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# The anisotropic first-neighbour contribution to the hyperfine field in hexagonal-close-packed Co: a nuclear magnetic resonance study of diluted alloys and multilayers

M Malinowska<sup>†‡</sup>, C Mény<sup>†</sup>, E Jedryka<sup>†‡</sup> and P Panissod<sup>†</sup>

<sup>†</sup> Institut de Physique et Chimie des Matériaux de Strasbourg, 23 rue du Loess, 67037 Strasbourg, France

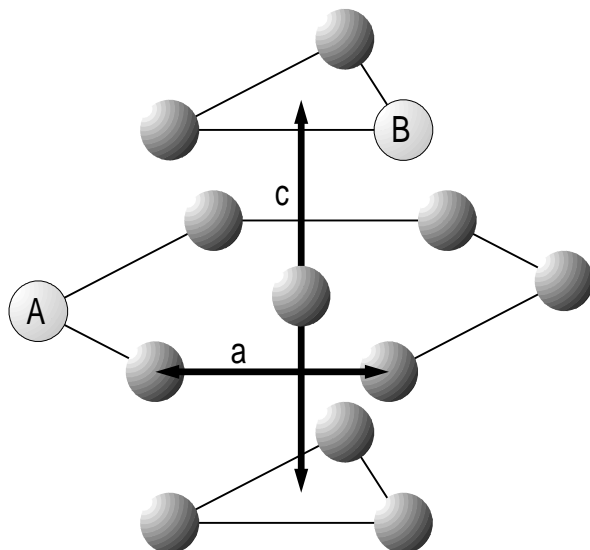
<sup>‡</sup> Institute of Physics, Polish Academy of Sciences, Allica Lotników 32/46, 02668 Warszawa, Poland

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**Abstract.** <sup>59</sup>Co NMR has been studied in CoCr, CoRu, CoMn and CoCu hexagonal alloys with impurity concentrations below 1 at.% and in two-dimensional alloys present at the interfaces of the corresponding Co/M multilayers, where Co adopts the hcp structure. Two low-frequency satellites have been observed for all of the alloys studied, whereas only one is observed for cubic alloys. Their origin is related to the presence of two inequivalent sites in the first coordination shell of Co, because hcp Co departs from the ideal compact structure. From the results on Co/Cr and Co/Ru multilayers it is deduced that the impurity neighbours out of the *c*-plane depress the Co hyperfine field less than the in-plane impurity neighbours. Comparison with the alloys CoCr and CoRu suggests a similar conclusion for CoMn alloys. As to CoCu systems, the situation is the opposite, although there is some ambiguity in the analysis of hcp Co/fcc Cu multilayers, because Co might be stacked in a fcc structure at the interface with Cu.

## 1. Introduction

*Ab initio* calculations of hyperfine fields in ferromagnetic materials have shown significant progression recently, mostly for isolated impurities in cubic alloys (see, e.g., [1–3]). The presence of an alien atom in the first-neighbour shell results in a shift of the hyperfine field of Co, the value of which is characteristic of a given impurity (see, e.g., [4–7]). Experimental information about the effects of impurity atoms on the hyperfine field of the matrix element can be obtained by the NMR study of diluted alloys. As a result of alloying, a number of regularly spaced satellite lines are observed in the NMR spectra of Co alloys, which correspond to the presence of one, two, etc, nearest-neighbour impurities (NN) around the Co atom. The effect of impurities being additive has been found to be generally the case for cubic alloys (with the exception of CoPt alloys) [6]. However, in the case of some alloys with the hcp structure, each satellite can be split further due to the anisotropic character of the structure, as previously suggested by Kawakami [7]. For example, in the case of CoCr alloys, the satellite lines that we observed in the NMR spectra are broad and asymmetric [6]. This asymmetry is a result of the two inequivalent positions at which a first-neighbour atom can be located in the hcp lattice. One site (labelled as an ‘A’-type site in Kawakami’s paper—see our figure 1), corresponds to atoms located in the close-packed plane of the central Co ion (distant from it by the lattice constant *a*). The other sites (‘B’-type sites) are those located in the planes above and below the central atom. In the case of an ideal



**Figure 1.** A schematic representation of first-neighbour positions in the hcp structure.

hcp structure (with a  $c/a$  ratio equal to 1.63), they are also distant from the central ion by the distance  $a$ . However, real hcp Co crystal has a  $c/a$  ratio slightly different from the ideal one (1.622) [8]. This slight modification of the distance from the central Co atom may introduce a difference in the way in which its hyperfine field is influenced by an impurity atom, depending on which type of site it will occupy. This effect has been previously observed in the form of two distinct satellite lines for **CoCr**, **CoMn**, **CoTi** and **CoV** alloys [7].

