Journal of Magnetism and Magnetic Materials 350 (2014) 199–208

Contents lists available at ScienceDirect

Journal of Magnetism and Magnetic Materials

journal homepage: www.elsevier.com/locate/jmmm

Current Perspectives

# Neutron scattering—The key characterization tool for nanostructured magnetic materials

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ARTICLE INFO

*Article history:*
Received 20 September 2012
Received in revised form
10 January 2013
Available online 20 September 2013

*Keywords:*
Nanomagnetism
Neutron scattering
Magnetic interfaces

ABSTRACT

The novel properties of materials produced using nanoscale manufacturing processes often arise from interactions across interfaces between dissimilar materials. Thus, to characterize the structure and magnetism of nanoscale materials demands tools with interface specificity. Neutron scattering has long been known to provide unique and quantitative information about nuclear and magnetic structures of bulk materials. Moreover, the specialty techniques of polarized neutron reflectometry and small angle neutron scattering (SANS) with polarized neutron beams and polarization analysis, are ideally and often uniquely suited to studies of nanostructured magnetic materials. Since neutron scattering is a weakly interacting probe, it gives quantifiable and easily-interpreted information on properties of statistically representative quantities of bulk, thin film and interfacial materials. In addition, neutron scattering can provide information to complement that obtained with bulk probes (magnetization, Kerr effect) or surface measurements obtained with scanning probe microscopy or resonant soft x-ray scattering. The straightforward interpretation and the simultaneous availability of structural information, make neutron scattering the technique of choice for the structural and physical characterization of many novel materials, especially those with buried interfaces, ones allowing for isotopic substitutions to decorate buried interfaces, or cases where the magnetic response to an external stimulus can be measured. We describe recent applications of neutron scattering to important thin film materials systems and future opportunities. Unquestionably, neutron scattering has played a decisive role in the development and study of new emergent phenomena. We argue with the advent of new techniques in neutron scattering and sample environment, neutron scattering's role in such studies will become even more dominant. In particular, neutron scattering will clarify and distinguish between intrinsic *vs.* extrinsic origins of unusual behavior which invariably plague novel materials. Key to realizing these opportunities will be the development of sample environment capabilities especially tailored to test the origins of novel phenomena, and techniques to collect, analyze and correlate neutron event detection with time dependent perturbations to the sample's environment.



## 1. Introduction

The discovery of new materials and the development of advanced materials preparation techniques and characterization tools have historically enabled the discovery of new phenomena. As a simple example, consider the time line starting with the discovery of superconductivity of elements, to compounds such as $Nb_3Ge$ and $Nb_3Sn$, to complex high temperature superconducting oxides and most recently to Fe-pnictides. The latest discovery called into question conventional wisdom that superconductivity and magnetism were mutually exclusive; in fact the co-existence of both order parameters can be mutually beneficial. With appreciation of the importance of competing order parameters, our attention is drawn toward development of new technologies based on, for example, colossal magnetoresistance or multiferroic behavior in complex oxides. Such behavior has been observed in bulk materials. Moreover, films grown with vapor deposition techniques enable the design and engineering of atomic, orbital and chemical structures and interfaces to promote specific functional behavior that offer many more opportunities [1] and new ground states that can only be realized at or near interfaces. A focus of this perspective is to show how neutron scattering, in particular polarized neutron reflectometry and small angle neutron scattering, has been used to clarify outstanding issues in nanostructured magnetic materials and especially evolution of the techniques to resolve the origins of magnetism that arise from interfaces.

Composite materials have long been important constituents of commercial products, and with improvements in the synthesis and characterization of materials at the nanoscale the importance

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http://dx.doi.org/10.1016/j.jmmm.2013.09.028

of interfaces becomes preeminent. Nanoscale materials necessarily imply that interfaces play a decisive role in determining the response of composites to their environment. This is particularly true of thin films which exhibit behavior different from the bulk owing to the (a) high surface-area-to-volume ratio, (b) growth method, (c) issues relating to epitaxial strain, misfit dislocations, twin and low angle grain boundaries and (d) coupling or interactions across interfaces of dissimilar materials, such as those giving rise to physical properties of the interface that do not exist in the constituent materials.

Many systems are composites of materials with dissimilar properties. The resultant behavior of the composite can be simple averages of the properties of its constituents, *e.g.*, hardness and toughness of ceramic-metal composites, or produce new behavior due to such factors as competing order parameters [2]. Examples include: the (a) competition between ferromagnetism and superconductivity in La–Sr–Mn–O (LSMO) and Y–Ba–Ca–O (YBCO) superlattices, and (b) formation of two-dimensional-electron-gas (2DEG) across interfaces with discontinuous (charge) polarizations, exchange bias, *etc.* Competition of order parameters requires the different components to be in intimate contact. This places metrics on the perfection (chemical and structural) of the interfaces that is governed by the coherence one order parameter into the *other* (opposing) material. For the case of magnetism, perfection may be required over a length scale equal to the exchange length (equivalent to the nearest neighbor distance). Alternatively, if the magnetism is controlled by the crystal structure, *e.g.,* via modification of spin–orbit coupling due to rotation of octahedra in a perovskite, the appropriate length scale may be a few unit cells [3]. The need for perfection poses significant challenges. Challenges for sample growth (*e.g.,* to mitigate or control diffusion across interfaces); challenges for materials characterization (*e.g.,* to measure structure and properties of interfaces confined to small volumes), and challenges for modeling (*e.g.,* to include materials science realities of diffusion and defects in calculations).

Before addressing possible effects due to interesting intrinsic physical phenomena, it is important to distinguish these from extrinsic effects. The types of extrinsic effects which are very important and will invariably appear when two materials are in intimate contact include: roughness, interdiffusion, oxygen deficiency, lattice expansions. Since the properties of materials depend very delicately on many structural and chemical parameters, it is crucial to understand all these parameters quantitatively at the atomic level. Neutron scattering has a major strength to address this challenge, since it simultaneously provides chemical and magnetic structural information at the atomic and nanoscales [4].

As an example, the electrical conductivity at the interface between $LaAlO_3$ (LAO) and $SrTiO_3$ (STO) single crystal films is greatly enhanced [5]. This observation was extraordinary since in the bulk LAO and STO are insulators, but the interface between them might take on a new electronic character or property. However, the origin of conductivity and magnetism in LAO/STO heterostructures is far from settled. For instance, oxygen vacancies at the LAO/STO interface may be responsible for the novel properties arising from the pulsed laser deposition process instead of more exotic mechanisms. Recent reports suggest that excess oxygen may migrate into the LAO film from the STO substrate, or that cation diffusion across the interface may contribute to the conductivity [6,7]. These types of controversies surrounding the discovery of novel physical phenomena can only be resolved by synergetic efforts between synthesis and quantitative structure and property characterization.

It is abundantly clear that no single tool can completely characterize local structure and properties of an interface especially if the interface is buried. Moreover, no technique yields a comprehensive characterization that is representative of the entire sample or interface. Rather, progress in interfacial science requires integration of results from a myriad of techniques, modeling and the development of experimental protocols to test the response of interfaces to external stimuli as a means to understand properties of imperfect interfaces.

