to exist at the coercive field for a coupled system.[4] However, though the variation of the domain size with applied field was addressed for weakly as well as strongly coupled systems, there is so far no investigation of the evolution of the domain characteristics along the growth direction of the ML. In previous work, we have observed that the strength of the AF coupling and the GMR values correlate with the structural evolution for increasing number of bilayers (N) of a ML.[3] In the present work, we report on the domain structure evolution in AF-coupled Co/Cu ML stacks as observed from neutron scattering measurements. These MLs show a high degree of morphological vertical correlation and small lateral correlation of the interface roughness.[3] We observe the domain structure of the Co layers to evolve from large to small vertically correlated domains along the stack. The larger ones remain AF aligned beyond the apparent saturation field, while the antiparallel alignment for the smaller breaks up at lower external fields.

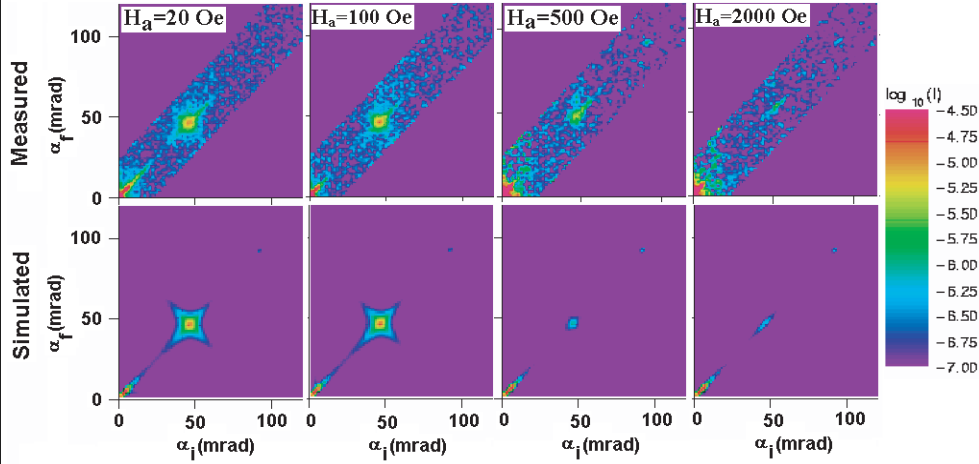


Figure 1. SF intensity maps (measured and simulated) at different fields for $[\text{Co/Cu}]_{N=40}$ sample.

Details of the preparation and characterization of similar $\text{SiO}_2/\text{Co}(1.45 \text{ nm})/[\text{Cu}(1.02 \text{ nm})/\text{Co}(1.45 \text{ nm})]_{N=10 \text{ or } 40}$ structures as used here have been described [3]. The SF intensities near 46 mrad for $H_a = 500 \text{ Oe}$ are larger than the NSF intensities due to diffuse scattering and indicates the presence of lateral inhomogeneities caused by domains. This is best seen in the SF intensity maps shown in Fig. 1 for different H_a . The $\frac{1}{2}$ -order Bragg peak at $\sim 46.5 \text{ mrad}$ is broad along $Q_{\parallel}$ as well as along $Q_{\perp}$. Its intensity rapidly shrinks with distance from the specular line and also decreases with increasing H_a . Although all four channels are fitted within the single domain approximation, we note that the widths of the $\frac{1}{2}$ -order Bragg peak of the SF intensities could not be reproduced because these peaks are diffuse in nature. Conventional magnetometry (MOKE and SQUID) yields a smaller value of the saturation field than the value determined from off-specular polarized neutron scattering. Thus, PNR reveals the magnetic state in more detail. Smaller domains most likely at the top of the ML with a size of $0.5\text{-}1.5 \mu\text{m}$ are saturated at a lower field of $\approx 500 \text{ Oe}$, whereas the larger domains ($\geq l_{\parallel} \approx 20 \mu\text{m}$) beneath remain AF-coupled beyond apparent saturation at $\approx 1.5 \text{ kOe}$.

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Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	HAD-05-006, HAD-05-017		
Experiment title:	Confinement Effects of Diblock Copolymers in Lamellar Micro-emulsions I, II		
Dates of experiment:	9.5., 27.5., 13.6., 18.8. - 20.8., 29.8., 26.9. - 29.9., 3.11.2005	Date of report:	17.03.2006
Experimental team: Names	Addresses		
Simona Maccarrone Henrich Frielinghaus	Forschungszentrum Jülich GmbH, Institut für Festkörperforschung, D-52425 Jülich Forschungszentrum Jülich GmbH, Institut für Festkörperforschung, D-52425 Jülich		
Local Contact:	Ulrich Rücker		

Experimental report text body

The current work (PhD thesis) of Simona Maccarrone considers amphiphilic diblock copolymers in micro-emulsions. The domain size of the oil and water domains is changed by the selection of the oil chain length. Thus, the ratio between the domain size and the polymer size is varied. Confinement takes place when both sizes are similar. While a large part of the work focuses on bicontinuous microemulsions, which are characterized by phase diagram measurements and SANS experiments, the study should be extended to lamellar microemulsion with confined polymers. The quasi-Bragg peak is very sharp, and therefore conventional SANS experiments should be combined with SANS measurements at HADAS. HADAS allows to measure with a very high resolution in one direction ($\Delta Q_x/Q_x \approx 1\%$, in this direction the sample is aligned), while the resolution is relaxed in the other direction. The analysis of the bicontinuous microemulsions involves a theoretical apparatus, which neglects the pressure that one domain exerts on the surfactant film (more precise the osmotic pressure of the membrane). This pressure is included in the Caillé theory, which describes lamellar microemulsions. At higher confinement the pressure of the polymer on the membrane should be non-negligible, and therefore the consideration of lamellar microemulsions should be included in the work.