In this work we attempt to investigate in more detail this splitting of the first satellite in the following hcp Co alloys: **CoCr**, **CoRu**, **CoMn** and **CoCu** with impurity concentrations below 1 at.%. In order to attribute the values of satellite splitting obtained from the study of diluted alloys to one of the two inequivalent impurity sites, we have also analysed the results of NMR studies performed on the corresponding two-dimensional systems, i.e. multilayers, in which thin hcp Co layers alternate with layers of the respective metal. Indeed, in multilayers with  $\langle 001 \rangle$  growth orientation, only Co atoms at the interfaces have alien neighbours and, moreover, most of these neighbours lie in the adjacent hexagonal plane, so one of the two inequivalent neighbour positions is expected to dominate.

## 2. Experimental details

Polycrystalline hcp Co and its alloys with Cr, Mn, Ru, and Cu have been prepared by arc melting in a water-cooled copper hearth and remelting ten times to improve the homogeneity of the solid solution. The impurity concentrations were 0.2, 0.6 and 1.0 at.%. The purity of the starting materials used was 99.99 at.% for Co and at least 99.9 at.% for the impurity elements. The ingots were subsequently annealed at 900 °C for 24 h then slowly cooled down to 400 °C over two days and annealed at this temperature for four days. After this annealing treatment, a single hcp phase is observed in the samples by means of x-rays and by means of NMR as well. In order to preserve a homogeneous hcp phase, the NMR samples were cut from the ingots in the form of plates 0.2 mm thick (no grinding, no cold rolling),

and the x-rays indicated no preferred texture. The plates were further electro-polished to remove surface roughness that favours fcc stacking.

Co/X multilayers have been prepared by UHV evaporation on mica substrates covered with a hexagonal Ru buffer layer about 150 Å thick in order to favour hcp stacking. From investigations using *in situ* RHEED, *ex situ* x-ray measurements, and NMR, the Co/Cr and Co/Ru multilayers were found to be hcp with  $\langle 001 \rangle$  (*c*-axis) growth orientation. The structure of the Co/Mn multilayers was found to show considerable dependence on the thickness of Mn [9]; we shall not draw here any conclusion from their study. In most Co/Cu multilayers, both Cu and Co are fcc. However, we have succeeded in growing, by slow UHV evaporation, a single layer of Co, 20 Å thick and mostly hcp, on a  $\langle 111 \rangle$ -oriented single crystal of fcc copper; the Co layer was subsequently capped with an iron layer in order to avoid NMR signals from a second Cu/Co interface of bad quality.

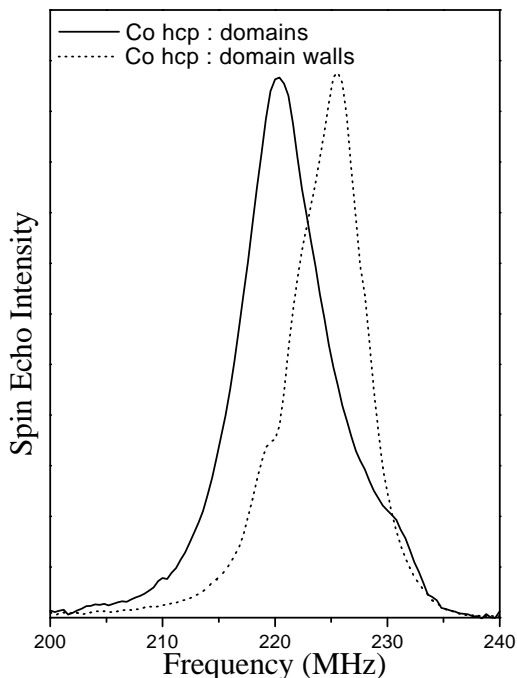
$^{59}\text{Co}$  NMR spectra were recorded every 1 MHz in zero external field. In the case of alloys, the frequency range 150–250 MHz was swept at 4.2 K. For the multilayers, the experiment was carried out at 1.6 K over the frequency range 60–240 MHz. An automated, phase-sensitive, spin-echo spectrometer was used (the same as the one used in [6]).

