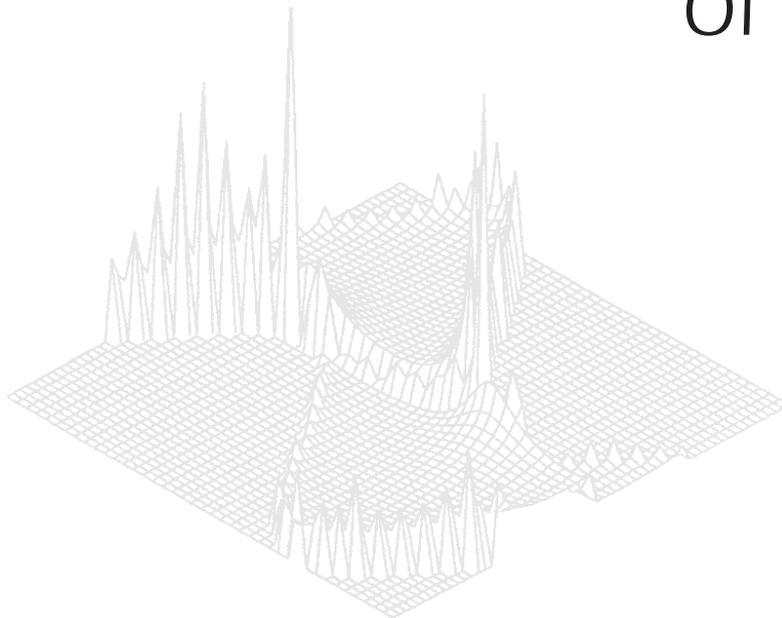

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Nuclear Orientation as a Tool for Investigation of Magnetic Multilayers with Rare Earths*

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Abstract

Low-temperature nuclear orientation (NO) is presented as a useful tool to study the behaviour of rare earth (RE) ionic magnetic moments in magnetic multilayers. NO is shown to give rather direct information about the direction of RE ionic moments in such low-dimensional systems. In particular, the perpendicular magnetic anisotropy (PMA) can be directly monitored using RE atoms as probes. The potential of NO is demonstrated by our recent results, which concern the Fe/Tb multilayers. We have studied NO of ¹⁶⁰Tb in Fe(40 Å)/Tb(*x* Å) (*x* = 5–30) and found that the Tb magnetic moments show PMA at low external magnetic fields (B_{ext}). PMA of the Tb spins is found to be more pronounced when the Tb layer is thinner. It was found that B_{ext} has a complicated influence on the Tb magnetic moment misalignment, which is connected with an interplay between PMA, the exchange interactions and the shape and magnetic crystalline anisotropy.

1. Introduction

In the last decade, together with the progress of new ‘clean’ sample preparation techniques, much attention has been devoted to the investigation of magnetic metallic systems which have at least one spatial dimension of homogenous phase reduced to a level of several units or tens of Angstroms. Such systems (clusters, dots, monolayers, multilayers etc.) may show magnetic properties different from those of the bulk state, which on one hand attracts physicists because of fundamental problems of magnetism and which on the other hand may be important for new industrial technologies. For understanding microscopic properties of such magnetic structures, especially of those consisting of several elements, a detailed knowledge of individual magnetic moment behaviour is desired.

There are only few methods which are ‘element’ selective and which are in addition sensitive enough to study samples with rather small total amount of atoms, which is naturally characteristic for low-dimensional (LD) systems. Among them, magnetic x-ray dichroism, neutron diffraction and some hyperfine interaction techniques (i.e. NMR, Mössbauer spectroscopy, perturbed angular correlations, low-temperature nuclear orientation etc.) can bring rather direct

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information about the size of a magnetic moment localised on a given atom and sometimes even about its magnetic moment direction.

In this contribution we shall concentrate on the scope of the low-temperature nuclear orientation technique (NO) in a study of LD systems. Simultaneously, as a demonstration of this technique, we shall give a summary of our recent NO results of the magnetic Fe/Tb multilayers, which gave for the first time direct evidence of perpendicular magnetic anisotropy of rare earth (RE) atoms in such materials. The paper is organised as follows: In Section 2 the main features of NO, which are relevant for a study of LD systems (especially of those containing rare earth elements), are described. Simultaneously, some experimental aspects are treated, especially the sample requirements for an application of the NO method. Section 3 contains a short review of recent results and open problems in the investigation of the Fe/RE multilayers. Finally, in Section 4 our NO results with the Fe/Tb multilayers (partly published as Trhlík *et al.* 1996*a*, 1997) are briefly summarised and discussed.