In this perspective we describe strategies, requirements and new opportunities to characterize magnetic thin films and interfaces with neutron scattering. We focus on small angle scattering of neutron beams in transmission (SANS) or reflection (neutron reflectometry or NR) geometries. Because the scattering takes place reasonably close to the origin of reciprocal space, SANS and NR detect variation of nuclear and magnetic scattering from nm-to-micron length scale inhomogenities. In the case of NR, the scattering occurs from nearly planar interfaces or patterned surfaces. SANS probes the nuclear and magnetic contrast across interfaces that are not necessarily planar, *i.e.*, SANS is ideal for studies of nanomagnetic systems in three dimensions. Both techniques are sensitive to magnetism even without polarized neutron beams; however, a polarized beam incident on the sample provides additional information about the correlation between nuclear and magnetic structures. Polarization analysis of the scattered beams, removes this constraint, thus, making possible rigorous separation between nuclear and magnetic scattering.

## 2. Techniques

### 2.1. Polarized neutron reflectometry

Polarized neutron reflectometry (PNR) is ideally suited to measure the nuclear and magnetization depth profiles across planar interfaces. Because reflection only occurs when either or both of the nuclear or magnetic compositions change across an interface, the technique is intrinsically sensitive to interfacial magnetism. Thus, PNR can distinguish magnetism at an interface from contamination within a substrate or on the sample. As such, the concerns raised by Garcia [8] in attribution of magnetism to nanoscale structures from bulk magnetometry are not germane to PNR. Extensive reviews of PNR can be found in Refs. [9–11].

Briefly, in PNR the intensity of the specularly reflected neutron beam is compared to the intensity of the incident beam as a function of wavevector transfer, $Q$ ($=4\pi\sin\theta/\lambda$ ), where, $\theta$ is angle of incidence and $\lambda$ is neutron wavelength), and neutron beam polarization (Fig. 1). Typically, $Q$ is changed by either changing $\theta$ (typically of order 1°) or $\lambda$ (typically of order 0.2–1.4 nm). The specular reflectivity, $R$, is determined by the neutron scattering length density (SLD) depth profile, $\rho(z)$, averaged over the lateral dimensions of the sample. The SLD is the depth dependent variation of the index of refraction of the sample. The (kinematic) reflectivity is related to the square of a Fourier transform of the SLD profile. $\rho(z)$ consists of nuclear and magnetic SLD's such that $\rho^{\pm}(z)=\rho_n(z)\pm CM(z)$, where $C=2.9109\times10^{-9}$ Å$^{-2}$ (kA/m)$^{-1}$, and $M(z)$ is the magnetization (a moment density in kA/m) depth profile [10,11]. Unlike non-resonant x-ray scattering, the nuclear and magnetic neutron scattering lengths have comparable magnitudes. The $\pm$ sign in $\rho^{\pm}(z)$ denotes neutron beam polarization parallel (opposite) to the applied field and corresponds to spin-dependent reflectivities, $R^{\pm}(Q)$. Thus, by measuring $R^{+}(Q)$ and $R^{-}(Q)$, $\rho_n(z)$ and $M(z)$ can be obtained separately. If the net magnetization is rotated away from the applied field, polarization analysis of the specularly reflected beam provides information about the projection of the net magnetization vector onto the sample plane. For example, polarization analysis with PNR is useful for identifying the easy axes of different magnetic layers.

Requirements for PNR experiments include: extremely smooth samples ( $<1$ nm root-mean-square roughness), uniformity of film

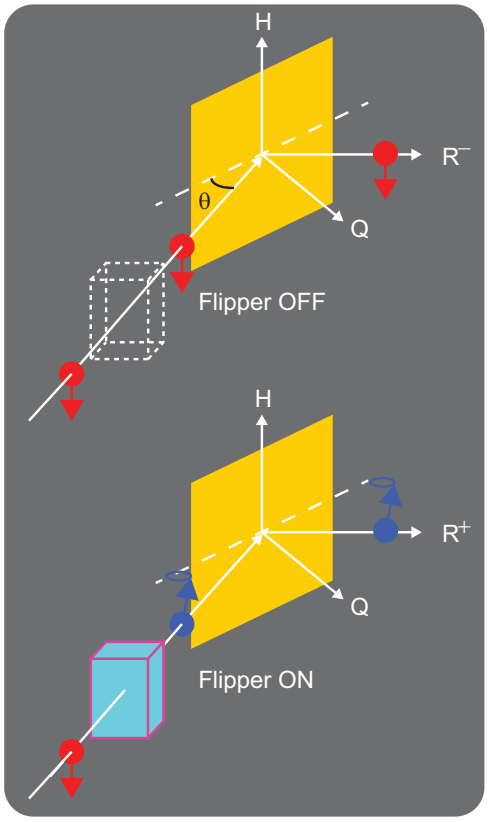


**Fig. 1.** Schematic diagram of a polarized neutron reflectometry experiment. A polarized neutron beam (polarization shown down in the upper figure) strikes a sample at an angle of incidence $\theta$, and then reflects from it. The difference between the incoming and outgoing wavevectors is the wavevector transfer $Q$. The intensity of the reflected beam is measured relative to the incident beam intensity to obtain the spin down reflectivity $R^-$ as a function of $Q$. (lower) A neutron spin flipper is turned on to reverse the polarization of the neutron beam. In this example, the spin-up reflectivity $R^+$ is measured.
Figure courtesy of K. O'Brien.

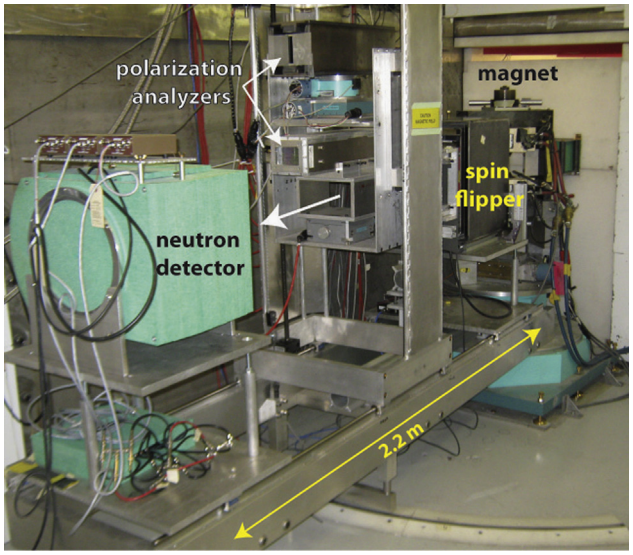


**Fig. 2.** Picture of the Asterix reflectometer-diffractometer at the Manual Lujan Jr. Neutron Scattering Center. The sample is located between the pole pieces of the magnet.