Compared to the bicontinuous microemulsions the lamellar microemulsion contains higher amounts of surfactant. Therefore the domain size is quite small. Especially due to the lack of a temperature control, the temperature window of the lamellar microemulsion was rendered quite large (typically $\sim 18^\circ\text{C}..32^\circ\text{C}$), and therefore high surfactant contents of 25%..30% were chosen. Initially the widely used C_{10}E_4 surfactant was selected, but to extend the range to lower confinement another C_{14}E_5 surfactant was involved. Four samples contained too few surfactant (20%) and coexistence of a bicontinuous and a lamellar microemulsion was obtained. Eighth samples were measured successfully with varying polymer size and constant surfactant amount. A reference sample without any polymer allowed to measure the change of the bending rigidity and the osmotic pressure according to the added polymer. All experiments are conducted at film contrast.

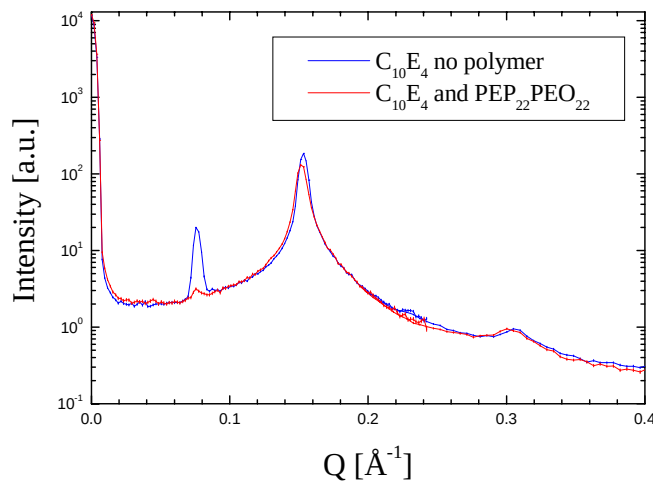


Figure 1: Logarithmic intensity as a function of the scattering vector Q (SANS-plot). On the left the primary beam is measured. At $Q=0.15\text{\AA}^{-1}$ the lamellar structure of the surfactant film is reflected (film-contrast). The superstructure of the domains appears at $Q=0.08\text{\AA}^{-1}$ due to the imperfect contrast matching. The second order peak is visible as well. Changes in the peak width are tiny.

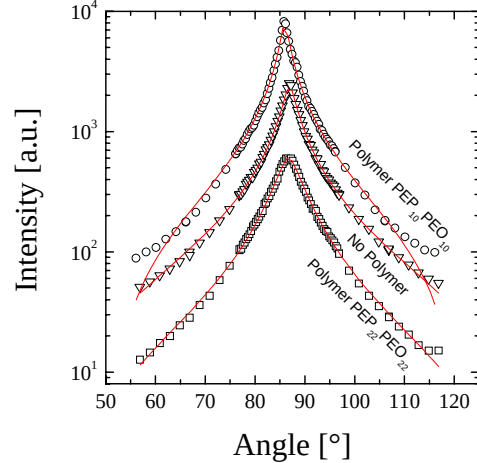


Figure 2: Rocking curves of three samples. The zero angle is at 90° . Intensities of the upper two samples are shifted by a factor of 3 and 10. Fit curves were used to describe the divergence phenomenologically. The highest curve is the sharpest, the lowest one is the broadest.

The measured curves (SANS and rocking) are evaluated in the following manner. The normal SANS scan shows several properties (Fig 1), namely (a) the primary beam at small scattering angles, (b) the quasi Bragg peak is observed at $Q=0.15\text{\AA}^{-1}$, (c) the superstructure of the domains is found at $Q=0.08\text{\AA}^{-1}$ due to imperfection in the contrast match, and (d) a second order peak is observed at $Q=0.3\text{\AA}^{-1}$. Furthermore at low enough scattering angles (before the upturn of the primary beam) the scattering intensity can be extrapolated to the forward scattering $S(0)$. Using the integral intensity of the primary beam, this $S(0)$ can be calculated on an absolute scale, and therefore $S(0)$ can be connected to the osmotic pressure. The quasi Bragg peak and the second order peak and intensities around (but not the superstructure peak) can be described by the Caillé theory. The detailed numerical handling has been described by Monkenbusch [1], only an additional integral due to the relaxed resolution in the perpendicular direction was necessary. The intensities were well described with an arbitrary amplitude factor, but the resulting bending rigidity κ and the osmotic compressibility B do not meet the expectations. The obtained κ was around $200k_B T$ while from SANS experiments one would expect 0.5 to 1 $k_B T$. A similar discrepancy was obtained between B and $S(0)$. Furthermore, the changes in the peak sharpness are not extremely high, since the divergence is predicted to have an exponent $-(2-\eta)$. More promising are the rocking curves (Fig 2). Here the divergence has an exponent $-2(2-\eta)$. The sharpness of the peaks can be read off already visually.

All attempts to extract values for the bending rigidity had the following problem. With respect to the reference sample (no polymer), the obtained changes $\Delta\kappa$ had varying signs independent of the selected confinement. So the tendency increased / decreased bending rigidity was not observed within the accuracy of the experiment. The precision of this experiments is not high enough. While describing the SANS curves with the Caillé theory lead to unphysically high κ values, the determination of η by a phenomenological fit of the rocking curves lead to reasonable absolute values of κ . Nonetheless the changes of κ were not *systematically*.