2. Low-temperature Nuclear Orientation

(2a) General Description

NO is based on the fact that if radioactive nuclei with nonzero spin are at least partly oriented in space (which can be caused by action of e.g. a hyperfine field at temperatures low enough), the radioactive radiation emitted by them is anisotropic. In NO experiments the emission rate is detected in some direction and normalised by the rate from an isotropic nuclear ensemble (temperature $T \rightarrow \infty$) to obtain the anisotropy W . The general expression for W and its derivation is somewhat complicated (see Krane 1986; Brewer 1990), but it can be written for our case (γ -rays, radioactive nuclei in thermal equilibrium with lattice, hyperfine interactions with a symmetry high enough etc.) as

$$W = 1 + \sum_{k=2,4} Q_k U_k A_k B_k (|\mathbf{B}_{\text{hf}} + \mathbf{B}_{\text{ext}}|, V_{zz}, T) \langle P_k(\cos\phi) \rangle, \quad (1)$$

where Q_k are the constants given by detection geometry (≈ 1 in most cases), $U_k A_k$ are the constants given by nuclear decay (known from other experiments for the nuclei used in solid state applications) and B_k are the orientation functions given by the nuclear moments and the spin ($B_k \rightarrow 0$ for $T \rightarrow \infty$, and $B_k = \text{constant}$ for $T \rightarrow 0$). Here \mathbf{B}_{ext} , \mathbf{B}_{hf} and V_{zz} are the external magnetic field, the hyperfine field and the electric field gradient, respectively, which act on the nucleus. Here we suppose that the electric field gradient has cylindrical symmetry, the axis of which coincides with the $\mathbf{B}_{\text{hf}} + \mathbf{B}_{\text{ext}}$ direction. Further, P_k are the Legendre polynomials [i.e. $P_2 = (3\cos^2\phi - 1)/2$ and $P_4 = (35\cos^4\phi - 30\cos^2\phi + 3)/8$] and ϕ is the angle between the axis of symmetry mentioned above and the detection direction.

Because nuclear magnetic moments are rather small ($\sim 10^{-26}$ JT⁻¹) and B_{hf} have also limited values ($B_{\text{hf}} \sim 10\text{--}100$ T), one can have a measurable effect of W at temperatures below ~ 100 mK. Nevertheless, for most applications temperatures down to 10 mK are necessary.

As one can see from (1), NO can give information on the values of hyperfine interaction parameters, having some relation to the value of the *ionic* magnetic

moment localised around a nucleus, as well as on their directions, connected with the ionic magnetic moment direction. Because of the high sensitivity of NO and its total element selectivity, the magnetic ground state of extremely diluted systems and/or systems consisting of several kinds of magnetically active elements can be studied with advantage. Reviews of such NO studies of bulk magnetic solids were given by Brewer (1986, 1990) and Turrell (1986).

An extension of NO applications to LD systems seems to be a further natural step. Surprisingly, except for the research summarised here (i.e. ^{160}Tb : Fe/Tb multilayers), there are only a few other reports or attempts in this field as yet ($^{110\text{m}}\text{Ag}$: Fe/Ag multilayers—Vanneste *et al.* 1996, Phalet *et al.* 1997; ^{60}Co : Co/Pt multilayers—Hagn 1995; ^{60}Co : Fe/Co multilayers—Trhлік *et al.* 1996b).

(2b) *Example: NO of ^{160}Tb*

Let us demonstrate the potential of some NO applications in LD systems considering the NO of ^{160}Tb in detail:

Hyperfine interactions. As is usual for most RE elements, both hyperfine interactions (i.e. the magnetic dipole B_{hf} and electric quadrupole V_{zz} one) are strong and are close to those of the free ion state even in a crystal lattice. Simultaneously, the symmetry axis of the total hyperfine interactions exactly coincides with the ionic magnetic moment direction for the free Tb ion and is only slightly disturbed by putting a Tb ion into a solid (see e.g. McCausland and Mackenzie 1979). The relation (1) is therefore a good approximation for NO experiments with Tb.

The values of the hyperfine interaction and nuclear parameters of ^{160}Tb in bulk Tb (Brewer *et al.* 1988) lead to the full saturation of B_k ($B_2 = 1.443$) (and therefore of W) for temperatures below 10 mK. This is also demonstrated by our results of ^{160}Tb : Fe/Tb multilayers (see Fig. 1), where the hyperfine interaction should be similar as in bulk Tb. We can conclude that when the temperature is low enough and the hyperfine interactions are of favourable intensity, an exact knowledge of the latter is not necessary and W is determined only by the misalignment of ionic Tb magnetic moments (i.e. $\langle P_k \rangle$).