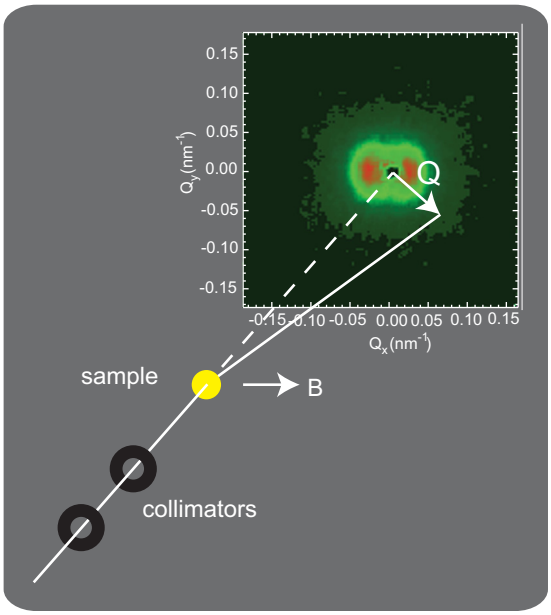


**Fig. 3.** Schematic of a small angle neutron scattering experiment. SANS experiments can also be performed with polarized beams and polarization analysis of the scattered beam.

thickness (variation $<5\%$), large area samples (typically $1+$ cm$^2$), access to user facilities, and patience (trips typically last a week or so). The lower limit for detection of magnetization is of order 1 kA/m. The most successful experiments are generally ones exploring magnetization of tens of kA/m. The ability to detect very thin magnetic layers is usually proportional to the largest $Q$ value (exceptions to this rule-of-thumb can be realized with specially tailored film geometries). This in turn is determined by the sample quality, the magnitude of the magnetization, instrument background and neutron flux. Typically variation of the magnetization depth profile occurring over length scales of 1–2 nm is significant.

Sample environments for NR and SANS include cryomagnets providing fields as large as $11+$ $T$ and temperatures as low as 300 mK, pressure cells, mechanical devices (to apply bending stress to substrates), and facilities for irradiation with light. An example of a polarized neutron reflectometer is shown in Fig. 2.

### 2.2. Small Angle neutron scattering

SANS (Fig. 3) is ideally suited to measure the change of nuclear and magnetization contrast across non planar interfaces. SANS provides information about regions, domains and particles bounded by interfaces across which either the nuclear or magnetic scattering length density changes. The length scales probed by SANS vary between 10′s of nm to microns. Perhaps counter intuitively, it is easier to measure shorter length scales because the SANS is furthest from the very intense primary (*i.e.*, unscattered) neutron beam. Like NR, polarized neutron beams allow measurements of the correlation between nuclear and magnetic SANS. However, since domains producing magnetic SANS maybe uncorrelated with the features giving rise to nuclear SANS, polarization analysis can be critically important, and moreover, polarization analysis permits rigorous separation of nuclear and magnetic SANS. Confinement of scattered intensity in NR about the specular reflectivity allows the use of supermirrors for polarization analysis with relative ease. However, use of mirrors is very challenging for polarization analysis of the widely divergent SANS. The availability of $^3$He cell polarizing filters (Fig. 4) has enabled polarization analysis for SANS and successful exploitation of SANS for studies of magnetic materials [12]. In some cases neutron scattering with polarization analysis enables measurement of correlations of magnetism that transcend the physical granularity of the material. (In the case of

NR, the magnetism and nuclear structures are usually correlated in the direction along the film's surface normal.)

While SANS of magnetic nanoparticles, wires, *etc.* are routine, requirements for *SANS experiments of thin films* include: thin film volume larger than 0.0003 $cm^3$ or thin film mass bigger than 1 mg, and substrates with extremely smooth surfaces. The latter is required to minimize the background from the substrate so as not to swamp the SANS from the thin film. Because the neutron beam is hardly absorbed (*i.e.,* weakly scattered), multiple samples can be stacked in order to achieve the necessary volume and/or mass limits. An example of a SANS instrument is shown in Fig. 4.

Additional discussion of the application of SANS to quantitatively determine magnetic structure can be found in an article by A. Michels *et al.* elsewhere in this perspective series.

## 3. Materials phenomena

### 3.1. Emergent magnetism in transition-metal-oxide heterostructures

The possibility to break inversion ($I$), time ($T$) and gauge ($G$) symmetries at an interface combined with the ability to control chemical composition achieved with thin films, may produce novel emergent phenomena [13]. Examples of these include ferroelectricity (a consequence of $I$), magnetism (a consequence of $T$) and superconductivity (a consequence of $G$). For instance, an interface between polar and non-polar materials may result in a net interfacial charge with a concomitant electrostatic potential. This potential could be reduced by tilting structural octahedra in a perovskite which may cause canting of an antiferromagnetic structure and development of a ferromagnetic moment [1]. Octahedra can also be tilted as a consequence of stress, thus affecting spin–orbit coupling and magnetism. Alternatively, the free energy of an interface may be reduced by diffusion of cations (including magnetic ones) across the interface. In the latter scenario, ferromagnetism would be a consequence of chemical doping as opposed to being an example of an emergent phenomenon.

Notable challenges in transition-metal-oxide (TMO) research include characterization of weak interfacial magnetism distinct from its surroundings and to determine its origin. For instance, is magnetism a consequence of charge transfer across the interface (and concomitant valence change of a magnetic cation), or due to interdiffusion? Does the observation of a new interfacial phenomenon require emergence (*e.g.,* breaking of $I$, $T$ or $G$ symmetries), or might it originate from defects or interdiffusion? How can we distinguish between the competing explanations? One useful approach is to use the influence of environmental parameters, *e.g.,* temperature, magnetic field, electric field or stress, on interfacial properties with probes that are intrinsically sensitive to interface structure. In this regard, and especially for magnetic phenomena, neutron scattering is ideally suited.

Because different mechanisms may produce the same laterally averaged neutron scattering contrast across an interface, specular neutron reflectometry and SANS may be unable to identify a unique solution. For example, Fig. 5 (left) shows an example of an interface that is composed of regions that are perfectly sharp over short lateral length scales that vary randomly in height (*i.e.,* the height fluctuations are uncorrelated) over large length scales. In the middle of Fig. 5 is an example of an interface that is perfectly smooth over large lateral length scales, but is not sharp over small length scales normal to the interface plane (as in a diffuse interface). In these examples, the projections of the scattering length density along the surface normal (Fig. 5, right) is the same, yet the origin of interface magnetism could be very different, *e.g.,* emergence *vs.* diffusely located magnetic spins. Although neutron scattering provides a laterally averaged answer and therefore is unable to distinguish between these scenarios, other techniques suffer the same shortcoming perhaps at different length scales. For example, electron energy loss spectroscopy, has the same problem if the height fluctuations in Fig. 5 (left) occur over length scales comparable to the sample thickness through which the electron beam travels. Correlation of a stimulus (an environmental parameter) and response of the sample might be able to differentiate between different scenarios. Neutron

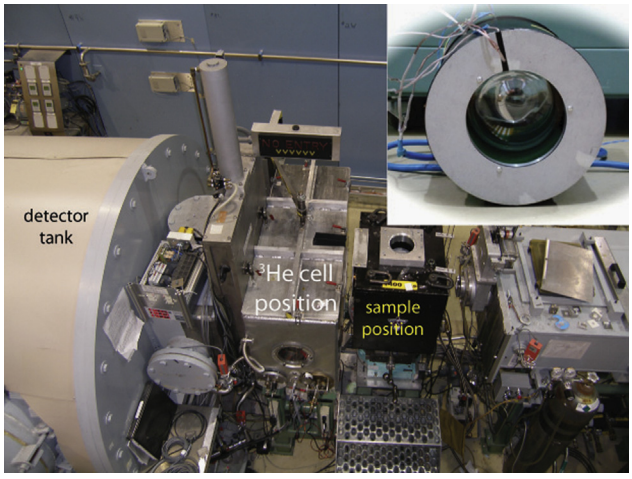


**Fig. 4.** Picture of a SANS machine in the region of the sample. The neutron beam travels from right to left. (inset) Picture of a $^3$He filter used to analyze the polarization of the scattered neutron beam.
Figure courtesy of Dr. W.C. Chen of the University of Maryland and NIST.

