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Physica B 267–268 (1999) 154–161

PHYSICA B

Polarized neutron reflectometry – a historical perspective

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Abstract

Born in the early 1980s to study magnetic films, polarized neutron reflectometry (PNR) has enjoyed growing popularity as witnessed by the number of instruments assembled at neutron research centers. PNR has proved its usefulness by providing information as diverse as the penetration depth of the magnetic field in superconductors and the absolute value of the magnetic moments in ultrathin ferromagnetic layers; yet its widest application has become the study of the magnetic configurations in multilayers. Two types of reflectometers have been constructed: time of flight and crystal analyzer. The relative merits of the two types are discussed in the light of present and future applications. © 1999 Published by Elsevier Science B.V.

PACS: 75.70. − i; 07.60.Hv; 74.76. − w

Keywords: Neutron reflectivity; Polarized neutrons

1. How it started

Polarized neutron reflectivity (PNR) has reached a maturity perhaps surprising in view of its young age. Born in the middle 1980s, it was devised as an analytic tool to measure the magnetic depth profile of thin films or in proximity of surfaces and interfaces. Fortunately, its deployment was paralleled by the evolution of techniques capable of producing reliable magnetic films with novel magnetic properties. Maturity has come to PNR in two ways: its role in research has become considerably better defined, and the results obtained by different laboratories have become quite consistent. Goal of this report to give a current perspective focussing on the work done during the past years. I discussed previous accomplishments in an earlier review [1]; several other reviews have since appeared [2–7].

Neutron reflectivity is an optical technique: the interaction of neutrons with the medium through which they propagate is described by a potential whose magnitude is related simply to the scattering length density of the nuclei and the magnetic induction B in the material:

$$V_{\text{eff}} = V_{\text{n}} + V_{\text{m}} = \frac{2\pi\hbar^2}{m} bN + \boldsymbol{B}\cdot\boldsymbol{s}, \qquad (1)$$

where b is the mean of the scattering lengths over the N atoms occupying a unit volume and s is the neutron spin. The trajectory of the neutron in this potential is obtained by solving the Schrödinger

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0921-4526/99/$ – see front matter © 1999 Published by Elsevier Science B.V.
PII: S0921-4526(99)00053-8

equation. If V_{eff} is a function only of the depth from the surface (as in a stratified medium) only the z-component of the motion perpendicular to the surface is affected by it: the motion in the plane x, y (parallel to the surface) is that of a free particle. Let us assume for simplicity that B is parallel to the neutron spin and parallel to the reflecting surface. After separating the variables, the Schrödinger equation along z reduces to

$$f''_{\pm} + [k_0^2 - 4\pi(bN \pm cB)]f_{\pm}, \tag{2}$$

where $k_0 = 2\pi \sin\theta/\lambda$, and θ is the angle of the neutron beam with the surface, λ the neutron wavelength.

For any layer n in the medium for which the potential is constant the solution of Eq. (2) is given by the sum of two exponentials, giving the flow of neutrons in either direction of z:

$$f_{\pm} = t_{\mathrm{n}}^{\pm} \exp(\mathrm{i}k_{\mathrm{n}}^{\pm}z) + r_{\mathrm{n}}^{\pm} \exp(-\mathrm{i}k_{\mathrm{n}}^{\pm}z), \tag{3}$$

where

$$k_{\mathrm{n}}^{\pm} = \sqrt{k_0^2 - 4\pi(bN \pm cB)}.$$

The coefficients of the two exponentials are determined by the conditions of conservation of matter and flux at each boundary. The spin-dependent reflectivities are simply of the form $R = |r_0|^2$, where r_0 is the coefficient of the second term in Eq. (3) for the region above the surface. In general the reflectivity is unitary for most materials up to values of $q_z = 2k_0$ of the order of 0.01 $\mathrm{\AA}^{-1}$, and decreases rapidly beyond that limit with an asymptotic q_z^{-4} dependence. We have shown the simplest magnetic case – all magnetic fields are collinear. Even in more complex cases it is straightforward to calculate exactly the reflectivities from the potential. This procedure is to be followed close to the critical value of q_z: at larger q_z the reflectivities are well approximated by the scattering theory in the first Born approximation. When in the spinor equations f_+, f_- cannot be separated, spin-flip processes take place.