The quality of the alignment was checked by conventional SANS measurements where the Bragg peaks were sharp in all directions. Anyhow the present understanding of the malfunction of this experiment is the imperfect aligning. All samples were heated up to $\sim 50^\circ\text{C}$ prior the cooling down to room temperature. At high temperatures the sample was one phase, then, at intermediate temperatures, there was a 2-phase region with coexisting lamellar and microemulsion phases, and finally the sample arrived at the 1-phase lamellar state. The intermediate 2-phase region might be the reason for individual perfection of alignment of each sample.

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Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	HAD-05-007		
Experiment title:	Stability of domain structure in exchange bias system CoFe/MnIr		
Dates of experiment:	11.05.-19.05.2005	Date of report:	14.06.2006
Experimental team: Names	Addresses		
Katharina Theis-Bröhl	Ruhr-Universität Bochum Institut für Experimentalphysik IV 44780 Bochum		
Boris Toperverg			
Local Contact:	Ulrich Rücker		

Experimental report text body

We performed polarized neutron reflectometry studies on the instrument HADAS on an exchange bias (EB) system with imprinted magnetic microstripes. The sample studied is a $\text{Co}_{70}\text{Fe}_{30}$ (28 nm)/ $\text{Mn}_{83}\text{Ir}_{17}$ (15 nm) ferromagnetic/ antiferromagnetic bilayer with buffer layers Cu (28 nm)/ SiO_2 (50 nm) on $\text{Si}(111)$ and a TaO-Ta (9 nm) top layer prepared by magnetron sputtering. The initial EB direction was set by field cooling in a magnetic in-plane field of 1 kOe after annealing at 275°C which is above the blocking temperature. Subsequently the film was covered by photoresist, which was patterned into equally spaced stripes with a width of 2.5 μm and a periodicity of 5 μm . Then the sample was bombarded with He^+ ions with a fluency of $1 \cdot 10^{15}$ ions/ cm^2 at 10 keV. During bombardment a magnetic field of 1 kOe aligned opposite to the initial EB direction was applied. This resulted in a stripe-like pattern with alternate sign of the unidirectional anisotropy, and hence the sign of the EB in neighboring stripes.

In previous measurements performed at the reflectometers HADAS and ADAM (ILL) we studied the easy axis behavior. [1] We found that the remagnetization within the different areas occurs at different magnetic fields, resulting in a field range with antiparallel orientation of magnetization. With the PNR measurements including quantitative data treatment using DWBA we were able to quantitatively determine the angle of magnetization. Instead of 180° (antiparallel alignment) we found that the magnetization is tilted and the angle between the magnetization in the two different areas is only 120° resulting in a net transverse orientation of magnetization. We explained this behavior with a model using energy contributions due to exchange between ferromagnetic layers, exchange between ferromagnetic and antiferromagnetic layers and anisotropy. In the intensity maps we found three different features of intensity: specular reflectivity, off-specular Bragg diffraction lines from the magnetic period and off-specular diffuse scattering from domains smaller than the coherence length. We observed Bragg reflections for $n = \pm 1$, whereas for $n = \pm 2$ they are suppressed. This is caused by the structure factor, resulting from the equal width of adjacent magnetic stripes in a unit cell.

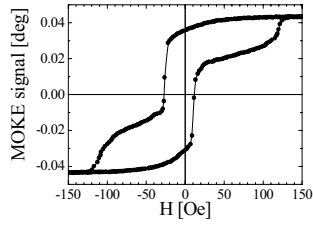


Fig. 1: Longitudinal MOKE hysteresis loop performed on the sample studied in the PNR experiment.

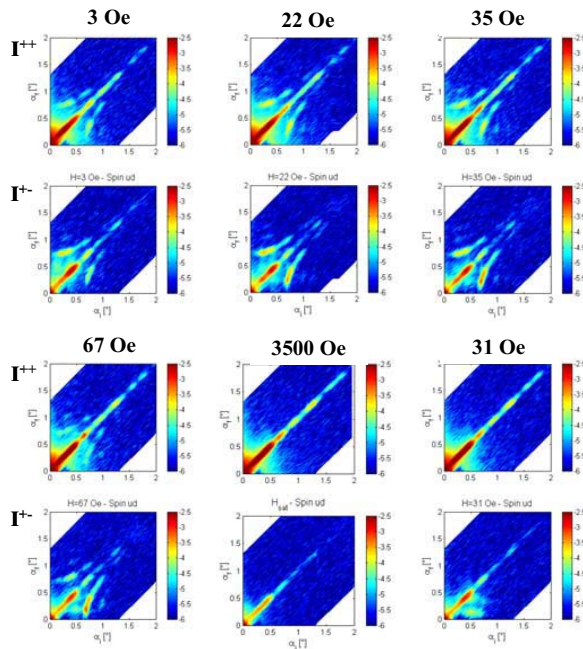


Fig. 2 Polarized intensity maps taken at different magnetic fields.

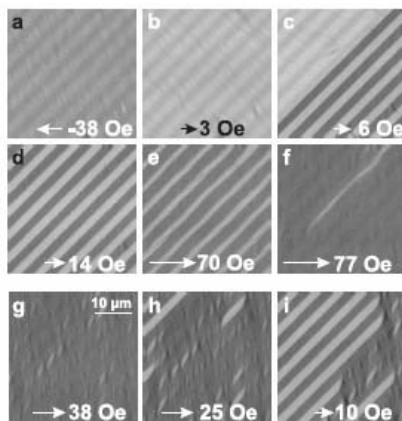


Fig. 3: Kerr microscopy measurements performed with an angle of 45° between sample and applied field.