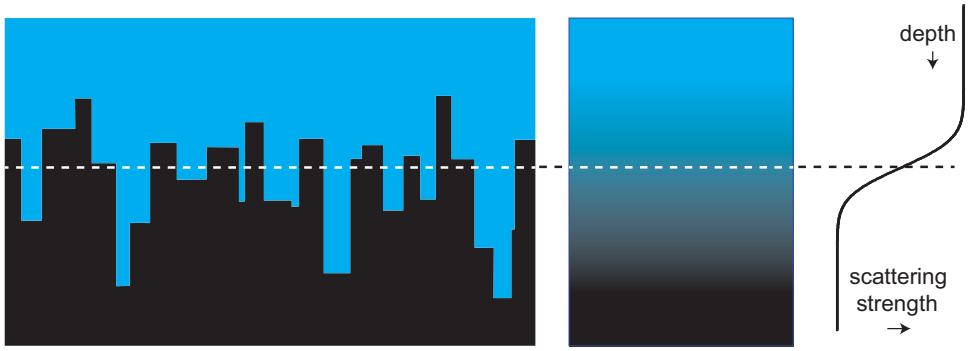


**Fig. 5.** Diagrams illustrating different scenarios of a (left) rough interface and a (middle) smooth but diffuse interface between two materials (blue and black). For the cases where the scattering power averaged over the lateral dimensions of the sample (represented by the left and middle panels) yield the same error function (right), specular neutron reflectivity cannot distinguish between either case. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

scattering lends itself naturally and has been used in measurements in which the magnetism is perturbed externally and the response is measured simultaneously thus correlating the stimulus with the response.

### 3.2. Beyond exchange bias in transition element magnets

Exchange bias, the shift of the hysteresis curve due to interfacial coupling of a ferromagnet (FM) to (typically) an antiferromagnet (AFM), has been a source of interest to condensed matter physicists and materials scientists for over five decades. It is remarkable that despite the heavy use of exchange bias in the storage and sensor industries, the fundamental mechanisms giving rise to exchange anisotropy have only recently been clarified experimentally. Yet, a quantitative predictive theory of exchange bias is still elusive—mostly because the effect is fundamentally attributable to extrinsic properties of materials, *e.g.,* to defects. The understanding so far is partial without the emergence of a unifying theory, which encompasses all different phenomena in different materials systems with predictive capabilities.

The lack of a unified understanding may be rooted in that exchange bias has continually produced unexpected experimental "surprises" which may agree with one model but not another [14,15]. For example: (i) Exchange bias may depend on the experimental method used with very different unidirectional anisotropy energies obtained from reversible compared to irreversible methods [16]. (ii) Contrary to naïve expectations the loop shift can sometimes be in the same direction as the cooling field ("positive exchange bias"). This last effect discovered in $FeF_2$–Fe bilayers using high cooling fields, and originates from antiferromagnetic coupling between the layers in contrast to the ferromagnetic coupling assumed in most theoretical models. (iii) Recent work on CoO–Py [17], NiO–Co [18] and $FeF_2$–(Co,Fe) [19,20] bilayers have claimed intriguing "memory effects" in the AFM even in the saturated state (of the FM). (iv) Ferromagnets with transition temperatures lower than that of the AFM layers remarkably exhibit exchange bias in the paramagnetic state [21,22]. (v) Synchrotron radiation studies found that in some cases although global exchange bias is absent, exchange bias appears domain by domain [23]. (vi) Recent observations of exchange bias in complex oxide heterostructures may suggest an altogether different means—one relying on charge transfer across interfaces—to achieve novel magnetic behavior [24] or even a quantum mechanism [25]. All the above mentioned puzzles imply that it is crucial to understand the details of the physical and magnetic structure. This requires combined, quantitative, local and global structural, magnetic and chemical studies such as synchrotron radiation, neutron scattering and X-ray diffraction combined with magnetization, Kerr effect and magnetotransport.

Neutron scattering may play a crucial role to unravel the key issues and the hidden secrets of exchange bias because it is a tool that can directly and quantitatively determine the physical and magnetic structure at the local scale. Important physical phenomena such as: (a) the type and location of pinned uncompensated moments in the AFM [26,27], (b) how domains are imprinted into the AFM [28,29], (c) magnetic structure of the FM during reversal [30–32], (d) effect of magnetic crystalline orientation [33], and (e) local *vs.* global exchange bias [34] and its effect on the behavior of nanostructured bilayers are just a few of the key issues. Each one of these could be the subject of an independent major research project on its own.

Many studies imply that very regular and smooth interfaces generally do not exhibit exchange bias. This is a very important observation since the tendency has been to try to enhance the exchange bias by improving interface quality. However, as discussed above, the key may be precisely in the opposite direction although the type of defects needed to increase the exchange bias is not known. There are several FM-AFM systems that use AFM's with a high Neel temperature, which, however do not exhibit exchange bias. For instance, epitaxial $Fe_xO_y$ on MgO show narrow rocking curves and small ($<5$–$7$ Å) interfacial roughness. However the exchange bias with these AFM's is very small, perhaps nonexistent. This implies that either: (a) defects play an important role, (b) the exact interfacial interaction (*i.e.* the particular AFM-FM combination) is crucial or (c) the magnetic structure close to the interface is substantially modified. This further implies that studies in which the interfacial structure is modified and localized spatially in a well-defined manner may crucial. Neutron scattering studies of radiation damaged samples to localize defects at well-defined locations may produce the desired understanding.

An alternative way to change exchange bias is to use light or time dependent magnetic fields to affect the interfacial coupling. Very surprising and interesting effects have been found in time dependent pump–probe experiments of exchange bias systems. The feasibility of such "pump–probe" experiments has recently been demonstrated by Zhernenkov *et al.* in a neutron reflectometry study of the response of Fe magnetization to microsecond-time-varying AC magnetic fields [35]. Application of time-dependent fields and neutron scattering, for example to determine whether the pinned uncompensated magnetization remains pinned in the presence of time dependent fields, may aid our understanding of exchange bias as it is applied in real world materials (*e.g.,* the stray field sensed by a recording head is not static). Furthermore, investigations of exchange bias in complex oxide systems should also prove interesting, because these materials tend to exhibit photo-induced-conductivity and/or photo-induced-magnetization, and thus are susceptible to light irradiation. Furthermore, tailoring orbital magnetization of a complex oxide interface may provide intriguing opportunities to affect exchange coupling across complex-oxide-interfaces and thus, exchange bias.