A reflectometer is a simple instrument (Fig. 1) [8]: a neutron beam of wavelength λ hits a sample surface at an angle θ and is reflected from the surface at the same angle θ. The instrument is

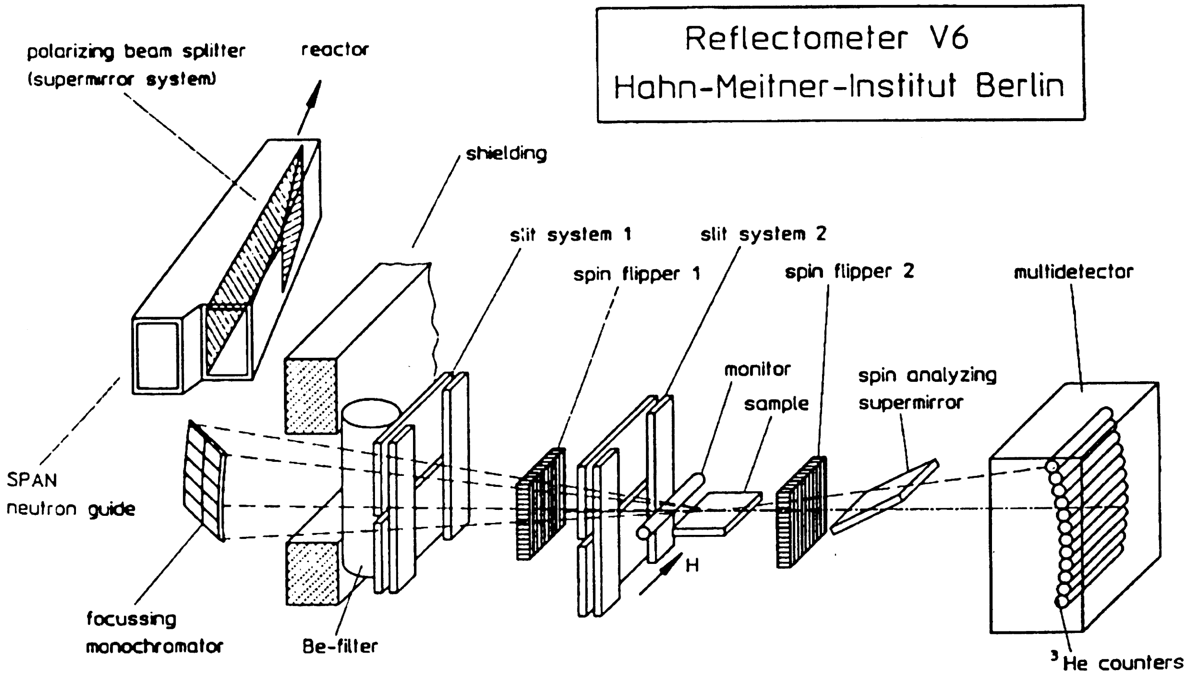

Fig. 1. Scheme of a crystal analyzer reflectometer (Ref. [8]).

practically a diffractometer with resolution sufficient to separate transmitted and reflected beams at values of q_z where the reflectivity becomes unitary. Reflectometers have been constructed at both steady state and pulsed neutron sources. The reflectivity is solely a function of the momentum transfer along the z direction, $q_z = 4\pi \sin\theta/\lambda$: a range of q_z can be spanned either by changing the wavelength, and keeping fixed the angle of incidence, or by changing the angle of incidence at fixed wavelength. In the former mode the wavelength is selected by Bragg-reflecting the beam from the moderator with a crystal analyzer (CA). In the second mode the neutron beam is chopped in short pulses: the neutron wavelength is sorted out by the time of flight (TOF) from chopper to detector. Reflectometers built at steady-state sources are of both kind, while at pulsed sources only TOF reflectometers are feasible. Both types of instruments have distinct advantages. In a TOF instrument a substantial region of q_z is covered simultaneously, without changing the footprint on the sample, by the entire neutron spectrum. On the other hand, with a CA instrument one can use all available neutrons to measure the intensities at a selected value of q_z. The choice of the best instrument is thus dictated by the experiment to be performed.