Nuclear orientation constants. The spectrum of the γ -ray irradiation connected with the decay of ^{160}Tb is rather complicated. The main γ -line with energy of 299 keV corresponds to the nuclear transition having $U_2A_2 = -0.365$ and $U_4A_4 = 0.000$ (Marshak *et al.* 1989). The relation (1) becomes then very simple and allows direct extraction of $\langle P_2 \rangle$ from W .

Direction of ionic magnetic moments. The next step, i.e. evaluation of the Tb ionic magnetic moment misalignment angle ϕ from $\langle P_2 \rangle$, is complicated by the fact that NO with γ -rays cannot distinguish between up and down magnetic moment directions because of forward-backward symmetry. Moreover, in case the misalignment angle is not unique, some model of its distribution must be taken into account and only parameters of this model can be obtained. Nevertheless, some extreme cases of a magnetic moment direction (or at least a tendency to them) can be clearly seen (see e.g. the NO study of ^{160}Tb in bulk Tb—Grimm *et al.* 1983). In the case of planar samples one can unambiguously conclude whether the magnetic moments lie in the sample plane or are turned out of it. Some special positions are illustrated in Fig. 2.

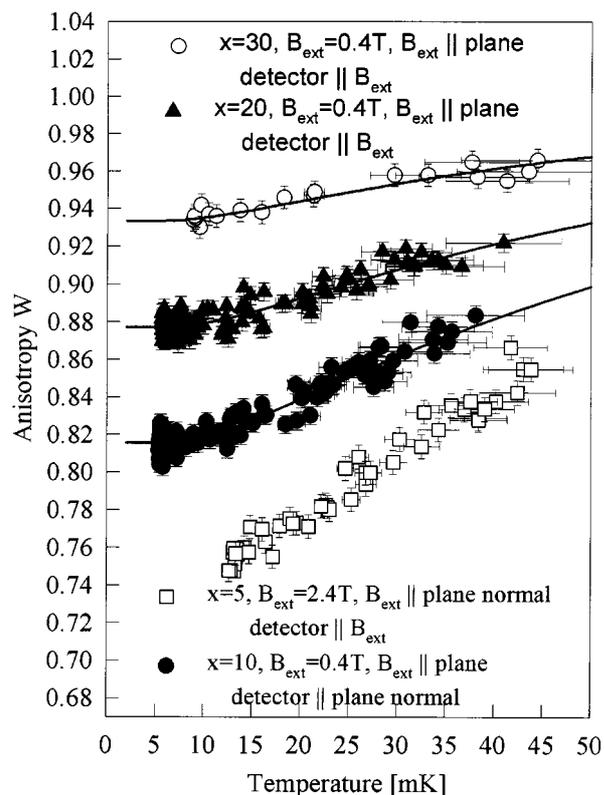


Fig. 1. Temperature dependence of W for $\text{Fe}(40 \text{ \AA})/\text{Tb}(x \text{ \AA})$ for various values of B_{ext} and for various positions of the sample, B_{ext} and the detector.

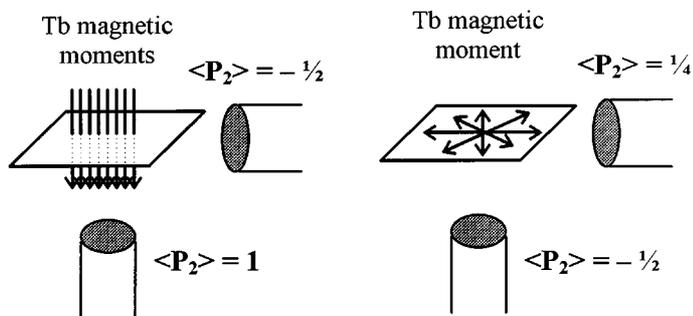


Fig. 2. Extreme cases of the Tb magnetic moment orientation and corresponding values of $\langle P_2 \rangle$.

(2c) Some Experimental Aspects

Both the preparation of samples of LD systems and the NO experiment are very sophisticated fields and we do not intend to discuss these subjects here thoroughly. Concerning the former, a lot of modern methods are now available

and its choice depends on a specific LD system to be studied. The 'classical' NO experiment is well described by Brewer (1990) or Brewer and Chaplin (1986). Here we constrain ourselves only to few remarks which mainly refer to requirements concerning samples, making NO experiments with them possible and useful.