### 3.3. Influence of electric field on magnetism

In 2000, Ohno et al. [36]. measured the sheet Hall resistance of a field effect transistor that contained a Mn-doped InAs layer. From these measurements they concluded that application of a negatively biased potential increased the hole concentration of the (In,Mn)As layer, thus, promoting ferromagnetism. In the Ohno experiment, a 125 V potential was applied across a 0.8 μm thick insulator producing an electric field of order $10^8$ V/m. The lateral dimension of the device was of order tens of μm. An entirely different means of using electric field to modify magnetism involves passing an electric current through a laterally nanometer-sized pillar of conducting and magnetic material. The current induces a (spin-)torque on the magnetic spins, causing them to change direction [37,38]. Looking into future applications involving complex oxides, one could imagine combining control of conductivity with electric fields and magnetic layers to develop new spintronic technologies such as suggested by Chakhalian et al. [39], or to couple electric field, strain (discussed later) and magnetism in multiferroic materials or composites [40].

Such studies would clearly benefit from the direct observation of interfacial magnetism. However, a significant challenge is to apply a large electric field ($\sim 10^8$ V/m) across the thin dimension of a film having relatively large surface area ($\sim 1$ cm$^2$ samples are needed for neutron reflectometry). An electrical short across one portion of the sample may be sufficient to negate the electric field over the rest, thus, compromising the measurement. The risk of failure increases dramatically with the area of the sample. In the electronic materials community, this risk is mitigated by patterning an electrode with small lateral dimensions [41]. Provided the smoothness and uniformity of the film is preserved by the patterning

process, the influence of electric field on a magnetic film [42] may be feasible to study with neutron scattering [43]. Alternatively, self-assembly of magnetic dots is another practical approach to achieve samples suitable neutron scattering [44].

Another approach to apply an electric field to a large area sample was pioneered by Weisheit *et al.* [45]. for magneto-optic-Kerr-effect studies, and adapted by Zhernenkov *et al.* [46]. for neutron scattering. As shown in Fig. 6, the magnetic (CoPd) film was deposited on a Pt-coated substrate and immersed in an electrolyte solution. Once a potential is applied, an insulating ionic layer grows on the film's surface across which a large electric field can be sustained. Zhernekov *et al.* observed an increase of the saturation magnetization of a CoPd film by increasing the applied electric field to $10^8$ V/m (Fig. 7). They attributed the influence of the electric field on the saturation magnetization to a modification of the Fermi level by the electric field. Such an experiment may have even more dramatic results in complex oxides. Since many oxides are nominally insulating, the electric field can penetrate further into the sample. The electric field may affect the hole carrier concentration or charge transfer, thus, directly affecting superexchange and double-exchange mechanisms that mediate magnetism. Alternatively, an electric field may affect the configuration of orbitals at the interface through a piezoelectric (strain) mechanism. For applications in studies of complex oxide films and interface, one would require either a conducting substrate, *e.g.,* Nb-doped $SrTiO_3$, or a conducting buffer layer suitable for epitaxial growth. Furthermore, the studies would be limited to temperatures above the freezing point of the electrolyte.

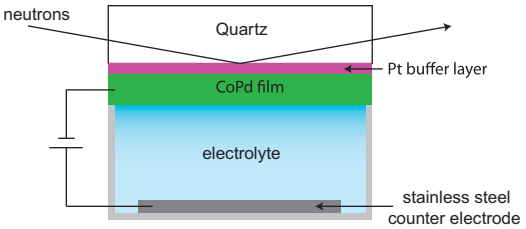


**Fig. 6.** Schematic diagram of an electrolyte cell used to apply an electric field across the surface of a magnetic film. The thicknesses of the Pt and CoPd layers are greatly exaggerated.

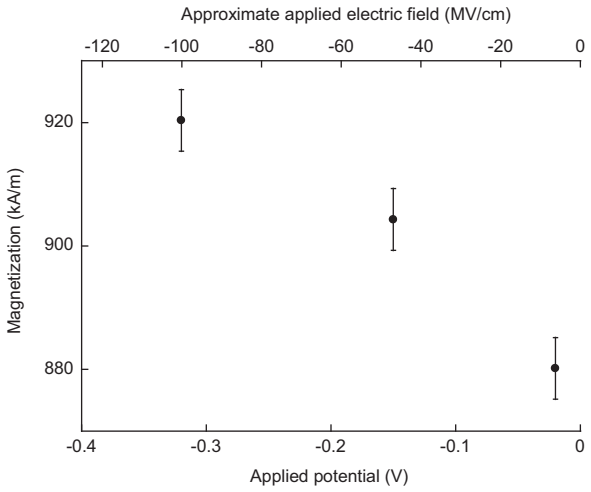


**Fig. 7.** Magnetization of the 7.2 nm portion of the CoPd layer in contact with the electrolyte solution as a function of applied potential.

### 3.4. Influence of strain on magnetism

In Hwang *et al.* [13] review of emergent phenomena at oxide interfaces the authors argue that due to the strong correlation between charge, spin and orbital degrees of freedom, strain can profoundly affect magnetism. For example, strain may cause metal-oxide octahedra to tilt, thus influencing occupation of orbitals and spin–orbit coupling. Such effects can be manifested in the anisotropy, ordering temperature or saturation magnetization of the material. Indeed epitaxial strain is often thought to be the origin of uniaxial anisotropy in some oxides [47].

However, the role of strain and magnetism in complex oxide films is far from understood. For example, unlike bulk manganites, in which compressive stress can favor the ferromagnetic phase, theoretical and experimental studies of manganite films paint a decidedly mixed picture. In particular, experimental studies have explored the influence of strain on magnetism by comparing films grown under different conditions, on different substrates or with different chemical compositions. However, none of the experiments have examined the exclusive role of stress on magnetic properties. Namely, more than the “stress knob” was adjusted. Thus, it is not surprising that no consensus has emerged on the influence of strain on magnetism in manganite films.

Because of the aforementioned shortcomings, three new experimental approaches have been developed to explore the influence of stress on magnetism. The first involves epitaxial growth of a suitable film on $BaTiO_3$ [48]. The magnetization of the film is then measured as the sample is cooled through successive phase transformations of the $BaTiO_3$ substrate. Provided the film remains clamped to the substrate, after each phase transformation, the film's lattice expands in directions parallel to the film's surface. The second approach involves epitaxial growth of a film on a lead zirconate titanate (PZT) substrate [49]. By applying an electric field to the PZT substrate, and provided the film remains clamped to the substrate, compressive stress can be applied to the film. More generally, clamping an epitaxial film to the substrate strains it far beyond what is possible with bulk materials. Thus, a larger range of functional behavior is perhaps possible with thin films than in bulk materials [1].

The strain in the film resulting from either of the aforementioned techniques decays as a function of distance from the film/substrate interface. Thus, the strain is non-uniform and measurement of bulk magnetization, *e.g.,* magnetometry, will provide a strain-averaged result. However, the strain non-uniformity and the possible concomitant non-uniformity of magnetization *vs.* depth can be quantitatively measured with PNR. Thus, the combination of either stress-application-technique with PNR provides the means to investigate flexomagnetism [50], *i.e.* the influence of the strain gradient on magnetization. Owing to the large surface area to volume ratio of nanoscale structures, flexomagnetism can significantly affect magnetic properties of these materials.