Appropriate devices are added to the reflectometer to polarize the incoming neutrons along an applied magnetic field or to analyze the polarization of the reflected beam (Fig. 1). Conventionally, the direction of initial polarization is fixed. The sample may change the polarization of the neutron; yet a conventional analyzer chooses, among the reflected neutrons, those polarized along the same direction as the polarizer. Reversal of the neutron spin is obtained by energizing flippers placed before and after the sample. The reflectivities are then characterized by the sign of the neutron polarization before and after reflection with respect to the reference field: R^{++}, R^{+-}, R^{-+}, R^{--}. As an example, in Fig. 2 are presented the reflectivities from a multilayer of Fe/Cr in which the magnetization of subsequent Fe layers was suspected to be non-collinear [9].

When spin-flip occurs, the interpretation of the entire q_z range of spin-dependent reflectivities in terms of a magnetic structure is not always transparent, although it might be so in particular cases. The intensities of the diffraction peaks can be analyzed in terms of the kinematical theory. For instance, the bottom figure shows an AF peak at $q_z = 0.045\ \text{Å}^{-1}$. Its intensity is proportional to:

$$I^{++} = I^{--} = |M_{\parallel}|^2;\ I^{+-} = I^{-+} = |M_{\perp}|^2, \tag{4}$$

where $M_{\parallel}$, $M_{\perp}$ are the projections of the staggered magnetization parallel and perpendicular to the neutron quantization axis. After making a similar analysis on the ferromagnetic peak (at $q_z = 0.09\ \text{Å}^{-1}$) the angle between the two sublattice magnetizations has been obtained. This kind of analysis does not apply to smaller values of q_z. As it can be observed in Fig. 2, R^{+-} decreases with q_z in the total reflection region; it is easy to show analytically that $R^{+-} \to 0$ when $q_z \to 0$. Since the relationship between spin-dependent reflectivities and spin structure is not transparent, details of the non-collinear structure are obtained by model fitting.

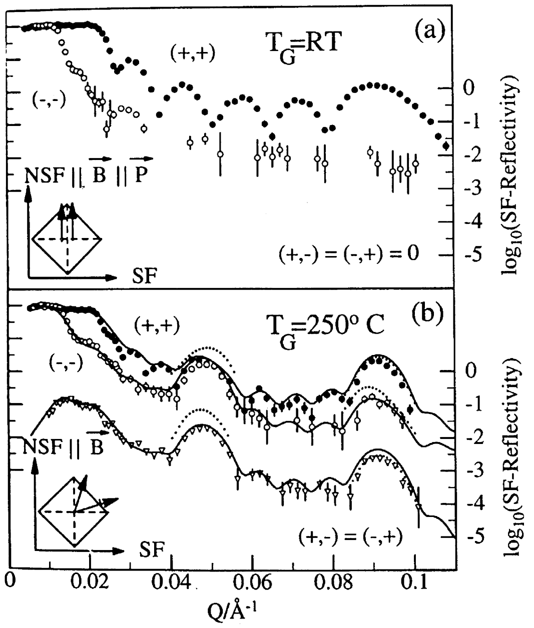

Fig. 2. Spin-dependent reflectivities from two samples [Fe(52 Å)/Cr(17 Å)] × 5 grown at different temperatures T_G. For $T_G = 250°\text{C}$ the magnetization of subsequent Fe layers is at an angle of 50°. From Ref. [9].