In the present experiment we rotated the sample by 45° and performed PNR measurements at different magnetic fields along the hysteresis loop shown in Fig. 1. The field values for which intensity maps were collected are: 3.5 Oe, 8 Oe, 17 Oe, 35 Oe, 53 Oe, 67 Oe, 71 Oe (after negative saturation), 3.5 kOe (saturation), and 31 Oe (after positive saturation). In Fig. 2 several intensity maps are presented. We measured two non-spin-flip (NSF) and two spin-flip (SF) maps for each field value, but presented in Fig. 2 are only the I^{++} and the I^{+-} maps. For comparison we show in Fig. 3 Kerr microscopy measurements taken also at 45° sample rotation with respect to the applied field and taken at different fields along the hysteresis loop.

In saturation at 3500 Oe only NSF specular reflectivity is visible because the magnetization of the film is completely oriented along the field orientation.

The measurement at 3 Oe was performed just before the first reversal. Off-specular Bragg diffraction, however, can already be observed with a rather strong intensity. Obviously, rotational processes and formation of domains (concluded from off-specular spin-flip scattering) are different in the two magnetic stripe regions which leads to a rather strong magnetic contrast as can also be seen from a Kerr microscopy image in Fig. 3. Increasing the field to 22 Oe leads to less off-specular diffuse scattering and stronger Bragg-diffraction lines. This comes from the fact that now the magnetization of one part of the stripes is reversed and that small domains have disappeared. Further increase of the field results in a beginning reversal of the second part. Due to the canting of the stripe orientation with respect to the field the reversal is quite different compared to the easy axis behavior (field applied along the stripes). In case of the easy axis magnetization the stripes reverse one after the next. In the 45° case the width of the reversed magnetic stripes increases on the expense of the width of the stripes which are not yet reversed. This can clearly be seen in Fig. 3 in the Kerr microscopy image taken at 70 Oe. In the PNR

measurement we observe this effect by the fact that second order Bragg diffraction lines occur, observed already at 35 Oe. The effect is even stronger at 67 Oe. Furthermore, again off-specular diffuse scattering is present, resulting from small domains which hardly can be seen from the Kerr microscopy image in Fig. 3. The maps taken at 31 Oe after saturation in a positive magnetic field do not show off-specular Bragg diffraction lines but strong off-specular diffuse scattering. Again this observation is in very good agreement with the Kerr microscopy image taken at 38 Oe where ripple domains clearly can be observed but not the magnetic stripe structure.

This work was supported by DFG through SFB491.

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Experimental Report of Neutron Scattering Experiments at the FRJ-2 Reactor

Proposal number:	HAD-05-008		
Experiment title:	Multiply bound polymer brushes: understanding loops from triblock copolymers		
Dates of experiment:	14.06. – 21.06.2005	Date of report:	22.06.2006
Experimental team: Names	Addresses		
Mark Dadmun J. Kevin Rice	Chemistry Dept., University of Tennessee, Knoxville, TN 37996 Chemistry Dept., University of Tennessee, Knoxville, TN 37996		
Local Contact:	U. Ruecker		

Experimental report text body

Reactive compatibilization schemes, to produce copolymer interfacial modifiers, appear to be the most industrially applicable means to efficiently produce useful polymer blends from normally immiscible polymer systems. Blocky type copolymers have been shown to be the best interfacial modifiers, as the loops that are formed across the interface, which allow for physical entanglements across the interface, are critical to strengthening the inherently weak interfaces in immiscible polymer blend systems. We want to better understand the reactions of telechelic polymers, in terms of the relationship between kinetics, interfacial morphology, reactivity, and molecular weight, in order to optimize loop formation at soft polymeric interfaces.

To this end, we have undertaken a neutron reflectivity study to elucidate the optimal parameters involved in forming molecular loops through the reaction of telechelic polymers at soft polymeric interfaces. We began investigating the reaction of telechelic polymers at a soft polymeric interface using a model bilayer system consisting of polystyrene and poly(methyl methacrylate), and corresponding telechelics containing various end group functionalities capable of in situ coupling, and monitored the reaction of the deuterated polystyrene telechelics at the interface with neutron reflectivity. Through the use of this model system, the relationships between telechelic molecular weight, functional group reactivity, and reaction time versus reaction rate and extent of reaction were elucidated.

Three reactive pairs were studied: epoxy-carboxylic acid, anhydride-amine, and epoxy-amine. The effect of telechelic molecular weight was also investigated through the use of several different telechelic molecular weights for each reactive pair investigated. Samples were annealed at 150° C under vacuum for 15 minutes, 4 hours, 24 hours, 72 hours, and 108 hours, and reflectivity measurements were taken on the initial as cast films and after each successive annealing step. A representative plot of the evolution of the scattering length density profile with annealing time for the 20K molecular weight telechelic in the epoxy-carboxy system can be seen in Figure 1. It was found that the reactivity of the various functional groups played a significant

role in the kinetics of the in situ copolymer formation process. The anhydride-amine system showed maximum reaction within 0.25 hours, while the carboxy-epoxy and epoxy-amine systems required greater than 24 hours for complete reaction to occur. It is also speculated that different interfacial morphologies result from the reaction of differing functional groups to form copolymer at the immiscible interfaces although the same relationship seems to exist between telechelic molecular weight and total interfacial width, regardless of the end group functionality.