Because NO needs radioactive isotopes as probes in proper sites in the host material, the first requirement is to assure that this is indeed the case. The best variant would be to put them there '*in situ*' during building a sample, which is unfortunately not so easy in case of LD systems, prepared mostly by MBE, evaporation or sputtering. To our knowledge there are only a few laboratories under construction, where this approach will be (at least partly) possible (e.g. here at the I. K. S., K. U. Leuven). High energy implantation of radioactive isotopes into samples prepared previously by usual techniques is not too promising, as one needs the probes to be at known positions, which cannot be assured for LD systems by implantation.

Up to now the only possibility to put radioactive probes into a system seems to be irradiation of prepared samples in advance by thermal neutrons in a reactor. In fact, all NO experiments mentioned above were done with such samples. Unfortunately, the fact that one is limited only to this type of activation narrows somewhat the number of elements which can be studied by the NO method, generally having a suitable isotope for almost every element. A combination of the following demands must occur: (i) reasonably high thermal neutron cross section of the original stable isotope in order to be able to activate LD systems containing small amount of atoms, (ii) suitable half-life of a created radioactive isotope to have time to carry out a NO experiment and (iii) radioactive isotope parameters suitable for NO. In the case of RE this condition limits us to Tb (i.e. ^{160}Tb —half-life $T_{1/2} = 72.3$ days), Nd (i.e. ^{147}Nd — $T_{1/2} = 11$ days) and Ce (i.e. ^{141}Ce — $T_{1/2} = 32.5$ days).

3. Fe/RE Multilayers

Fe/RE multilayers show some unusual magnetic properties and are attractive also as potential candidates for magneto-optical recording media (see the recent review by Shan and Sellmyer 1996). Their most interesting feature seems to be the perpendicular magnetic anisotropy (PMA) which is shown by Fe/RE with RE = Pr, Nd, Tb, Dy and Eu in some temperature ranges and Fe and RE thicknesses, as found by magnetisation measurements (e.g. Shan and Sellmyer 1990; Shan *et al.* 1990) and by ^{57}Fe Mössbauer spectroscopy (e.g. Scholz *et al.* 1991; Mibu *et al.* 1993). PMA is mostly accepted to be forced by the single-ion anisotropy of rare-earth atoms near the Fe/RE interface, which is caused by interaction of unquenched orbital angular momentum with a crystal lattice having a broken symmetry around this interface (see e.g. Shan and Sellmyer 1996). An interplay of this mechanism, shape anisotropy, crystal anisotropy within RE layer and exchange interactions within and between individual layers seems to create complicated magnetic moment structures not yet fully understood.

Much attention has been paid especially to the Fe/Tb multilayers with layer thicknesses of several units or tens of Å. Their structural studies (Dufour *et al.* 1991; Fujiwara *et al.* 1996; Richomme *et al.* 1996; Scholz *et al.* 1994) have shown that the Fe layers are polycrystalline when they are thicker than about 20 Å, and amorphous when being thinner. A Tb layer structure is not well established.

Structures from amorphous to crystalline with some texture are reported by various authors. A special role in the PMA mechanism is attributed to the interface region, the thickness of which is estimated to be about 10 Å, slightly depending on substrate deposition temperature (the interface region is thinner for a cooled substrate). A controversy exists on the effect of heat treatment on both interface region and PMA. While Sajieddine *et al.* (1996) report an enhancement of PMA for Fe(30 Å)/Tb(22 Å) by annealing, which is attributed to a homogenisation of the interface towards an Fe–Tb amorphous alloy, Scholz *et al.* (1991) have found that annealing cancelled PMA for the similar system and, simultaneously, the interface phase disappeared. A suppression of PMA is also shown by Fe/Tb after 100 keV Ar ions irradiation (Tosello *et al.* 1996).

Although the RE atoms are assumed to play a crucial role in the PMA mechanism, the behaviour of their magnetic moments has been studied much less than that of the Fe ones (mostly by ^{57}Fe Mössbauer spectroscopy), probably because of experimental difficulties. Except for the results reviewed in this paper we are aware only of the other following studies: Hosoi *et al.* (1989, 1992) have deduced some information about the RE magnetic moment behaviour in Fe/Dy and Fe/Nd multilayers from neutron diffraction studies. A Tb out-of-plane magnetisation in Tb/Fe was found by circular magnetic x-ray dichroism (Attenkofer *et al.* 1993). Surprisingly, a random Dy magnetic moment orientation in Fe/Dy was reported (^{161}Dy Mössbauer spectroscopy studies), while the Fe magnetic moments were found to show PMA (Tappert *et al.* 1996). Thus, as one can see, more direct measurements would be very helpful.

