

# Conversion electron Mössbauer spectroscopy with a linearly polarized source

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## Abstract

A Mössbauer polarimeter setup is presented here in combination with conversion electron Mössbauer spectroscopy and it is applied for the study of the ‘bulk-spin-flop’ transition, the switching of the Fe-layer magnetizations in an MgO/ $^{57}\text{Fe}(2.6\text{ nm})/\text{Cr}(1.3\text{ nm})_{20}$  antiferromagnetically-coupled, epitaxial superlattice.

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## 1. Introduction

Mössbauer spectroscopy (MS) has been very successful in determining the local magnetic and electronic structure in non-isotropic (thin single crystal, epitaxial film, textured foil and textured powder) absorbers even by using an unpolarized single-line source. The full analytical potential of the polarization dependence of the relative intensities can of course only be exploited when using a magnetically split source. Source matrix polariza-

tion in transverse or longitudinal magnetic field, sometimes combined with filter techniques has been reported in the literature. These methods are often called ‘Mössbauer polarimetry’ and have been elaborated theoretically and experimentally in detail long time ago [1–11].

Magnetic thin films and nanostructures display a richness of magnetic properties not present in bulk materials [12]. In thin magnetic films the magnetization, as a rule, due to shape anisotropy, is confined in the film plane. However, strain, local alloying or other effects may result in strong out-of-plane surface and interface anisotropy and a consequent out-of-plane magnetization (e.g. [13]). Due to the relatively shallow escape range of the low-energy electrons emerging in consequence to

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the nuclear de-excitation, conversion electron Mössbauer (CEM) spectroscopy has become an established local probe method in the analysis especially of Fe-containing thin films and surfaces. In numerous  $^{57}\text{Fe}$  Mössbauer studies out-of-plane magnetization was observed through the variation of the second and fifth line intensity as a function of the decreasing film thickness (e.g. [14]).

Conventionally, CEMS is performed with a single-line (unpolarized) source with  $\gamma$ -rays at perpendicular incidence to the film surface. However, such experiments provide no information about the direction of the magnetic hyperfine field *within the film plane*, which may be vital in various thin film studies. Antiferromagnetically (AF-) coupled multilayer systems of epitaxial confinement on single crystal substrates [15–17] and exchange spring structures [18–20] are recent examples to mention. Sometimes a series of spectra are taken with an unsplit source at various angles of  $\gamma$ -ray incidence [19–21], but in such experiments, the respective projection of the hyperfine field can only be determined with a large experimental error.

Here we report for the first time on a combined application of Mössbauer polarimetry and conversion electron Mössbauer spectroscopy. This technique uses a linearly polarized source and perpendicular incidence of the  $\gamma$ -rays; however,

the polarization direction of the source can be finely adjusted. Technical details of such a polarimeter setup and its application to study the bulk spin-flop transition in an [ $^{57}\text{Fe}(2.6\text{ nm})/\text{Cr}(1.3\text{ nm})$ ] $_{20}$  epitaxial multilayer are presented here.

## 2. Experimental

The magnetically split  $^{57}\text{Co}(\text{Fe})$  source was prepared by electrodeposition of  $^{57}\text{Co}$  onto a high-purity  $\alpha$ -Fe foil of 15  $\mu\text{m}$  thickness followed by a diffusion heat treatment at 1093 K in  $4 \times 10^{-4}$  Pa [22].

The schematic arrangement of the polarimeter is shown in Fig. 1. The source foil was placed and vibrated within the poles of a ‘polarizer’: a pair of NdFeB permanent magnets connected with a butterfly-shaped iron yoke and homogenizing iron armature producing a magnetic field of 270 mT in the source foil plane. (The homogeneity of the field was better than  $10^{-3}$  over the active source surface of 5 mm diameter.) The polarizer was mounted on an optical bench fixed to the yokes of an electromagnet. The polarizer could be rotated with an accuracy of  $\pm 1$  degree around the optical axis relative to the vertical field direction of the electromagnet.