The experiments carried out here have shown that the in situ reaction of telechelics at soft, immiscible polymer/polymer interfaces to form copolymer surfactant molecules, contrary to most theoretical treatments of the process, is not a diffusion-controlled process. While no current theory of the reaction process fits well with our data, we have elucidated several parameters, including telechelic molecular weight and the magnitude of the reduction in interfacial tension, that appear to play a role in the kinetics of the interfacial reaction process. We have shown that copolymers formed from smaller molecular weight telechelics produce a higher reduction in interfacial tension, as evidenced by the larger interfacial widths, at modified interfaces, as opposed to higher molecular weight telechelics. The interfacial excesses measured at the longest annealing time for the smaller molecular weight copolymer samples (<80K molecular weight telechelic) are all above the theoretical value at which the interfacial tension should be zero. This leads us to believe that the triblock copolymers formed from lower molecular weight telechelics are able to pack more tightly at the interface and that these interfaces modified with low molecular weight telechelics are more susceptible to thermal fluctuations, leading to more copolymer formation and a greater reduction in the interfacial tension.

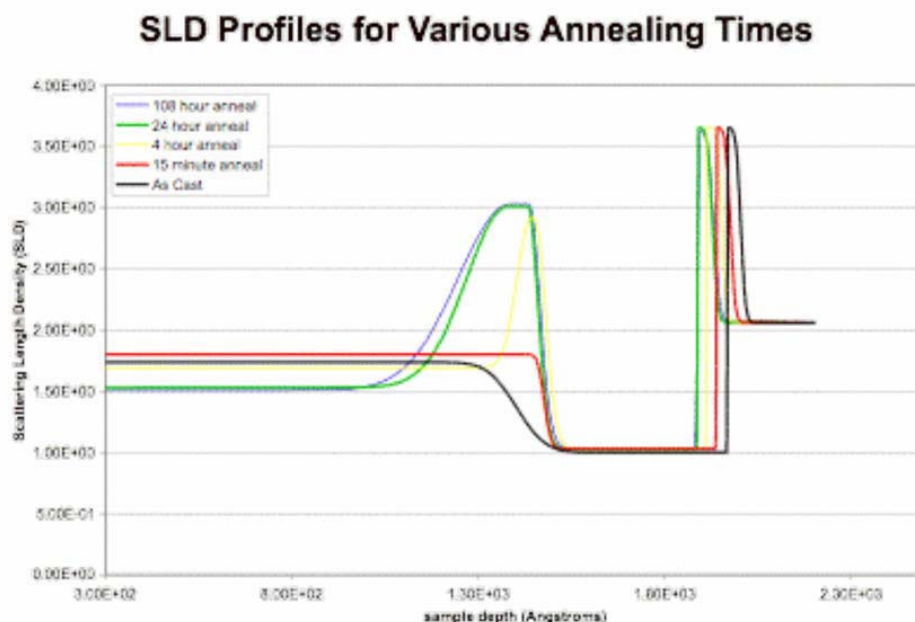


Figure 1. Evolution of the scattering length density profile with annealing time for the 20K molecular weight telechelic sample.



Experimental Report of Neutron Scattering Experiments at the FRJ-2 Reactor

Proposal number:	HAD-05-009		
Experiment title:	Understanding the structure of brushes and formed from imbedded functionalities		
Dates of experiment:	21.06. – 27.06.2005	Date of report:	22.06.2006
Experimental team: Names	Addresses		
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Experimental report text body

These reflectivity experiments were completed to elucidate the structure and properties of polymer brushes that are created from these imbedded functionalities and compare them to other polymer brushes. The experiments were completed on three polymer brush systems, which consist of deuterated polystyrene brushes that are approximately 50 nm long. The goal of the experiment is to determine the correlation between the ability of an initiator to grow a polymer brush to its depth in the initial film. The results from the three samples examined demonstrate that the three samples exhibit very different profiles for the deuterated polymer brush.

In the resultant density profiles, the first two clearly have significant amount of brush that grows from the initiator layer away from the interface, as the interface between the deuterated brush and protonated film is very broad. However, the third sample grows almost all chains from the protonated layer surface, as evidenced by the sharp interface between the layers. Clearly there exist fundamental differences in these films, however, they were synthesized to be very similar. We are currently working with our collaborators attempting to elucidate the cause for this observed variation in the brush structure. Clearly the results indicate that initiator may grow polymer chains from any depth, however, we must determine what parameters are important in controlling this behavior. Further experiments are planned to elucidate these factors.

Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	HAD-05-010		
Experiment title:	Magnetic field distribution in $\text{YBa}_2\text{Cu}_3\text{O}_7$ / $\text{La}_{2/3}\text{Ca}_{1/3}\text{MnO}_3$ multilayers		
Dates of experiment:	22.8. - 29.8.2005	Date of report:	December 9, 2005
Experimental team:			
Names:	Addresses:		
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Local contact:	Kentzinger Emmanuel		

Temperature dependent neutron reflectivity measurements showed an unexpected behaviour of the magnetic field distribution in superlattices consisting of $\text{YBa}_2\text{Cu}_3\text{O}_7$ (YBCO) and $\text{La}_{2/3}\text{Ca}_{1/3}\text{MnO}_3$ (LCMO) [1]. To get a better insight in this field distribution, measurements at different temperatures with polarized neutrons and spin analysis were performed on HADAS.

The sample consisted of eight double layers YBCO / LCMO on a SrTiO_3 substrate with layer thicknesses of 252 Å and 260 Å, respectively. It was first cooled to 5 K in an in-plane applied magnetic field of $H = 100$ Oe. A strong correlation of the magnetic roughness was observed out-of-plane and a weaker one in-plane of the layers. This correlations disappeared when heating the sample to $T > 130$ K (proposal number HAD-05-001).