4. NO Experiments with the Fe/Tb Multilayers

(4a) Samples

The $[\text{Fe}(40 \text{ Å})/\text{Tb}(x \text{ Å})]_{30}$ ($x = 5, 10, 20$ and 30) multilayers were prepared by alternate evaporation in UHV (10^{-9} Torr range) on a polyimide film that was kept at -50°C to minimise an intermixture at the interfaces. A Cu layer with a thickness of 3000 Å was then evaporated onto the multilayers to protect them and to enable soft soldering to the cold finger of a refrigerator. The samples were supplied by Professor Shinjo's group at Kyoto University (see e.g. Hosoi *et al.* 1989, 1992; Mibu *et al.* 1993 for other results with similar systems).

To obtain the radioactive probe ^{160}Tb the samples were activated in a reactor, in an Ar atmosphere and at temperatures below 80°C . The total neutron dose was of the order of 10^{18} cm^{-2} .

(4b) Experimental Details

The NO experiments were carried out using the Leuven and Prague NO facilities in the temperature range 7–50 mK and the B_{ext} range 0–8.5 T, B_{ext} being both parallel and perpendicular to the sample plane. Up to three pure Ge detectors (one always along B_{ext} and the other two only in case of B_{ext} along the sample plane being placed as shown in Fig. 3) were used to monitor the γ -rays. Here only the results of the 299 keV line are presented and discussed. A ^{54}Mn : Ni NO thermometer was used.

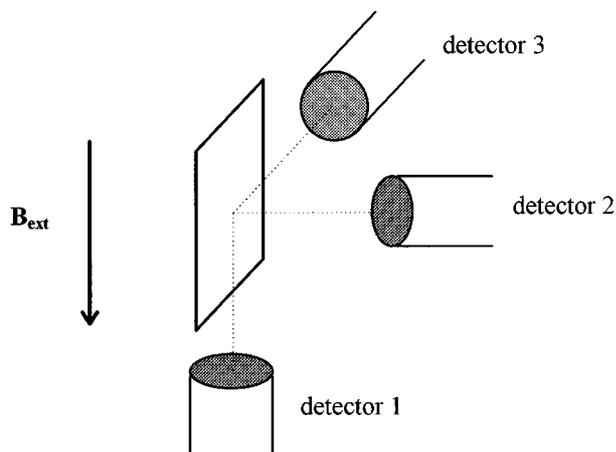


Fig. 3. Three detectors set-up for the low B_{ext} measurement.

(4c) Hyperfine Interaction Information

To find the hyperfine interaction of Tb in our multilayers, the temperature dependence of W was measured at several B_{ext} (see Fig. 1). The data for the 299 keV line were fitted with three free parameters, i.e. B_{hf} , V_{zz} and $\langle P_2 \rangle$, using (1). We have found that two of these parameters, i.e. B_{hf} and V_{zz} , are almost completely correlated with each other. This is caused by the fact that W is close to the saturation region in the temperature range measured and is predominantly controlled by the population of the two lowest Zeeman nuclear sublevels ($m = 3$ and 2 for the ^{160}Tb case), the energy gap of which is given by a linear combination of B_{hf} and V_{zz} only (see e.g. Brewer 1990). Therefore we fixed $V_{zz} = 38.9 \times 10^{21} \text{ V m}^{-2}$, which is the value for Tb in bulk Tb (Brewer *et al.* 1988; Sano and Itoh 1972) and close to the value for Tb in Tb-Fe intermetallics (e.g. in TbFe_2 — $39.9 \times 10^{21} \text{ V m}^{-2}$; de Azevedo *et al.* 1985). Using such an approach we obtained the value of B_{hf} to be 365(10) T for all our samples, with no visible correlation with the Tb layer thickness. This value coincides with that of Tb in TbFe_2 (371 T; de Azevedo *et al.* 1985), but is rather far from that of Tb in bulk Tb (304.7 T; Brewer *et al.* 1988; Sano and Itoh 1972). A similar tendency was found for Dy in $\text{Fe}(40 \text{ \AA})/\text{Dy}(48 \text{ \AA})$ (Tappert *et al.* 1996) and its origin is not yet clear.