The third approach involves using a mechanical jig to apply pure bending stress to a film [51]. Unlike in the first two approaches, which require epitaxy between the film and either $BaTiO_3$ or PZT, a mechanical jig can apply stress to any film/substrate combination—provided, the substrate is reasonably thin ($<250$ μm thick). Fig. 8 shows a four point bending jig that can apply uniform bending stress across large area sample (*e.g.,* 1 $cm^2$ samples) suitable for neutron scattering [52]. Even though the substrate is “thin”, its thickness is still orders of magnitude greater than that of the film. Therefore, the resulting strain in the film is uniform as a function of depth (thus, flexomagnetism cannot be investigated using a mechanical jig). To date the maximum strain that can be applied to a sample consisting of a ceramic substrate for neutron scattering studies is of order $\pm 0.01\%$. Even though the maximum strain is small, it can still significantly affect the

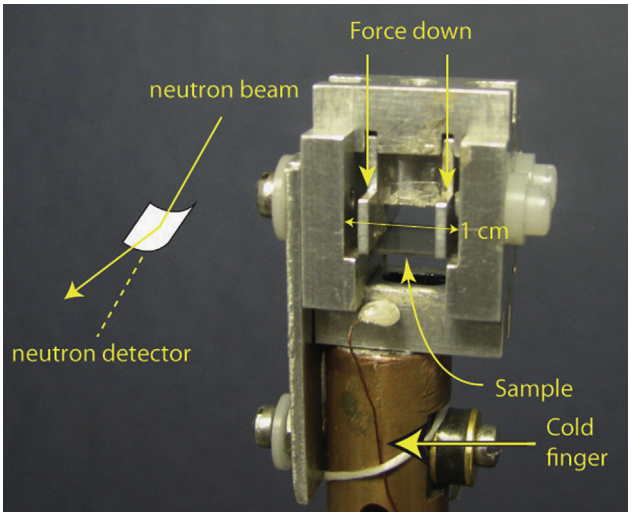


**Fig. 8.** Picture of a four point bending jig used in neutron reflectometry experiments. A screw (not shown) adjusts the force applied to a sample causing the sample to bend in the shape of a cylinder (left).
Figure courtesy of Dr. S. Singh of Bhabha Atomic Research Center, India.

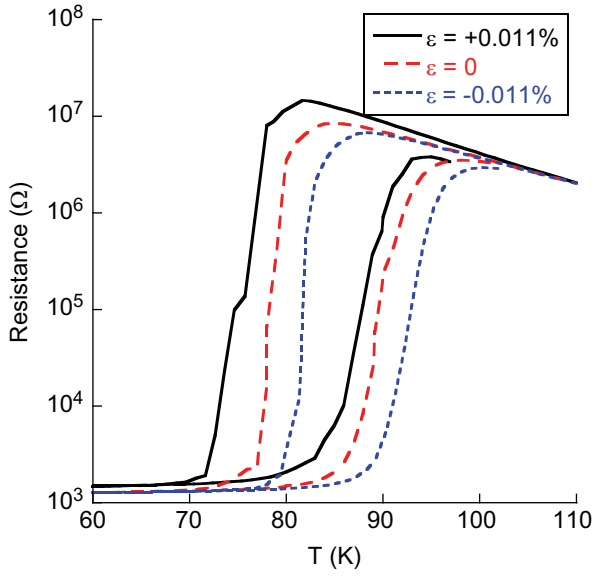


**Fig. 9.** Shift of the hysteresis in the conductivity of a $(La_{0.4}Pr_{0.6})_{0.8}Ca_{0.2}MnO_3$ thin film as a function of strain.
Figure courtesy of Dr. S. Singh of Bhabha Atomic Research Center, India.

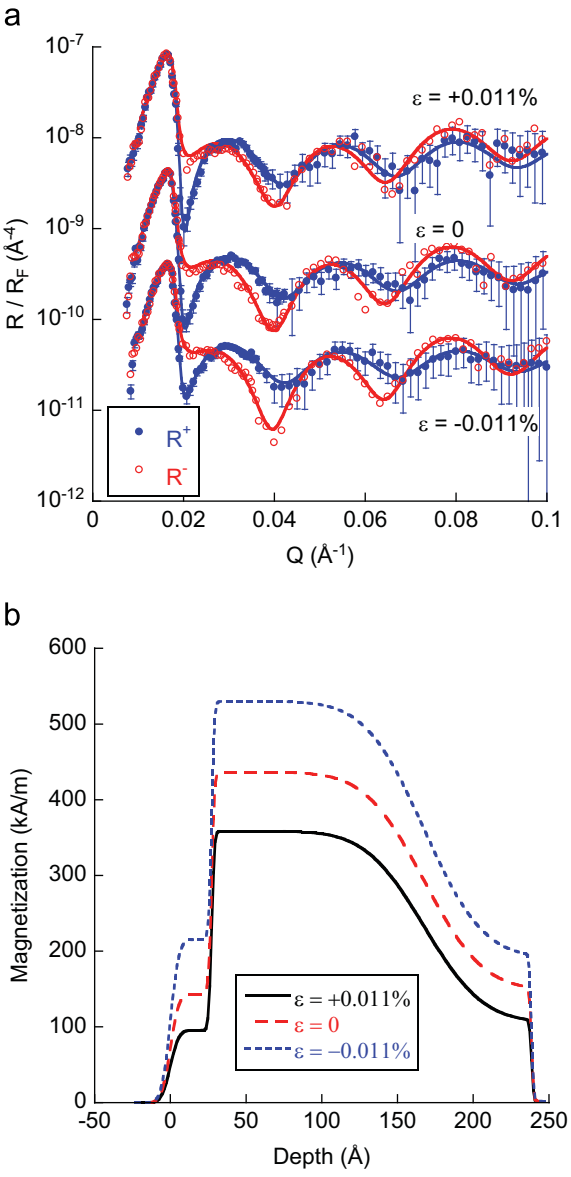


**Fig. 10.** (a) PNR data of a $(La_{0.4}Pr_{0.6})_{0.8}Ca_{0.2}MnO_3$ thin film taken as a function of strain at 78 K. (b) The strain dependence of the saturation magnetization depth profile obtained from the neutron data. [The variation of the magnetization with depth is due to the variation of chemical composition with depth and not strain].
Figure courtesy of Dr. S. Singh of Bhabha Atomic Research Center, India.

metal-insulator transitions (Fig. 9) and saturation magnetization (Fig. 10) for a particular manganite $(La_{0.4}Pr_{0.6})_{0.8}Ca_{0.2}MnO_3$ film [52].

Pressure cells provide a method to apply uniform stress to magnetic films. The cells, however, are restricted to temperatures above the freezing point of the hydrostatic medium although helium gas can be used to avoid the freezing problem. A picture of a pressure cell used for neutron reflectometry is shown in Fig. 11. This cell operates at temperatures between 300 and 573 K and provides pressures up to 200 MPa (helium gas pressure cells can provide in excess of 800 MPa). Mechanical jigs, pressure cells and neutron scattering are useful to investigate the influence of elastic bending stress (compressive or tensile) on a wide variety of strain-sensitive systems, including interfacial magnetism of complex oxide heterostructures, superconductors, piezomagnetic and multiferroic materials.

