



# **Structure investigations and perturbed angular correlation measurements on magnetic multilayers**

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#### Abstract

The crystalline structure of epitaxially grown Fe/Cr and Co/Ag superlattices is investigated by comparing the information derived from X-ray diffraction with information derived from a microscopic technique like perturbed angular correlation (PAC). We illustrate the usability and sensitivity of the PAC technique to discriminate the possible structural phases and to probe the magnetisation by measuring the magnitude and orientation of the hyperfine fields.

## **1. Introduction**

Multilayers prepared by alternate deposition of magnetic and non-magnetic materials provide suitable specimens for studying the magnetic behaviour at interfaces and in two-dimensional systems.

The studied superlattices are MBE grown. In-situ RHEED is performed to monitor the quality of the superlattices during growth. The structural order is checked by the use of standard X-ray diffraction techniques using  $K_{\alpha}(Cu)$  wavelengths.

A microscopic magnetic study by means of hypeffine field (hff) measurements can be done by perturbed angular correlation (PAC) spectroscopy [1]. This technique determines the nuclear Larmor frequency  $\omega_L = B_{hf} \mu / \hbar I$  by measuring the time variation of the anisotropy in nuclear radiation emitted by an oriented probe nucleus.  $I$  is the nuclear spin of the probe and  $\mu$  its magnetic moment. The anisotropy function  $R(t)$  is calculated by proper combinations of the 90° and 180° detector pairs. A Fourier transform of the  $R(t)$  spectrum for each probe environment contains a frequency (and its harmonics) that is directly related to the local magnetic field.

The <sup>111</sup>Cd activity is implanted at 80 keV in the multilayers in doses of up to  $10^{13}$  ions/cm<sup>2</sup>. A conventional four  $BaF<sub>2</sub>$  detector slow-fast set-up with 0.7 ns (FWHM) time resolution is used.

## **2. Study of Fe / Cr multilayers**

A series of superlattices with Fe thickness  $t_{Fe} = 30$  A and a variable Cr thickness 6 A  $\leq t_{Cr} \leq 51$  A is epitaxially grown on a MgO(001) substrate. It is well known that very sharp interfaces exist, almost unbroadened by interdiffusion. The constituents grow epitaxially due to the small difference in lattice parameters. The giant magnetoresistance effect showed the well known oscillatory behaviour with a periodicity of 18  $\AA$  [2] and a short-period oscillation of periodicity  $3 \text{ Å}$  [3].

A typical PAC spectrum taken at room temperature on MgO/[Fe(30 Å)/Cr(6 Å)]<sub>10</sub> is shown in Fig. 1. The solid line is a theoretical fit, assuming a three-site model. A fraction of 43(1)% belongs to a site with a Larmor fiequency of 559(1) Mrad/s corresponding to the CdFe hyperfine field [4]. Another part, 19(1)% shows a low frequency, identified as probes in a Cr environment. Indeed, whereas Cr normally has a bee structure, strain induces small electric field gradients at the position of the nuclear probe, reflected in the  $R(z)$  spectrum by low electric quadrupole interaction frequencies. In this layer thickness the Cr is probed as non-magnetic. About 40% of the probes feel a poorly defined hff, observed as a frequency with a large ( $\delta_{\text{gauss}} \ge 100\%$ ) distribution. These probes are assigned to be at the interface or at grain boundaries.

#### 3. Study of Co/Ag multilayers

Corresponding author. Fax: +32-16 29 59 19; e-mail: JO Co-based multilayer structures have recently attracted

HAN = MEERSSCHAUT%rsf%fys@cc3.kuleuven.ac.be. considerable interest because of their magnetic properties,

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Fig. 1. PAC spectrum of <sup>111</sup>Cd implanted in MgO/[Fe(30 Å)/Cr(6  $\tilde{A}$ )]<sub>10</sub>-

such as perpendicular magnetic anisotropy [5] and enhanced magnetoresistanee effects [6].

Samples with varying Co and Ag thicknesses, growth temperatures and deposition rates are grown on MgO, using Fe or Cr as buffer layers. From in situ RHEED spectroscopy there is an indication that it is possible to grow metastable fcc Co epitaxial on fcc Ag.

The samples are characterized by X-ray diffraction (XRD). The high-angle  $\theta/2\theta$  patterns of MgO/Cr(50  $\frac{3}{4}$ )/[Ag(20  $\frac{3}{4}$ )/Co(10  $\frac{3}{4}$ )]<sub>10</sub> (sample A) and MgO/Cr(50  $\rm \AA$ )/[Ag(5  $\rm \AA$ )/Co(10 A)]<sub>10</sub> (sample B), grown at 200°C, and that of MgO/Fe(50 A)/[Ag(20 A)/Co(43 A)]<sub>10</sub> (sample C) grown at room temperature, are shown in Fig.