The correlation of the magnetic roughness disappeared at a temperature $T' \approx 140$ K well separated from the transition temperature of the ferromagnet ($T_{\text{Curie}} \approx 240$ K) and the transition temperature of the superconductor ($T_c \approx 80$ K). Therefore the question arose whether T' represents a third phase transition in the superlattice or an effect induced by superconductivity at $T < T_c$, but preserved as particular magnetic ordering up to much higher temperatures than T_c .

In order to determine the temperature at which the magnetic correlations set in during cooling, further measurements on HADAS were performed: The same sample was subsequently cooled in an applied magnetic field of $H = 100$ Oe from room temperature to 160 K, 130 K, 90 K, 60 K and 5 K. No vertical correlation of the magnetic roughness was found as it was when heating, but the lateral correlation was found to increase independent of the temperature history at $T < T'$ (*Yoneda-like wings* in the $(+-)$ -channel) (Figure 1).

There are two possible explanations for the missing of the vertical correlation in the second experiment:

Either the *Bragg sheet* observed in the first experiment has been an artefact, or the lateral coherence length of the neutrons has been too small in the second experiment. The latter explanation is more likely, because the *Bragg sheet* has reproducibly been observed during the first experiment and because the diaphragm in front of the sample has been closer to the sample in the second experiment than in the first one. The decrease of the coherence length of the neutrons due to the shifted second diaphragm could be sufficient to oversee the vertical correlation. This would mean that the vertical correlation length is of the order of $25\text{ }\mu\text{m}$.

At $T > 90\text{ K}$ *Yoneda wings* were observed in the $(++)$ -channel. This means, that the upper LCMO-layer is not fully magnetized at higher temperatures, leading to an uncorrelated magnetic roughness at the surface. This effect occurs only when cooling.

Due to the smaller coherence length of the neutrons no vertical correlation was observed when cooling. To prove or disprove the independence of the history of temperature, this experiment should be repeated with a lateral coherence length of neutrons of more than $30\text{ }\mu\text{m}$ at the position of the second Bragg reflex.

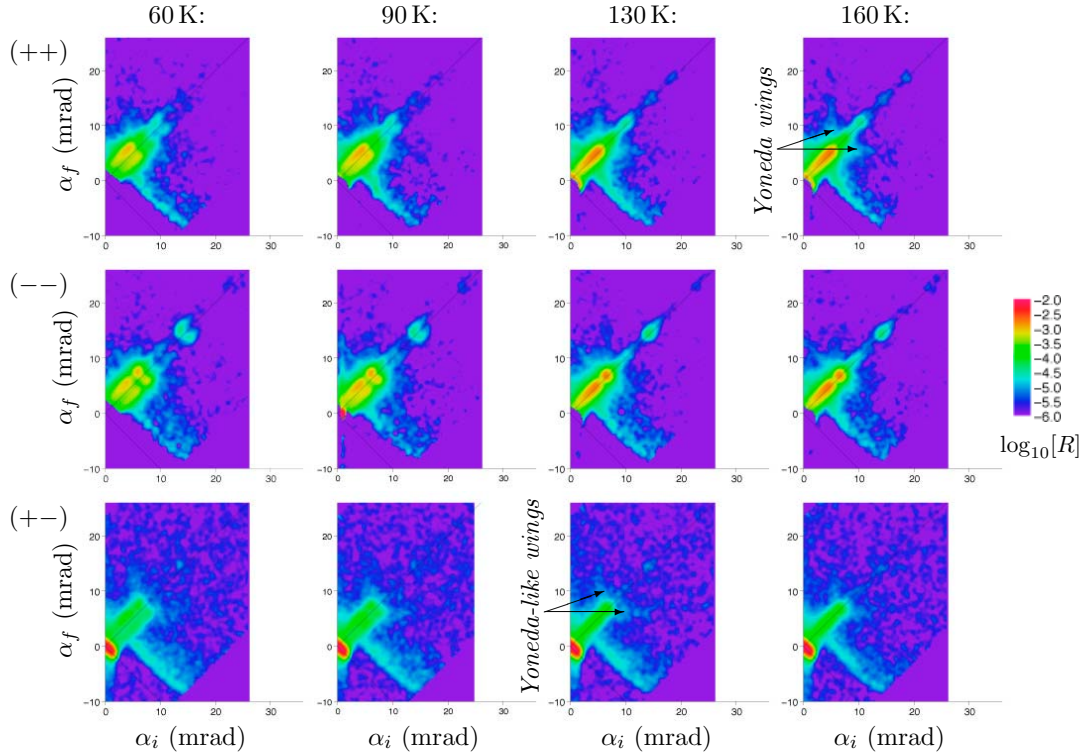


Figure 1: Reflectivity of a $[\text{YBCO} (252\text{ \AA}) / \text{LCMO} (260\text{ \AA})]_8$ multilayer in an applied magnetic field of $H = 100\text{ Oe}$ at different temperatures when cooling from room temperature. The two non-spinflip channels are shown with background subtracted, while the spinflip channel $(+-)$ is shown without background subtraction.