(4d) Low B_{ext} Measurements

The B_{ext} dependence of W for $T \leq 9 \text{ mK}$ was measured for $B_{\text{ext}} \leq 1 \text{ T}$ using three detectors in the geometry shown in Fig. 3. Some results [i.e. W for the $\text{Fe}(40 \text{ \AA})/\text{Tb}(10 \text{ \AA})$ and $\text{Fe}(40 \text{ \AA})/\text{Tb}(20 \text{ \AA})$ multilayers] are shown in Fig. 4. As was mentioned above, B_2 is fully saturated in this temperature range and we could easily obtain information on the Tb magnetic moment direction (i.e. $\langle P_2 \rangle$) from W considering proper $Q_k U_k A_k$ and the saturation value of B_2 . Nevertheless, to extract more accurate information, we recalculated the measured low-temperature W values for $\langle P_2 \rangle$ using equation (1) and also proper B_{hf} , V_{zz} and T . The results of such a recalculation are shown in Fig. 5.

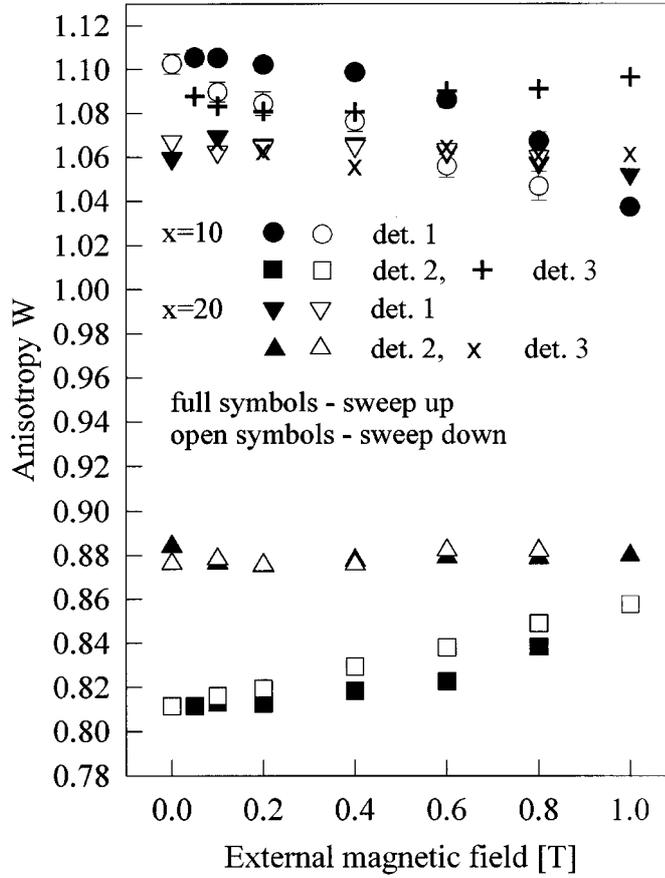


Fig. 4. The B_{ext} dependence of W for $\text{Fe}(40 \text{ \AA})/\text{Tb}(x \text{ \AA})$, with low B_{ext} and $T \leq 9 \text{ mK}$, and for three detectors (see Fig. 3).

One can see that detector 2, placed along the sample plane normal, gives $\langle P_2 \rangle > 0$, while detectors 1 and 3, placed in the sample plane, give $\langle P_2 \rangle < 0$. This can be taken as direct evidence that the Tb magnetic moments do not lie in the sample plane, but are turned out of the plane (compare Fig. 2) and show PMA. Moreover, these magnetic moments are turned out of plane more than would correspond to the random misalignment ($\langle P_2 \rangle = 0$). The zero B_{ext} points show a symmetry around the direction of the sample plane normal, i.e. $\langle P_2(\text{det. 2}) \rangle / \langle P_2(\text{det. 1}) \rangle = -2$ and $\langle P_2(\text{det. 1}) \rangle = \langle P_2(\text{det. 3}) \rangle$. When B_{ext} is applied in the plane the Tb magnetic moments are slightly turned in the direction of B_{ext} , which is demonstrated by the decrease of $\langle P_2(\text{det. 2}) \rangle$ and increase of $\langle P_2(\text{det. 1}) \rangle$, while $\langle P_2(\text{det. 3}) \rangle$ stays approximately unchanged.