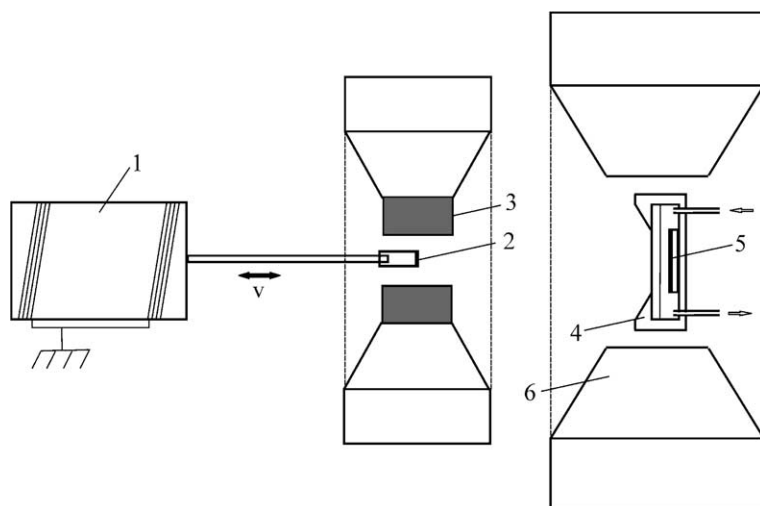


Fig. 1. The schematic arrangement of the polarimeter: (1) Mössbauer drive, (2)  $^{57}\text{Co}:\alpha\text{-Fe}$  source, (3) rotatable polarizer permanent magnets, (4) gas flow proportional counter, (5) sample and (6) electromagnet.

The samples were placed on the optical axis between the poles of the electromagnet. In CEMS experiments, the CEM detector itself enclosing the sample was placed between the electromagnet poles on a goniometer ensuring fine ( $\pm 0.1^\circ$ ) in-plane rotational adjustment of the detector and sample relative to the external field direction. The detector, a home made gas flow single-wire proportional counter was operated with a 95.3% He, 4.7% CH<sub>4</sub> gas mixture at a bias voltage of  $800 \pm 10$  V. The counter's internal chamber is of cylindrical shape with a diameter of 20 and height of 2 mm, the sample surface and an aluminised Mylar window providing the two opposite bases of the cylinder. The wire is mounted symmetrically at 1 mm distance from the sample surface. No change in the performance of the CEM detector was found up to 400 mT, the maximum field of the electromagnet in the described geometry. Although the vector sum of the internal and external fields was not identical in source and absorber in the different applied external fields, with the typically 0.24 mm/s FWHM (corresponding to 0.75 T) the deviations remained small to be accommodated within the line width of the components even at the maximum available field. Care was taken to decrease the stray field at the preamplifier and at the velocity drive, to 0.09% and 0.05%, respectively. At 400 mT central field, the change in the velocity calibration factor and zero velocity channel remained within the experimental error.

Transmission experiments were performed replacing the CEM detector with an absorber foil. Source-test transmission spectra were recorded with a 95.5% isotopically enriched <sup>57</sup>Fe calibration absorber film (of 200 nm thickness, capped with 30 nm Si) evaporated onto a thin mica substrate of negligible absorption at the 14.4 keV energy of the transition. A [<sup>57</sup>Fe(2.6 nm)/Cr(1.3 nm)]<sub>20</sub> epitaxial multilayer (superlattice) grown on MgO(001) substrate at 450 K by molecular beam epitaxy at  $4 \times 10^{-10}$  mbar base pressure was used in the spin-flop studies. The sample was one and the same piece as described elsewhere [15,16]. Magnetization measurements indicated AF coupling with a saturation field of  $H_s = 0.9$  T. After having exposed the Fe/Cr multilayer to a certain (increasing) in-plane external field CEM spectra were recorded in zero

external field. All experiments were performed at ambient temperature using the same <sup>57</sup>Co( $\alpha$ -Fe) source polarized in-plane by a field of 270 mT.