Fig. 2. High-angle X-ray pattern of samples A: MgO(100)/Cr(50  $\rm\AA$ )/[Ag(20  $\rm\AA$ )/Co(10  $\rm\AA$ )<sub>10</sub> B: MgO(100)/Cr(50  $\rm\AA$ )/[Ag(5  $A$ /Co(10 A)]<sub>10</sub> grown at 200°C; and C: MgO(100)/Fe(50  $A$ /[Ag(20 Å)/Co(43 Å)]<sub>10</sub> grown at room temperature. The peaks at  $43^{\circ}$  and  $94.3^{\circ}$  are from MgC, the peak at  $66^{\circ}$  from the buffer. The peak at  $44.5^\circ$ , if present, reflects epitaxial Ag(002) stacking. The Co peaks are explained in the text.



Fig. 3. PAC spectra of multilayer samples A and B *(see* caption to Fig. 2). The solid lines *are* theoretical fits.

2. The peaks at  $43^\circ$  and  $94.3^\circ$  correspond to  $(200)$  and (400) stacking of MgO. The peak at 44.5 °, if present, reflects epitaxial Ag(200) stacking. High-angle XRD experiments cannot give a decisive distinction between (220) stacking of fcc Co and (110) stacking of hcp Co, both of which result in a diffracting angle at  $2\theta = 75.9^{\circ}$  for  $K_{\infty}(Cu) = 1.5406$  Å. The peak at 53° can be due either to a strained fcc structure or to a body-centred tetragonal (bet) one, as suggested by Li et al. [7]. The metastable bee Co phase could eventually contribute to the peak at 66° and would not be distinguished from the buffer or substrate contributions.

PAC spectroscopy is used to identify these different crystalline environments. In Fig. 3 we compare the PAC spectra of samples A and B. The solid lines are theoretical fits based on the following arguments. Ag is non-magnetic



Fig. 4. Fourier transforms of the PAC spectra of samples A, B and C (see caption to Fig. 2). Lines 1, 2 and 3 correspond to bulk fee, stacking fault and hcp cobalt, respectively. Line 4 is the second harmonic of line 1, thus indicating other magnetization directions for fcc compared to hep Co.

but in a strained cubic structure, leading to a slow decay in the  $R(t)$  function, evidently more prominent in the sample with large Ag thickness (sample A). The oscillatory parts of the spectra reflect, through the Larmor frequency  $\omega_1$ , the hyperfine field in the Co layers in the hep and fee phases (415 and 346 Mrad/s, respectively [8]). We show in Fig. 4 the Fourier transforms of the PAC spectra for the three multilayers. The first peak as well as its second harmonic (peak 4) correspond to the hyperfine field in fcc Co, present in sample A. The spectrum of sample B reveals, besides fee (peak 1) and hcp (peak 3) structures, a predominantly stacking fault structure (378 Mrad/s, peak 2) which is suppressed in the Fourier spectrum due to its broad distribution (23 Mrad/s). The third sample can be analyzed using only hcp Co (line 3) with a frequency of 411 Mrad/s, as observed in bulk cobalt [8]. In none of the studied Co/Ag multilayers was the known hyperfine field of <sup>111</sup>Cd in the metastable bcc Co [9] observed.

## **4. Conclusions**

We conclude that PAC offers additional information compared to X-ray diffraction and RHEED spectroscopy for characterizing the different crystalline Co phases in Co/Ag multilayers. Because the nuclear probe is a diamagnetic atom, the PAC technique will be used to study the magnetism in Fe/Cr multilayers in the magnetic layer as well as the 'non-magnetic' spacer layer.

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#### **References**

- [1] Th. Wichert and E. Recknagel, in Microscopic Methods in Metals, ed. U. Gonser (Springer, Berlin, 1986) p. 317.
- [2] C.D. Potter, R. Schad, P. Beliën, G. Verbanck, V.V. Mcshchalkov and Y. Bmynseraede, Phys. Rev. B 49 (1994) 16055.
- [3] R. Schad, C.D. Potter, P. Beliën, G. Verbanck, V.V. Moshchalkov and Y. Braynseraede, J. Appl. Phys. 76 (1994).
- [4] B. Lindgren and Y.K. Vijay, Hyp. Int. 9 (1981) 379.
- [5] R. Krishnan, M. Porte and M. Tcssier, J. Magn. Magn. Mater. 103 (1992) 47.
- [61. W.P. Pratt, S.-F. Lee, J.M. Slaughter, R. Loloee, P.A. Schroeder and J. Bass, Phys. Rev. Lett. 66 (1991) 3060.
- [7] Hong Li and B.P. Tonner, Phys. Rev. B 40 (1989) 15.
- [8] F. Raether, D. Wiarda, K.P. Lieb, J. Chevallier and G. Weyer, Z. Phys. B 73 (1989) 467.
- [9] B. Swinnen, J. Dekoster, G. Langouehe and M. Rots, Phys. Rev. B submitted.