(4e) High B_{ext} Measurements

For the B_{ext} range of 0–8.5 T the anisotropy W was measured at low temperature with a detector placed in the B_{ext} direction only. Two different geometries of B_{ext} and the sample were used: B_{ext} along the sample plane and

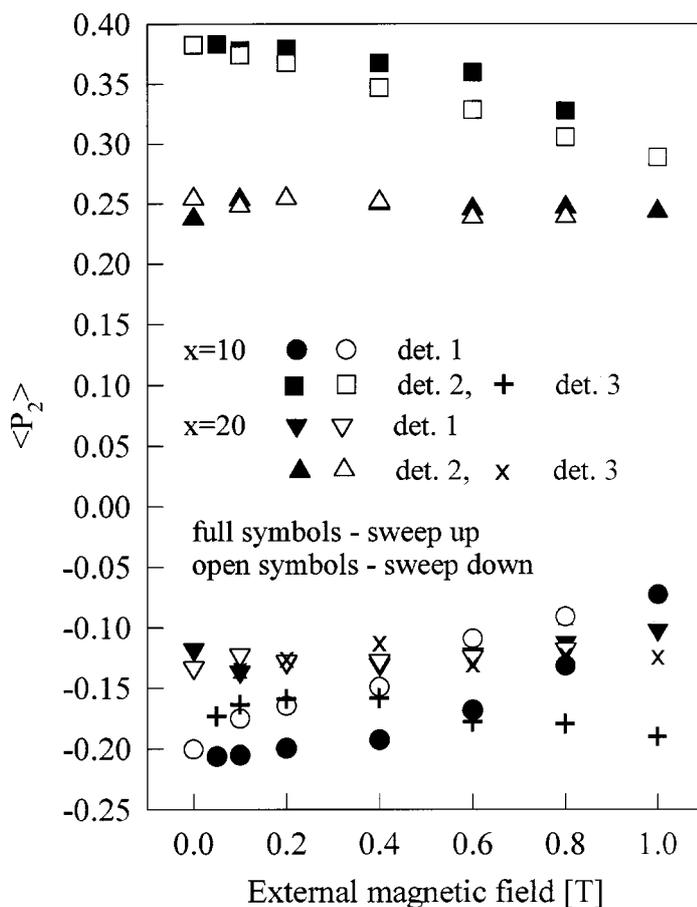


Fig. 5. The B_{ext} dependence of $\langle P_2 \rangle$ for Fe(40 Å)/Tb(x Å), with low B_{ext} , and for three detectors (see Fig. 3).

B_{ext} along the sample plane normal. Here W was recalculated for $\langle P_2 \rangle$ as in Section 4d and some results of the latter geometry are shown in Fig. 6.

(4f) Further Discussion

As was already pointed out in Section 4d, the most interesting feature of the multilayers studied, found by NO, is the presence of PMA of the Tb layer. This result contradicts the other results, which found a random RE magnetic moment misalignment in Fe/RE (i.e. Fe/Dy—see Section 3). On the other hand, our results support models of PMA which emphasise a dominant role of RE in the PMA mechanism.

A significant dependence of PMA on the Tb layer thickness was found. From a summary of the Tb layer PMA dependence in Fig. 7 one can see that at zero B_{ext} PMA increases when the Tb layer thickness decreases, but this tendency is broken down for the Tb layer thinner than 10 Å. The model proposed by us recently (Trhlík *et al.* 1996a, 1997), i.e. that only a thin Tb layer (adjacent to the