### 3. Results and discussion

The angular dependence of the intensity of the Mössbauer spectrum lines was first calculated by Frauenfelder et al. [23]. Here we restrict the discussion to the case of metallic <sup>57</sup>Fe below the Curie point with pure magnetic dipole radiation. Using an unsplit absorber (of Na<sub>4</sub>[Fe(CN)<sub>6</sub>]·10H<sub>2</sub>O) the Mössbauer line pattern of an <sup>57</sup>Co: $\alpha$ -Fe source splits into six lines with relative intensities  $\alpha$ ,  $\beta$ ,  $\gamma$ ,  $\delta$ ,  $\varepsilon$ ,  $\eta$  (from negative to positive velocities, from left to right in Fig. 2(a)) corresponding to the ( $3/2 \rightarrow 1/2$ ), ( $1/2 \rightarrow 1/2$ ), ( $-1/2 \rightarrow 1/2$ ), ( $1/2 \rightarrow -1/2$ ), ( $-1/2 \rightarrow -1/2$ ) and ( $-3/2 \rightarrow -1/2$ ) transitions, respectively. The intensity of each component is a function of the angle  $\theta$  between direction of the emission wave vector and  $H_i$ , the field at the nucleus (both in the source and absorber) and it is given by  $\alpha = \eta = 3(1 + \cos^2 \theta)/2$ ,  $\beta = \varepsilon = 2 \sin^2 \theta$ ,  $\gamma = \delta = (1 + \cos^2 \theta)/2$ . The absorber quantities (corresponding to the unsplit source-split absorber (magnetized <sup>57</sup>Fe) experiment in Fig. 2(b) are primed.

In a split-source, split-absorber experiment, the respective external magnetic fields  $B$  and  $B'$ , and also the respective hyperfine fields  $H_i$  and  $H'_i$ , may point in different directions. In the present experiments, both source and absorber are dominantly magnetized perpendicular to the wave vector. This  $\theta = 90^\circ$  transversal Zeeman effect results in the relative intensities of 3:4:1 ( $\alpha = \eta = 3$ ,  $\beta = \varepsilon = 4$ ,  $\delta = \gamma = 1$ ) of the outer:middle:inner lines in a split source–unsplit absorber or an unsplit source–split absorber experiment (Fig. 2(a) and (b), respectively). It is therefore natural to use a common coordinate system for each equivalent sites in which the  $z$ -axis points in the direction of the wave vector (optical axis) and the  $x$ -axis is parallel to the external field  $B$  on the source. The azimuth angle of the hyperfine field of the absorber is  $\varphi$ , therefore the resulting parallel and perpendicular intensity components scale by  $\cos^2 \varphi$  and  $\sin^2 \varphi$ . In the above geometry the fully resolved emitted/