### 3. Results and discussion

#### 3.1. Pure hcp Co

Owing to the hyperfine-field anisotropy in hcp Co [10, 11], it is known that  $^{59}\text{Co}$  has different resonance frequencies depending on whether the atom is sited inside Bloch domains or at the centres of domain walls. These two frequencies measure the different values of the hyperfine field for which the local moment is along the *c*-axis or lies in the hexagonal plane, respectively. In multidomain samples like ours, both populations are present, but the two different signals are easy to observe separately, since they require different excitation conditions. Indeed, the enhancement factor due to domain wall displacement is much larger at domain wall centres than at domain wall edges; it is also much larger than the enhancement factor associated with domain rotation [12]. Therefore the NMR signal from nuclei at domain wall centres, where the moments are in the hexagonal plane, is readily observed with short pulses and/or a low radio-frequency power, whereas long pulses and/or a much larger power are needed to observe the NMR signal from nuclei inside domains or at domain wall edges, where the moments are along the *c*-axis. Moreover, the relaxation at domain wall centres is faster than in domains (or at domain wall edges); therefore a shorter delay time between the pulses and a faster repetition of the pulse sequence will favour the signal from nuclei at the domain wall centre. In order to determine the best conditions for NMR observation of the signal from nuclei at the domain wall (centre) and that from the nuclei in the domain (or at the domain wall edge), we first performed an NMR experiment on a pure Co sample with the hcp structure.

The conditions used to excite the domain and domain wall NMR signals are given in table 1. The NMR spectra recorded for the two regimes of excitation are presented in figure 2. The NMR signal arising from regions where the moment is along the *c*-axis has a frequency of 220 MHz, while it peaks at 225.2 MHz for regions where the moment lies in the hexagonal plane. These NMR frequencies are close to the ones obtained previously by the spin-echo method [7] and are typical of multidomain hcp Co-containing structures. For single-domain, oriented crystals, the in-plane resonance lies at 229 MHz after correction for the demagnetizing field (see [11] for a spherical single crystal or [13] for a thick (001)-oriented Co film). The lower value, spreading between 225 and 228 MHz, which is observed



**Figure 2.** Zero-field  $^{59}\text{Co}$  NMR spectra recorded at 4.2 K from domains and domain walls of hcp Co. The domain NMR signal probes the hyperfine field along the  $c$ -axis and the domain wall NMR probes primarily the hyperfine field in the hexagonal plane.

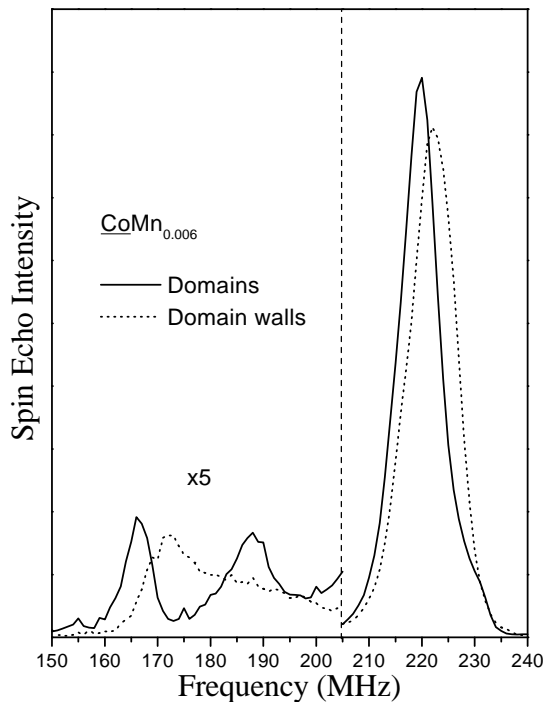
**Table 1.** Excitation conditions used to observed Co NMR from domain wall (centre) (magnetization in the hexagonal plane) and from domain (or domain wall edge) excitation (magnetization along the hcp  $c$ -axis).