References:

[1]: J. Stahn *et al.*, Phys. Rev. **B**, 71, 140509(R) (2005)



Experimental Report of Neutron Scattering Experiments at the FRJ-2 Reactor

Proposal number:	HAD-05-015		
Experiment title:	Polarized neutron reflectometry on discontinuous metal insulator multilayers		
Dates of experiment:	30.09.2005 to 06.10.2005	Date of report:	06.06.2006
Experimental team: Names	Addresses		
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Local Contact:	Dr. A. Paul, Dr. E. Kentzinger, Dr. U. Rücker		

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Introduction :

Polarized neutron reflectivity (PNR) studies have been performed on granular multilayers $[\text{Co}_{80}\text{Fe}_{20}(t_n)/\text{Al}_2\text{O}_3(3\text{nm})]_{10}$ for the nominal thicknesses $t_n = 1.5$ nm (percolating ferromagnet). Most of the measurements were done at $T = 150$ K with a new cryostat and in the field range $0.47 \leq \mu_0 H \leq 12$ mT.

Motivation :

Several types of interlayer coupling have been studied theoretically and experimentally (e.g. [1]). In magnetic multilayer systems with insulating spacer layers and flat interfaces, dipolar interactions are dominant. They favor antiparallel orientation of the intra-planar magnetization between adjacent layers. For rough interfaces the so-called Néel or orange peel interlayer coupling has to be considered [2,3]. In 1962, Néel [2] pointed out that there should be ferromagnetic coupling between adjacent films due to magnetic dipoles at the interface induced by a correlated morphological corrugation. Competition between these dipolar interactions and Néel coupling may lead to an unusually modulated magnetization depth profile in a magnetic multilayer system.

Recently we have reported of such a magnetization depth profile in metal-insulator multilayers of $[\text{Co}_{80}\text{Fe}_{20}(t_n)/\text{Al}_2\text{O}_3(3\text{nm})]_9$ with $t_n = 1.6, 1.7$ and 1.8 nm [4,5]. Here we report on a study of the effect of the number (odd or even) of bilayers on the modulated magnetization depth profile in a magnetic multilayer system. To this purpose, we have studied a DMIM sample with $t_n = 1.5$ nm comprising an even number (10) bilayers.

Results and Discussions :

Fig. 1 (a) shows PNR data taken at positive saturation (12 mT). The FM state of the sample can be recognized by the splitting of the reflectivity edges where $R^{++}(\theta)$, is shifted to higher angles in comparison to $R^-(\theta)$. The first order structural superlattice Bragg peak at $2\theta = 4.9^\circ$ is dominant in the R^- -channel. Its position corresponds to a bilayer thickness $d \approx 5.3$ nm in agreement with X-ray results (data not shown here). The presence of a weak signal in the two SF channels R^{+-} and R^{++} is primarily due to the finite flipping ratio of 18 corresponding to the limited polarization efficiency of 94 %. The oscillatory behavior (Kiessig fringes) of R^{++} and R^- is due to the multilayer structure of the sample. The PNR data shown in Fig. 1(c) were taken at the weak guiding field (0.47 mT) of the neutrons after negatively saturating the sample. They are compatible with the negative remanence as indicated by the low angle data close to the plateau of total reflection, e. g. at $2\theta = 0.8^\circ$ (arrow), where $R^- > R^{++}$. The presence of a weak signal in the two spin-flip channels is primarily due to the limited efficiencies (94%) of both the polarizer and analyzer. As there is no significant spin-flip scattering, there is obviously no magnetization component perpendicular to the applied field.

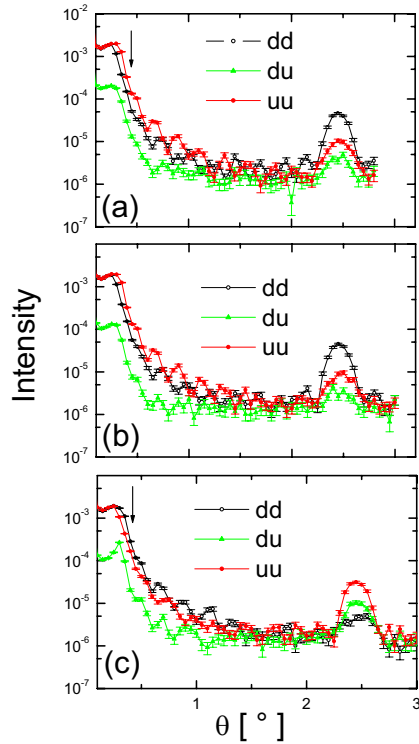


Figure 1: Spin-polarized neutron reflectivities R^{++} in red (dark gray), R^{-} in black and R^{+-} in green (light gray) vs. angle of incidence, θ , measured at $T = 150$ K and $\mu_0 H = 12$ mT (saturation) (a), close to the coercive field $\mu_0 H = 5.2$ mT (b), and close to remanence in the guiding field of the neutrons, $\mu_0 H = 0.47$ mT, after negative saturation (c) of ferromagnetic $[\text{Co}_{80}\text{Fe}_{20}(1.5\text{nm})/\text{Al}_2\text{O}_3(3\text{nm})]_{10}$.

Since this sample exhibited a sharp hysteresis, it was very difficult to measure at the coercive field. Fig. 1(b) shows a PNR measurement at a magnetic field a little higher than the coercive field. But as can be seen the field was sufficient to switch the magnetization to positive saturation. Therefore we were unfortunately not able to measure at the coercive field to observe the modulated magnetization depth profile as previously observed in other samples, which exhibit satellite peaks around the first Bragg peak. However, further attempts will be made on similar samples with even number of bilayers and rounded hysteresis such that it will be possible to measure at the coercive field.