Table 1
Polarized neutron reflectometers in the world

Instrum.	Source	Start up	Mode	Beam size (mm)	Q range $\mathrm{\AA}^{-1}$	Pol. An.	Comments
POSY I	IPNS, Argonne	1984	TOF	3×40	0.5	Yes	
CRISP	ISIS, Rutherford	1986	TOF	10×40		Yes	
BT-7	NIST Reactor	1990–1996	CA				In confinement building
NG-1	NIST Reactor	1996	CA	7×50	2.2	Yes	In guide hall
AMOR	SINQ	1998	TOF	50×50		Yes	
SPEAR	LANSCE, Los Alamos	1991	TOF	8×50		Yes	Part time in the polarizing mode
ROG	IRI, Delft	1988	TOF	5×30		No	
EVA	ILL Grenoble	1987	CA	30×30		No	For diffraction at grazing incidence
SPN	FLNP Dubna	1988	TOF	3×60		Yes	
G2-2	Orphée, Saclay	1995	CA				
ADAM	ILL Grenoble	1997	CA	15×40		Yes	
REFLEX	FLNP Dubna	1997	TOF			No	
V6	HMI Berlin	1992	CA	10×50	1.5	Yes	
V14	HMI Berlin	1996	CA	4×50	1.5	Yes	
PORE	KEK Tsukuba	1999	TOF	10×30	0.4		
	JAERI	1996	CA				Interferometer
D-17	ILL Grenoble	1999	Variable	30×70	1.0	Yes	

Eq. (4) indicates that polarizers and flippers must have very good efficiency to define accurately the direction of the magnetization. For instance, if the uncertainty in the polarization is 1%, from a purely magnetic signal the direction of the magnetic moments is determined only within 6°. It is also clear that good efficiency is more difficult to achieve for TOF instruments, which use an extended range of wavelengths. Yet the recent years has seen a sustained effort in fabricating efficient polarizers [10–12] and flippers [13]. Table 1 gives a compendium of the polarized neutron instruments [14–16] built up to now. The technical effort in constructing them is paralleled by the expansion of the scientific program.

2. Current research

2.1. Penetration depth in thick superconducting films

The penetration depth characterizes completely the diamagnetism of a film for applied magnetic fields below a critical field H_{c1}, above which an inhomogeneous state is created (in type-II superconductors) with the magnetic field penetrating along lines of fluxoids. With the magnetic field applied perpendicularly to the surface, arrays of fluxoids terminating at the surface have been observed by surface sensitive techniques. With the field parallel to the surface the fluxoids may be entirely within the material, and in this condition a penetrating probe (as neutron reflection) needs to be used. With regards to the penetration depth, the results obtained by different laboratories have been satisfactorily converging. This is not only true of conventional superconductors, like niobium [17,18], but also of the high T_c superconductor $\mathrm{YBa_2Cu_3O_{7-x}}$ where the measurements point to a penetration depth of the order of 1400 Å [19,20], in good agreement with the results obtained by muon spin rotation and by neutron scattering from a fluxoid lattice perpendicular to the surface.

The magnetic configurations above H_{c1} are still under discussion. From transport measurements it appears that the configuration of fluxoids is not universal, but depends heavily on the anisotropy of the coherent lengths, the thickness of the superconducting layers and the amount of the pinning centers. The anisotropy is reduced to shape anisotropy in a single film of niobium. Material anisotropy can be introduced by layering thin film of superconductor with metallic spacers (an extreme case is that of epitaxially grown high T_c materials). In all cases pinning centers may give rise to a disordered distribution of fluxoids not aligned with the field but straggling the film: the magnetic response is then basically described by the Bean model [21]. In the absence of pinning centers fluxoids should order into lattices. When the anisotropy is extreme, the fluxoid currents are expected to be located principally within the superconducting layers to minimize the tunneling through the non-superconducting layers (Josephson vortices). For a less anisotropic medium a different organization of fluxoids has been suggested [22]. Above H_{c1} a single row of fluxoids is formed with spacing d at the center of the film to minimize the repulsion from either surface; this splits into two rows above a second critical field.