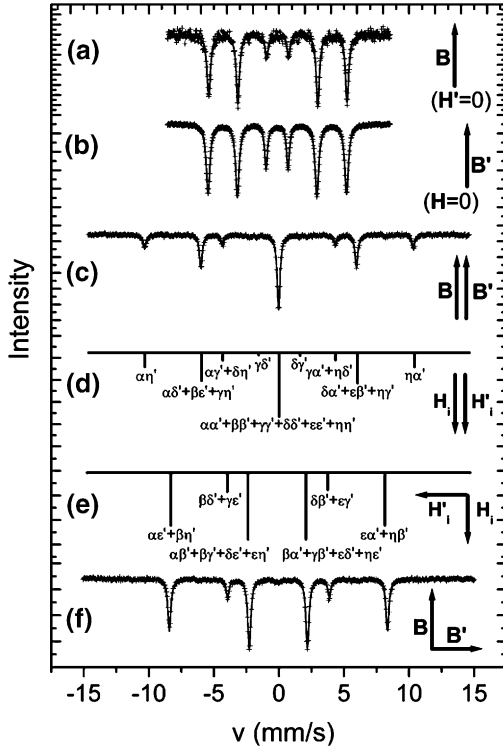


Fig. 2. Experimental spectra and stick diagrams of a  $^{57}\text{Co}:\alpha\text{-Fe}$  source versus a single line  $\text{Na}_4[\text{Fe}(\text{CN})_6]\cdot 10\text{H}_2\text{O}$  absorber (a),  $^{57}\text{Co}:\text{Rh}$  (single line) source versus an  $\alpha\text{-Fe}$  evaporated absorber film of 200 nm thickness (95.5% enriched in  $^{57}\text{Fe}$ ) (b),  $^{57}\text{Co}:\alpha\text{-Fe}$  source versus an  $\alpha\text{-Fe}$  evaporated absorber film of 200 nm thickness (95.5% enriched in  $^{57}\text{Fe}$ ), both in 270 mT external field, perpendicular to the optical axis in parallel and perpendicular orientations, (c,d) and (e,f), respectively. The symbols  $\alpha, \beta, \dots, \eta$ ,  $B$  and  $H_i$  represent the six absorber (unprimed) and source (primed) lines and the external and internal (hyperfine) magnetic fields, respectively. Note, that the internal field is antiparallel with the external one [24].

absorbed spectral lines are linearly polarized for all ( $\Delta m = 0, \pm 1$ ) transitions ( $\alpha, \alpha'$ : parallel,  $\beta, \beta'$ : perpendicular,  $\gamma, \gamma'$ : parallel,  $\delta, \delta'$ : parallel,  $\varepsilon, \varepsilon'$ : perpendicular and  $\eta, \eta'$ : parallel to the respective magnetic fields). In turn, only those absorber lines (of  $\alpha', \beta', \gamma', \delta', \varepsilon', \eta'$ ) are excited by any emission line of the source, which have the same polarization. Therefore in the present simple case one may express the spectra in terms of ‘parallel’ and ‘perpendicular’ components corresponding to the respective orientations of the homogeneous  $H_i$  magnetic field in the source relative to  $H'_i$  in the absorber (cf. Fig. 2(d) and (e)).

The degree of source polarization was determined first, using the  $^{57}\text{Fe}$  calibration film absorber of 200 nm thickness. Two transmission experiments were performed with parallel and with perpendicular fields on the polarizer and on the sample ( $\varphi = 0^\circ$  and  $90^\circ$ ), Fig. 2(c) and (f), respectively. The resulting spectra are convolutions of the six-line spectrum of source (with left-to-right relative intensities  $\alpha, \beta, \dots, \eta$ ) and the six-line spectrum ( $\alpha', \beta', \dots, \eta'$ ) of the absorber. Spectra corresponding to the two polarization states are independent. Consequently, in the ‘parallel case’ a line appears only if the respective hyperfine field component has the same direction. If these polarizations are perpendicular, then the line will appear in the spectrum only if the internal fields in source and absorber are aligned at right angles [24]. Transmission spectra were evaluated using a transmission integral fit by EFFINO [25] making use of the full polarization treatment of both source and absorber [26]. The theoretical spectra assuming full linear polarization ( $\alpha = \alpha' = \eta = \eta' = 3/2$ ,  $\beta = \beta' = \varepsilon = \varepsilon' = 2$ ,  $\gamma = \gamma' = \delta = \delta' = 1/2$ ,  $\theta = \theta' = \pi/2$ ) are shown in Fig. 2(d) and (e) for  $\varphi = 0$  and  $\varphi = \pi/2$ , respectively. The experimental results agree fairly well with the theoretical curves of full (parallel or perpendicular) polarization, however, a slight polarization admixture due to incomplete polarization of either source or absorber can also be observed. In fitting we assumed this minor admixture to be fully due to the other linearly polarized state. The fit provided an upper margin of 4.3% and 3.4% admixture of the other polarization state in the parallel and in the perpendicular cases (full fit curves in Fig. 2(c) and (f)), respectively. Further assuming the absorber to be 100% linearly polarized a fair *lower margin* of 96% is obtained for the source polarization itself.