### 3.5. *Laterally non-uniform films—magnetic phase separation*

The previous discussion has focused primarily on magnetism that changes as a function of depth in a film or interface. The collective interactions between charge, spin, orbital and lattice order that can cause the magnetism to vary in the direction normal to the film plane can also lead to phase separation or phase coexistence of magnetic and non-magnetic phases throughout bulk materials. Dark field electron and Lorentz microscopy measurements of the same bulk manganite sample provide compelling evidence for the existence of magnetic phase separation (in bulk material) [53]. Indeed SANS has contributed to our understanding of coexistence of ferromagnetic and non-ferromagnetic regions in bulk materials believed to be chemically uniform [54]. It is often expected that under the proper magnetic field and temperature conditions, cubic ferromagnetic metallic (FMM), orthorhombic antiferromagnetic charge-ordered insulating (AFM-COI), and pseudo-cubic paramagnetic insulating (PMI) phases coexist in some complex oxides.

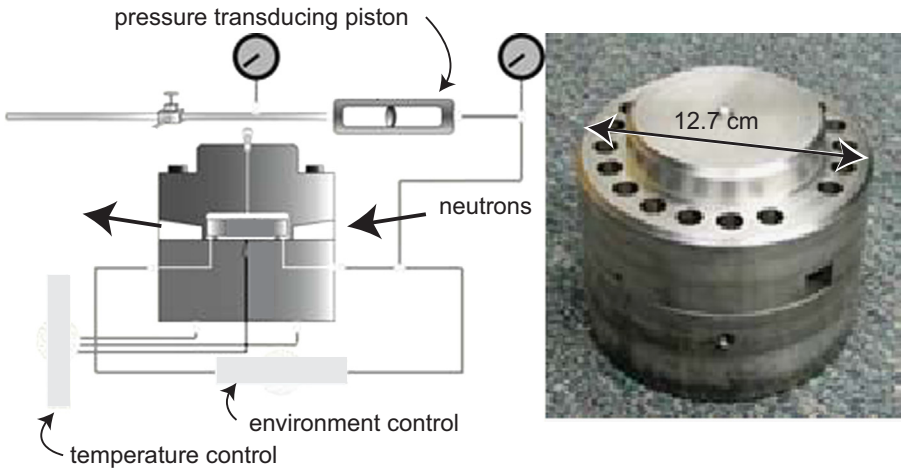


**Fig. 11.** (left) Schematic diagram of a pressure cell used for neutron reflectometry. (right) Picture of the cell: maximum pressure and temperature are 200 MPa and 573 K. Figure courtesy of Dr. J. Majewski of the Los Alamos National Laboratory.

Since phase separation is observed in the bulk, similar behavior should be present in thin films. The coexistence of metallic and insulating domains in thin films, primarily La–Ca–Mn–O (LCMO) films, has been observed using scanning tunneling (STM) and conductive tip atomic force (cAFM) microscopies. However, these probes cannot determine whether a metallic domain is also ferromagnetic. Instead, we presently rely on circumstantial evidence using magnetic force microscopy (MFM) that measures the second derivative of the stray field normal to the film surface, and its relationship with topography and conductivity. MFM images may not accurately represent the in-plane variation of the film magnetization, *e.g.,* the variation of the normal component of the stray field may be a consequence of growth on a vicinal (stepped) surface. Might defects, such as surface steps serve as nucleation sites for metallic domains [55] in a magnetically uniform film (in the plane of the film)?

Neutron scattering can help clarify the picture. Specifically SANS studies of thin films, *e.g.,* as in Laves phase materials (Fig. 12) [31] or phase separation in the cobaltites [56], have successfully discriminated between structural and magnetic disorder that occur with length scales of order 100 nm to microns. Changing magnetic properties using either field or temperature without affecting structural disorder provides the means to determine whether the magnetic length scales are consistent with metallic domains observed with scanning probes. In fact, polarized neutron beams and SANS offer a rigorous determination of the magnetization fraction which is correlated with structural, *i.e.* quenched, disorder in the material. Quenched disorder [57] and strain [58] have been proposed as means to promote phase separation which could be monitored by sophisticated SANS experiments involving pressure cells.

### 3.6. *Proximity effects and laterally patterned films*

The proximity between a FM and a dissimilar material (normal (N), superconducting (S), ferroelectric (FE), AFM, other FM) has produced much new physics and is the basis for a number of applications. These include: the discovery of giant magnetoresistance (GMR) and coupling in FM/N, collective pinning in FM/S, multiferroicity in FM/FE, exchange bias in FM/AFM, exchange spring in soft FM/hard FM systems, *etc.* In many of these cases, neutron scattering played a major role in uncovering and clarifying fundamental physics issues.

The coupling in (FM/N) superlattices was found to be very delicately dependent on the exact spacing between and roughness at the N layers. This allowed engineering superlattices with multistate configurations (*i.e.,* different magnetic configurations). As a consequence the magnetization of the material exhibits well defined discrete moments which simply depend on the number of layers rotated from the field direction. In this case, the number of states is determined by the number of layers that are ordered in a specific direction and can be controlled by the previous magnetic history. This provides the means for engineering multistate devices by simply increasing the number of layers in the stack [59].

The fabrication of nanomagnetic arrays in proximity with superconducting films has opened up a whole new perspective in the field of superconducting vortex pinning. Ordinarily, enhancement of pinning in superconductors is obtained by the incorporation of randomly distributed defects into the superconductors. The development of nanofabrication techniques allows the investigation of the interaction (or commensuration) of two periodic systems: the vortex lattice and the array of pinning sites. Moreover, vortex pinning by arrays of regular dots [60] has relevance and may serve as ideal model systems to other fields of physics including; vortex arrays in confined charged plasmas, epitaxial growth of elastically soft materials deposited on top of a rigid lattice, and a variety of hybrid systems in which the proximity effect plays an important role.

The relevant physics length scales are close to what can be artificially produced in the laboratory enabling studies in which matching effects between structural and physics length scales occur. Since the array geometries can be manipulated at will and with the wealth of existing superconductors, a very rich range of systems in which many of the relevant parameters can be varied allows studies of the effect of thermal fluctuations, commensuration, and interactions between different types of vortices (for instance magnetic and superconducting). In addition, these systems provide the means for the development of novel devices such as tunable “Josephson like” systems to modify the field-dependent critical current of Josephson junctions, to improve signal-to-noise of SQUID-based devices and microstrip band-pass filters, and to open up an avenue for the enhancement of critical currents in superconducting wires.