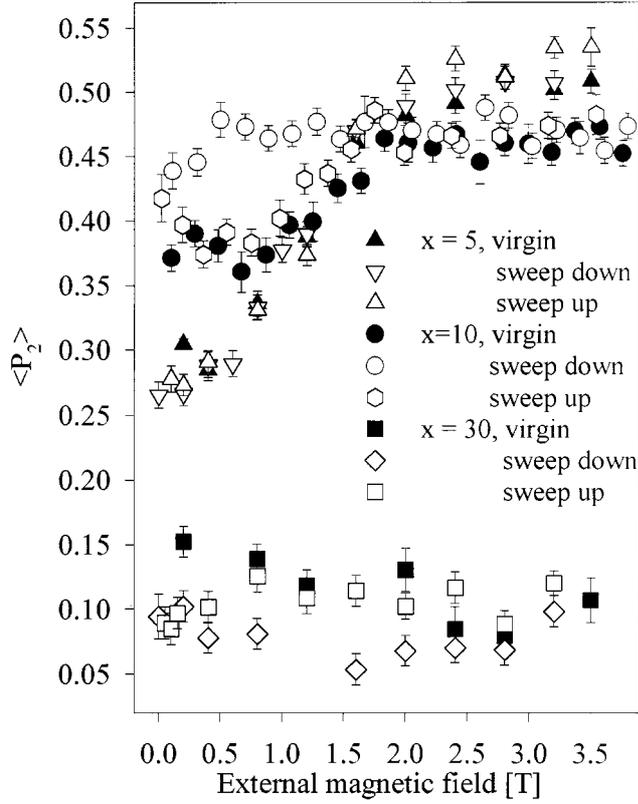


Fig. 6. The B_{ext} dependence of $\langle P_2 \rangle$ for Fe(40 Å)/Tb(x Å), with high B_{ext} . Here B_{ext} is parallel to the sample plane normal and the detector is parallel to B_{ext} .

Fe–Tb interface) shows PMA while the rest of Tb is randomly oriented—which could explain the tendency mentioned above—seems to be too simplified. The break-down thickness (~ 10 Å) roughly coincides with an assumed thickness of the Fe–Tb interface phase (see Section 3) and this coincidence could mean that just the Fe–Tb ‘interface’ phase is responsible for PMA.

A mutual interplay of the shape anisotropy of this composite system (consisting of Fe, Fe–Tb and Tb and having also magnetic inhomogeneity), PMA around or in the interface and other interactions (exchange, crystalline anisotropy) may create complicated structures. The complexity of the system is manifested e.g. by ambiguous influence of B_{ext} : (i) B_{ext} applied along the sample plane normal amplifies PMA for thinner Tb layer and suppresses it for thicker ones (see Fig. 7), (ii) a semi-saturation is reached at different B_{ext} , (iii) magnetic hysteresis appears at different B_{ext} : at higher values for Fe(40 Å)/Tb(30 Å) [and also for Fe(40 Å)/Tb(20 Å)—not shown in Fig. 6 for simplicity], at lower B_{ext} for Fe(40 Å)/Tb(10 Å) and almost missing for Fe(40 Å)/Tb(5 Å).

It is clear that, although NO can bring unique information on the Tb magnetic moment structure (i.e. $\langle m_{\text{Tb}}^2 \rangle$), this method cannot fully describe the complicated

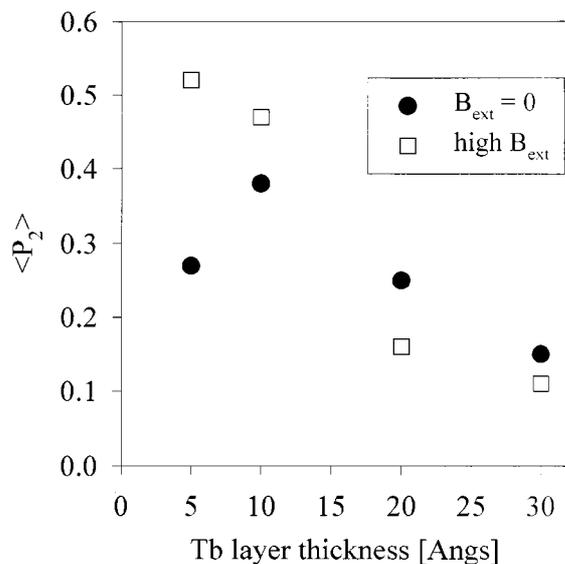


Fig. 7. The Tb layer thickness dependence of PMA characterised by $\langle P_2 \rangle$. The $\langle P_2 \rangle$ values were measured by a detector placed along the sample plane normal. The field B_{ext} was also applied along the sample plane normal.

magnetic structures mentioned. To solve the whole problem, a simultaneous study by magnetisation measurements (giving $\langle \mathbf{m}_{\text{Tb}} + \mathbf{m}_{\text{Fe}} \rangle$) and by ^{57}Fe Mössbauer spectroscopy (giving $\langle \mathbf{m}_{\text{Fe}} \rangle$) at low temperature (or magnetic ground state) is necessary and is in progress.

5. Conclusion

The potential of NO in the study of LD systems was outlined. As an example of the application of this technique the NO study of Fe/Tb multilayers was presented and discussed. Direct evidence of PMA of the Tb magnetic moments was given and it was shown that PMA increases when the Tb layer thickness decreases. The field B_{ext} has a complicated influence on the Tb magnetic moment misalignment, which we connect with an interplay between PMA, the exchange interactions, the shape and magnetic crystalline anisotropies.

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