The polarimeter setup calibrated this way was first applied to study the bulk spin–flop transition in a Fe/Cr epitaxial multilayer film. The periodic Fe/Cr AF-coupled epitaxial superlattice with even number of Fe layers is an interesting model of an ‘artificial layer antiferromagnet’. The neighbouring layer magnetizations point parallel and antiparallel to either of the two mutually perpendicular easy axes in the film plane (Fe[0 1 0] and Fe[1 0 0], parallel to MgO[1 1 0] and MgO[1  $\bar{1}$  0] directions of the

MgO(001) single crystal substrate, respectively). Generating an increasing external in-plane magnetic field by the electromagnet, the anisotropy-stabilized configuration becomes energetically unfavourable and at a critical field strength a sudden magnetization switching, the ‘bulk spin-flop’ (BSF) transition into the perpendicular easy axis is expected to happen. BSF had been studied by Synchrotron Mössbauer Reflectometry (SMR) [15] on the same  $[^{57}\text{Fe}(2.6\text{ nm})/\text{Cr}(1.3\text{ nm})]_{20}$  sample which was used in the present studies, and by polarized neutron reflectometry (PNR) on a sample of practically identical nominal  $[^{\text{nat}}\text{Fe}(2.5\text{ nm})/\text{Cr}(1.3\text{ nm})]_{20}$  composition [17]. Similar to the procedure in [15], the initial magnetic state of the film was carefully prepared by aligning the layer magnetizations parallel/antiparallel along a *single* easy axis. This easy axis was then adjusted parallel to the field direction of the polarizer and that of the electromagnet (the latter first in zero external field). Some typical conversion electron Mössbauer polarimetric spectra are displayed in Fig. 3(a)–(c).

The effective escape depth of the conversion electrons of the 14.4-keV transition of  $^{57}\text{Fe}$  (from where appreciable electron yield is detected) for Fe is about 1000 FeÅ (1 FeÅ = 0.0786  $\mu\text{g}/\text{cm}^2$ ) [27]. The total thickness of the above multilayered sample (regarded it as a ‘homogeneously active’ absorber [27]) is 768 FeÅ, a value less but comparable with the effective escape depth. Using expression (6) of [27] the contribution to the total electron yield in the CEM spectrum from the top  $^{57}\text{Fe}$  layer is a factor of three bigger than that of the bottom layer. Non-periodicity would therefore result in different ratios of the two linearly polarized contributions depending on whether the given component is in majority on top or at the bottom of the multilayer stack. The sample preparation, however, ensured a rather perfect periodicity. The evaluation was made using the following assumptions: (i) the multilayer sample is strictly periodic, (ii) the source is 100% linearly polarized in-plane in the  $\varphi = 0$  direction, (iii) spectra consist only of the two independent linearly polarized (‘parallel’,  $\varphi = 0$  and ‘perpendicular’,  $\varphi = 90^\circ$ ) components of (iv) pure  $\alpha$ -iron hyperfine pattern. The so fitted fraction of the perpendicular compo-