A one-dimensional lattice should give rise to an off-specular diffraction line with exit angle θ_f:

$$(1/d) = (\cos\theta_f - \cos\theta_i)/\lambda, \tag{5}$$

as derived from the conditions of conservation of energy and momentum for the neutrons. In practice, geometrical conditions severely restrict the observable d spacings, and the intensity of the diffraction line is expected to be very weak anyway. Up to now, the presence of a fluxoid lattice has been inferred only from the spin dependence of the reflectivity [20,23]. The effect of fluxoids on the reflectivities depends on their concentration as a function of z. If pinned at random, their effect would be detected just close to the value of q_z corresponding to total reflection. Instead, a line of fluxoids close to the center gives rise to a maximal spin dependence of the reflectivity at $q_z \sim 2\pi/(D/2)$ (D is the total layer thickness). An array of Josephson fluxoids in a multilayer should exhibit a maximal spin dependence of the reflectivity at the Bragg reflections of the multilayer.

2.2. Very thin films

For a film thickness below a few nanometers the magnetization of a ferromagnet is significantly altered from the bulk value in size, direction of magnetization and even type of magnetic order. These new properties are the result of a complex set of circumstances, such as the incomplete quenching of the orbital moments, the stretching (or compressing) of the lattice on the substrate, and the transfer of electrons between magnetic film and the substrate. Polarized neutron reflection has been used to determine the absolute value of the magnetic moment per atom (notably in Fe and Co) in very thin films, and its increment compared to the bulk values [24–28]. The results are in good agreement with those theoretically predicted as well as those obtained by alternative techniques recently developed [29,30].

Magnetic bilayers have also been studied. When in contact, the magnetization vectors of two layers, one of gadolinium, one of iron are oppositely aligned; however, in the presence of a magnetic field the softer exchange interaction within the gadolinium layers gives rise to twisted states [31,32]. The interaction between two layers of iron, interleaved with a metallic spacer, is strongly dependent on the nature and the thickness of the spacer, as studied in Fe/Cr/Fe [33] and Co/Cu/Co [34]. If two magnetic layers are unequal in thickness, chemistry or because one is anchored to an antiferromagnet, the system may behave as a spin-valve in the presence of a magnetic field. PNR allowed the study of the layer by layer magnetization of such composites [35,36].

2.3. Magnetic multilayers

First for very selected couples, then for a rapidly host of combinations of Fe, Co, Ni interleaved by most of the 3, 4, 5d non-magnetic metals it was found that the coupling between subsequent ferromagnetic layers oscillates from ferromagnetic (FM) to antiferromagnetic (AF) as the thickness of the non-magnetic spacers is varied. Magnetic fields ranging from a few tens to a few thousand Oersteds align the overall magnetization of AF multilayers, with a large change of magnetoresistance. The magnetic structure of the AF state has been observed directly by PNR and found to be of the type $+-+-$, with a simple doubling of the chemical periodicity. A number of papers have appeared to confirm this magnetic configuration in several systems, study the pattern of antiferromagnetic domains, their evolution with the onset of a magnetic field and its correlation with the magnetoresistance [37–42]. Analysis of the polarization of the reflected neutrons has been used to determine the direction of the magnetization as a function of depth. In this way it was confirmed the presence of 90° magnetic configurations for weak interlayer coupling, as justified if biquadratric terms in the magnetic exchange become important [9,43–47].

Less studied, but of growing interest, are multilayers with rare-earth spacers [48–52]. Multilayers of rare-earth/Fe or rare-earth/Co are inherently imperfect, in view of the large degree of mismatch of the lattice spacing of the two components. Such mismatch is, however, greatly reduced when the rare earth is hydrogenated. At the same time, the hydrogenation changes reversibly the band structure and metallic character of the material containing the rare earth, by an amount controllable with the hydrogen pressure. This line of research has just started; however for Nb/Fe, V/Fe superlattices it has been shown [53,54] that hydrogenation can switch reversibly the AF and FM states.