Conclusions and acknowledgements :

Lack of spin-flip scattering suggests that magnetization reversal occurs via domain wall motion in this ferromagnetic sample. Unfortunately we did not observe any off-specular scattering from which one can calculate the mean domain size in the sample. This might be due to the resolution limit of the neutron beam, which cannot resolve domains being larger than $\xi \approx 30 \mu\text{m}$ or the limited intensity available. Further attempts will be made on similar sample with even number of bilayers to check the effect of number of layers on the magnetization depth profile.

We thank Dr. A. Paul, Dr. Emmanuel Kentzinger and Dr. Ulrich Rücker for their support at HADAS and we gratefully acknowledge Forschungszentrum Jülich for the financial support for the work at Jülich.

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2. L. Néel, C. R. Acad. Sci. **255**, 1676 (1962).
3. B. D. Schrag *et al.*, Appl. Phys. Lett. **77**, 2373 (2000); S. Tegen *et al.*, J. Appl. Phys. **89**, 8169 (2001).
4. S. Bedanta *et al.* Phys. Rev. B (in press).
5. S. Bedanta *et al.* (to be published).



Experimental Report
of Neutron Scattering Experiments
at the FRJ-2 Reactor

Proposal number:	HAD-05-016		
Experiment title:	Test of neutron optical devices for TREFF@FRM-II		
Dates of experiment:	21.09.2005, 8.11.2005	Date of report:	28.06.2006
Experimental team: Names	Addresses		
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Stefan Mattauch	JCNS-FRM II, FZ Jülich		
Local Contact:	Ulrich Rücker		

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Due to the shutdown of the FRJ-2 reactor, major parts of HADAS will be used to build the TREFF reflectometer at the FRM-II reactor in Garching as a test and characterization beamline for neutron optical devices. For setting up this instrument and being able to characterize it and to compare it with HADAS, we need to check some neutron optical devices before using them to build TREFF.

Fig. 1 and 2 show in comparison the polarized neutron reflectivity from a standard NiTi $m=2$ supermirror and a nonmagnetic $m=2$ supermirror, where Ni is replaced by a nonmagnetic Ni-V-alloy. Both mirrors have been obtained from S-DH GmbH, Heidelberg.

The intense spinflip signal (see Fig. 2) clearly shows that the standard supermirror strongly depolarizes the beam. Therefore, this kind of neutron mirrors is not suitable to transport a polarized beam in the instrument. In contrast to that, the spinflip signal in Fig. 1 is completely due to the imperfect polarization of the beam in HADAS. No depolarization is caused by the nonmagnetic supermirror. The reflectivity is well comparable with a standard supermirror.

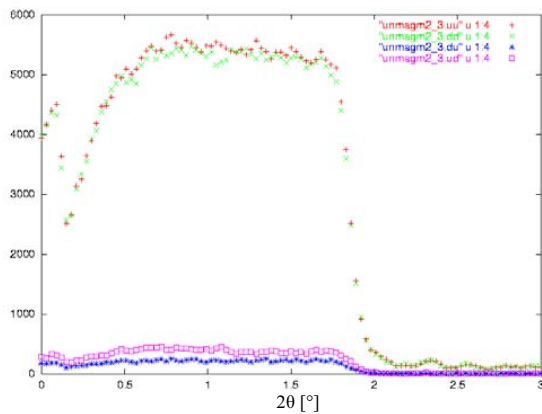


Fig. 1: PNR from a nonmagnetic $\text{Ni}_{1-x}\text{V}_x$ / Ti supermirror

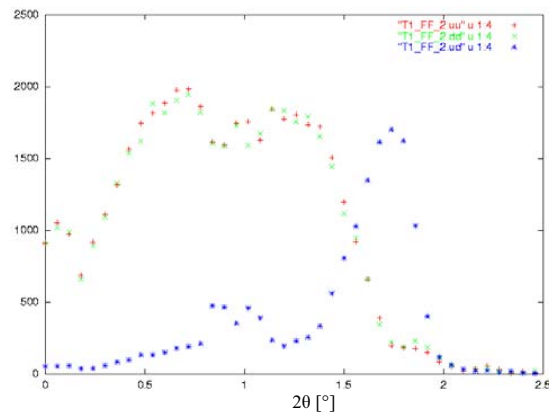


Fig. 2: PNR from a standard NiTi supermirror

Fig. 3 shows the reflectivity of unpolarized neutrons from a Ni coated glass plate, which is well bigger than the beam. It can be seen, that with an angular resolution of $\Delta\theta=0.4$ mrad, HADAS offers 5 orders of magnitude between total reflection and background. This experiment will serve as a reference for the characterization of TREFF.

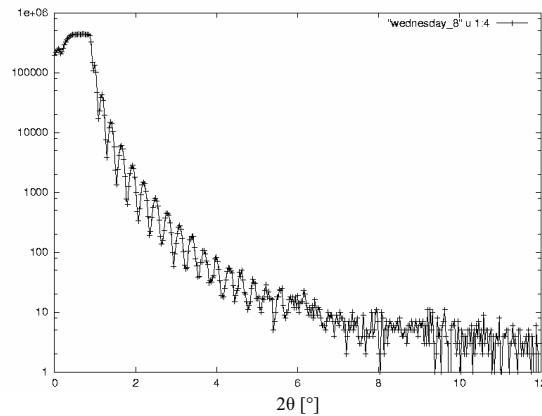


Fig.3: Reflectivity from a 80 nm thick Ni film on a glass substrate

In addition, several graphite crystals, a magnetic reference sample of 1 cm² size, and a polarizing supermirror which shows strong off-specular scattering were characterized to be able to compare TREFF's capabilities in different setups with HADAS.