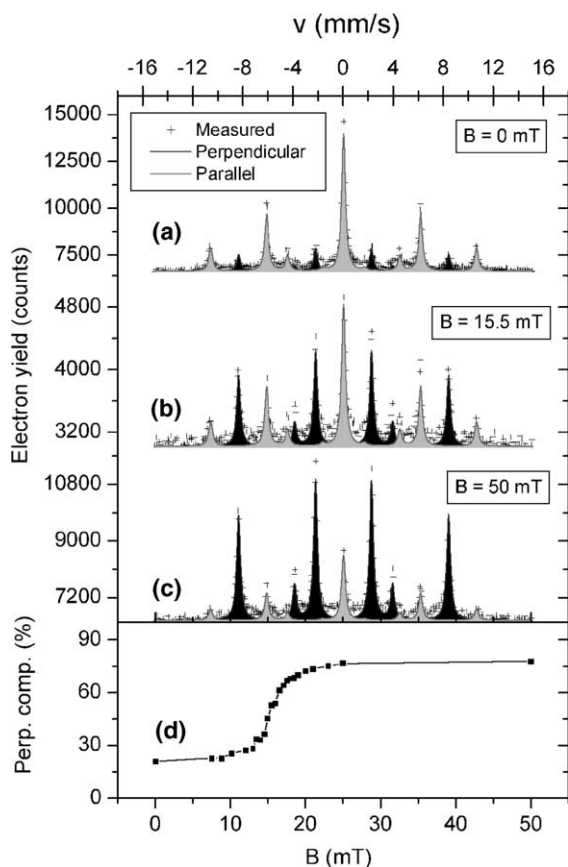


Fig. 3. Selected Mössbauer spectra (a–c) of a polarimetric study of the bulk spin-flop transition in a  $\text{MgO}/[\text{Fe}(2.6\text{ nm})/\text{Cr}(1.3\text{ nm})]_{20}$  using a linearly polarized  $^{57}\text{Co}(\alpha\text{-Fe})$  source. After having exposed the Fe/Cr multilayer to a certain in-plane external field, the CEM spectrum was recorded in zero external field. In increasing magnetic fields, around 15.5 mT, the layer magnetizations that were initially aligned parallel/antiparallel to the field, switch into the perpendicular easy direction, which is manifested in an increase of the perpendicularly polarized (black) spectrum component, the fraction of which is plotted in (d).

nent in Fig. 3(d) displays a transition centred at 15.5 mT with a transition width of 2.7 mT.

The BSF transition field was estimated to 13.5 mT in [16]. The present value of 15.5 mT obtained by CEM polarimetry seems more reliable, since the normalisation of the AF Bragg peak intensity is slightly subject to the accuracy of sample alignment procedure. The transition width depends on the details of the AF domain structure. When the present CEM spectra were taken, the sample had



already experienced bulk spin-flops after the last full saturation; therefore it consisted of majority large domains ('secondary domain state' [16]). Presently we have no model to relate the domain size distribution to the bulk spin-flop transition width.

At zero and 50 mT a remarkably high admixture of 20% of the other polarization component is found, i.e. a large fraction of the multilayer 'does not flip'. One possible reason for the 'non-flipping' domains is a large ferromagnetic (FM) component in the magnetization pinned by the external field. However, according to vibrating-sample magnetometry loops on this sample, the FM component is only 5% of the total film volume. This component is non-periodic and shows a hyperfine field distribution. First, it is indeed non-periodic, since at zero field, there was no detectable SMR AF peak intensity [15] on the same multilayer sample. Second, the hyperfine field distribution is justified, since considerable intensity of satellite components in the polarimetric spectra (cf. spectra and fits in Fig. 3(a)–(c)) is not taken into account by this simplified evaluation performed simply in terms of  $\alpha$ - $^{57}\text{Fe}$  Mössbauer spectra. A conventional perpendicular incidence CEM spectrum on this sample (taken with a single-line source, not shown here) shows 60% unperturbed (core Fe) and 40% perturbed (interface Fe/Cr, with hyperfine field distribution) contribution. Assuming the interface component to remain random in these relatively small fields,  $40/2 = 20\%$  of the sites on average, contribute to one of the two perpendicular polarization directions, which is indeed the case in Fig. 3.

This sensitivity to the random interface contribution shows a remarkable advantage of the present polarimetric technique over reflectometry [15–17], which is primarily sensitive to the periodic component of the interface contribution. Moreover, while SMR and PNR can only be performed at large facilities, the present technique needs relatively simple laboratory experiments.

#### 4. Summary

In summary, a linearly polarized  $^{57}\text{Co}(\alpha\text{-Fe})$  magnetically split source assembly was fabricated with

a polarizing field of 270 mT, resulting in a linear polarization of the  $^{57}\text{Co}(\alpha\text{-Fe})$  source of at least 96%. The polarimeter setup was used to study the 'spin-flop' transition in a Fe/Cr multilayer. A sharp increase of the perpendicular component from 20% to 80% within a few mT was observed with an average spin-flop field of 15.5 mT. The non-flipped intensity is attributed to an extended random interface region in which the local magnetization and consequently the hyperfine field sensed by the  $^{57}\text{Fe}$  probes shows a broad angular distribution, not distinguishing between the macroscopic easy and hard directions of magnetization.

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