3. The outlook

Even from this incomplete review it becomes evident that the bulk of the experimental work concerns the study of magnetic multilayers. To determine the details of the magnetic profile of the single repeat unit a large q_z region needs to be explored. At large q_z the signal of the reflected beam is practically zero, except at Bragg reflections which, for a typical bilayer thickness of a few tens of Ångstroms, appear at intervals $\Delta q_z \sim 0.5\ \text{Å}^{-1}$. Moreover, in epitaxially grown materials, diffraction lines due to the mean lattice spacing appear at $q_z \sim 6\ \text{Å}^{-1}$. This line of research actually antedates the development of reflectometry. For instance in superlattices of the type Gd/Y [55] large angle diffraction lines have been measured in the middle 1980s to see the occurrence of magnetic dead layers at the Gd/Y interfaces. More recently, in studying Fe/Cr superlattices, the link between the magnetization of Fe and the spin density wave in chromium was found by observing the magnetic satellites around the (0 0 1) diffraction line of chromium [45,56]. Common to all these endeavors is the requirement that the intensity be measured at a few, widely spaced points of q_z. To study these a reflectometer/diffractometer at fixed wavelength is the optimal instrument.

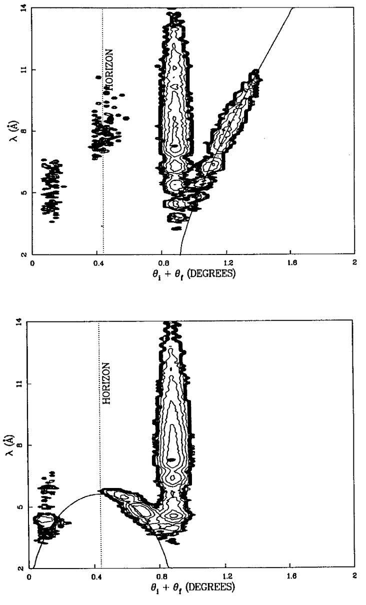

Fig. 3. Logarithmic contour plots of neutron intensities reflected from a cobalt film for an angle of incidence $\theta_i = 0.44°$. The bottom and the top pictures represent plots for neutrons polarized parallel and antiparallel to a magnetic field $H = 13$ kOe applied perpendicular to the surface. From Ref. [59].

Is there a future for the TOF reflectometry? As already stated, this instrument becomes preferable when the objective is to cover a substantial region of neutron momentum transfers. This is the case when some magnetic scattering is off the reflection line. Inhomogeneities in the plane of the film give rise to scattering which appears in a two-dimensional counter at an angle θ_f different from θ_i and at an angle ϕ away from the plane of reflection. If an FM or an AF multilayer is composed of in-plane domains, the magnetism is no longer uniform in the plane of the film, and the finite size of the domains gives rise to scattering around the direction of the reflected beam. This has been repeatedly observed [1,57,58]; from the width of diffuse scattering the domain size has been deduced. As visually shown by A. Fermon in the following paper, patterned magnetic structures give rise to a rich spectrum of diffraction lines.

Off-specular scattering of magnetic origin has been observed even in the absence of inhomogeneities, as a result of a spin-flip process in a magnetic field. In the experiment [59–61] the reflecting sample was a film of ferromagnetic cobalt. A magnetic field H, applied perpendicular to the film, was sufficient to provide a quantization axis to the neutrons but not to turn entirely the Co magnetization out of the plane. Some of the reflected neutrons flipped their spin, and their potential energy was changed by the amount of the Zeeman splitting. The laws of conservation of energy and momentum for the spin-flipped neutrons impose the condition $\theta_f^2 = \theta_i^2 \pm 1.47 \times 10^{-7} H \cdot \lambda^2$, where θ_i, θ_f are respectively the incident and reflected angles in radians, H is expressed in kOe and λ in Å. Neutrons reflected and spin-flipped were found to be reflected at an angle significantly different from the angle of incidence (Fig. 3) even in fields of a few kOe, although the Zeeman splitting energy amounts to less than 10^{-7} eV.

The examples above indicate that a fertile ground exists for an instrument of the TOF type, that can make simultaneous observations over a large span of scattering vectors. How wide is this field of research is a question that only the future “historians” will be able to answer.

Acknowledgements

My work was supported by US-DOE, BES-MS contract 31-109-ENG-38. I would like to thank Drs. F. Klose, J. Ankner and A. Schreyer for permission and help in reproducing their figures.

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