Unfortunately it is very hard to determine the location of the superconducting vortices, which play a crucial role in the understanding of the physics of these systems. Here is where neutron scattering may play a crucial role. For example, rich phase diagrams of vortex matter in bulk superconductors have been discovered using SANS [61]. In this case the expulsion of the magnetic induction from vortex cores provides the neutron scattering contrast that produces SANS. Such measurements are

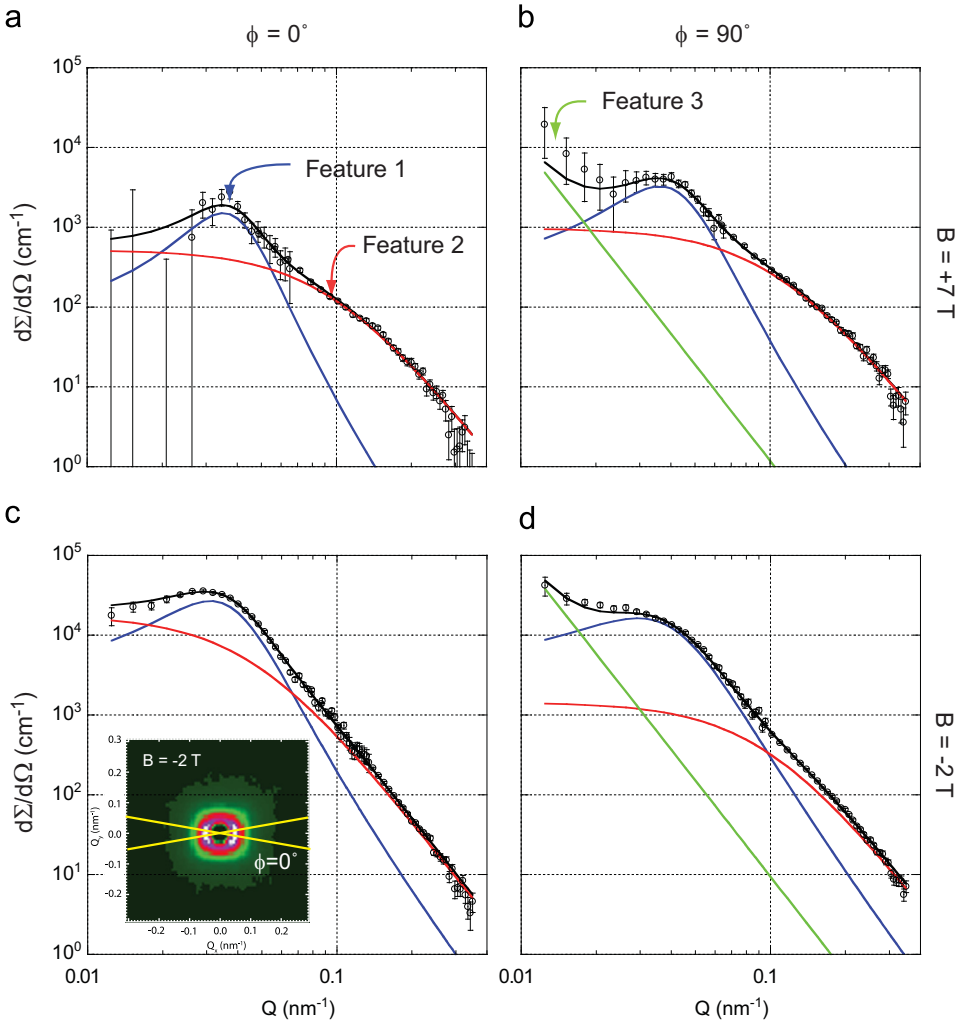


**Fig. 12.** Log–log plots of the absolute cross-section d/d obtained from SANS data averaged over sectors within 10° of $\phi=0$ and 90° as functions of applied field. (inset) One SANS data set showing a $\phi=0°$ sector. The data were collected from a stack of eight 1 $cm^2$ $DyFe_2/YFe_2$ superlattice samples. The superlattice films were 330-nm thick. Curves inferred from the data refer to the SANS produced by three scattering features: (1) small domains with magnetization correlated over distances corresponding to $190 \pm 44$ nm (2) randomly arranged domain walls with widths corresponding to ~20 nm and (3) micron or larger magnetic domains.

not necessarily restricted to bulk samples, as demonstrated by W.A. Hamilton *et al.* [62]. In their study, applied shear stress to an ionic viscoelastic fluid induced hexagonal ordering in ~0.5 μm thick layer near a surface. The ordering was detected using SANS in grazing incidence (reflection) geometry. One could imagine using such a technique first pioneered in the soft matter community to study of vortex matter in confined (*e.g.,* thin film) geometries.

## 4. Concluding remarks

This perspective article shows how neutron scattering can be used to clarify and possibly resolve outstanding issues in nanostructured magnetic materials. We are fortunate to have access to powerful neutron sources, to synthesis tools which allow engineering specific interfaces and to a variety of mutually compatible sample environment options. A continuing challenge is to catalyze coherent and synergistic activities of sample growers, those who characterize structure and properties (*e.g.,* neutron scatterers) and theoreticians/modelers to tackle outstanding problems in magnetic materials research that has and will continue to be important to society.

The physical properties of modern functional materials, especially magnetic ones, depend on the structural/chemical properties which need detailed attention from materials scientists/physicists dedicated to synthesis. In addition, the magnetic properties of these materials crucially depend on externally applied environments such as: temperature, magnetic field magnitude and orientation, magnetic history, electrical current, light *etc.* Moreover, the investigation of some of the basic issues which arise, invariably require application of controlled and perhaps time dependent environments. This highlights the important role sample environment plays in these studies. It is commonly accepted that is important to address the need for intense neutron beams, broad range of wavevector transfer, *etc.* by properly designing spectrometers and neutron sources. In addition, it is important to stress that sample environment plays a crucial a role, since many experiments are either impossible or rudimentary if the sample environment is not properly designed. In many cases, the availability of properly designed sample environment opens up a plethora of new research areas including for instance; (a) pump-probe experiments using electric currents and light, (b) exchange bias under different field preparation and conditions, and (c) magnetism of unusual materials while subject to time dependent fields.

In the interest of brevity, we have primarily focused on complex oxide films. However, the techniques described in this perspective are applicable to a variety of systems, *e.g.,* Fe-pnictide superconductors, vortex matter, topological materials in contact with magnetic films, patterned magnetically frustrated entities

(spin ice), and so on. The common feature shared by these systems is that the magnetic induction is not uniform over length scales that are conveniently probed by neutron scattering. While there is some overlap between neutron scattering and resonant x-ray scattering [63], the two techniques are mostly complementary.

This perspective article has focused on elastic neutron scattering, in which the energy of the neutron is the same before and after scattering. Inelastic neutron scattering is well established as the preferred method to provide the exchange coupling within the unit cell (or exchange stiffness) in bulk materials. For novel materials, the value of the exchange coupling across an interface, which can be obtained from the interfacial spin wave dispersion, is an exceedingly interesting quantity. Unfortunately, the lack of large enough volume of "interfacial materials" and the weak neutron scattering interaction have proven to be a significant impediment. However, the development of new neutron scattering techniques may show some promise [64].

Neutron scattering still enjoys and for the foreseeable future will enjoy advantages of: being mostly free of instrumental challenges (*e.g.,* since the neutron has spin, the polarization of the neutron beam is trivially changed with 99.9+% accuracy and precision), and neutron scattering data are trivially interpreted yielding quantifiable measurements in absolute units. Perhaps the key advantage (and limitation) of neutron scattering is its weak interaction with matter and therefore making neutron scattering the key technique for probing beneath the surface. The bottom line is: "Go forth and scatter!"

## Acknowledgments

Valuable discussions with Dr. S. Bader (ANL), Dr. J.A. Borchers (NIST), E.D. Dahlberg (UMN), Dr. J. Freeland (ANL) Dr. S.G.E. teVelthuis (ANL), and Q. Wang (LANL) are gratefully acknowledged. This work was supported by the Office of Basic Energy Science—Materials Sciences and Engineering, U.S. Department of Energy and Office of Basic Energy Science, DMR under Grant DE FG03-87ER-45332. Los Alamos National Laboratory is operated by Los Alamos National Security LLC under DOE contract DE-AC52-06NA25396.

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