	Pulse area (approximate), duration $\times$ RF field (Oe $\mu\text{s}$ )	Delay between the two pulses ( $\mu\text{s}$ )	Recycle time (ms)
Domain wall	0.03	2	0.5
Domain	0.60	100	25.0

for multidomain samples, results from an average over the spread of moment orientations at the domain wall centres. Sometimes a line at 229 MHz is also observed as a weak satellite to the main line at 225 MHz (see, e.g., [14]), but its assignment is not very obvious. Slight differences for the in-plane resonance frequencies could also result from a hyperfine-field anisotropy associated with different moment orientations in the hexagonal plane.

### 3.2. Diluted Co alloys with hcp structure

Figure 3 presents the NMR spectra recorded from a CoMn (0.6 at.%) alloy in the domain and domain wall regimes of excitation, as determined in the previous section. In both cases two satellite lines can be distinguished on the low-frequency side of the main Co peak. Because of the distribution of moment orientations in the wall, the satellites for the DWC spectra are broader and less resolved than in domain NMR. While the observation of two



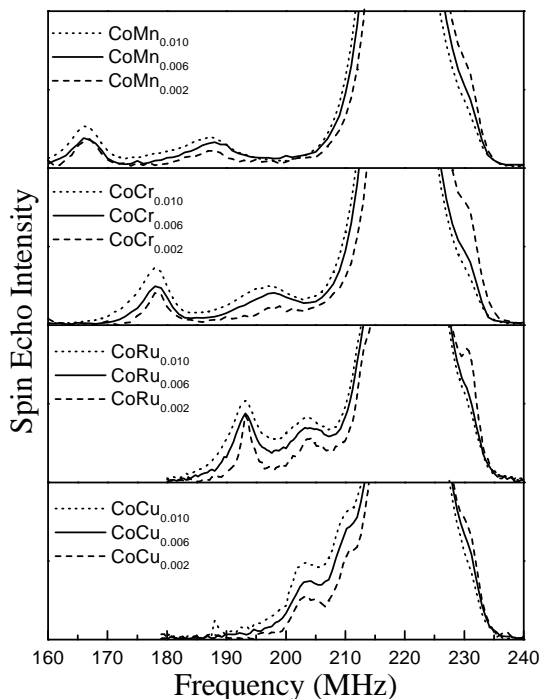
**Figure 3.** Domain and domain wall  $^{59}\text{Co}$  NMR spectra recorded at 4.2 K from  $\text{CoMn}$  alloy with 0.6 at.% Mn.

satellites has already been reported in a domain NMR investigation [7], this is the first report of a clear observation of the domain wall mode. For all of the samples studied, the position of both satellites as well as the position of the main Co peak is shifted in the domain wall spectra by about 5 MHz with respect to the domain NMR, just as in the case of pure Co. This means that the anisotropic contribution to the hyperfine field is about the same for Co atoms with one impurity neighbour, in either position, as for pure Co. Therefore, in the following, we will concentrate on NMR results obtained under conditions of domain signal excitation.

**Table 2.** Shifts of the two satellites to the main NMR line due to the presence of a single impurity in the first-neighbour shell of Co, either in the hexagonal plane or out of the plane (see figure 1). The right-hand column gives the average shift observed for more concentrated hcp alloys, obtained earlier.

Alloy	Satellite 1 shift (MHz)	Satellite 2 shift (MHz)	Average shift (from reference [6]) in concentrated hcp alloys (MHz)
$\text{CoMn}$	-32	-53	
$\text{CoCr}$	-20	-41	-32
$\text{CoRu}$	-16	-27	-25
$\text{CoCu}$	-10	-17	

Figure 4 presents the details of the domain NMR spectrum on the low frequency side of the main Co peak for four series of diluted Co alloys, with impurity concentrations of 0.2,



**Figure 4.** Domain  $^{59}\text{Co}$  NMR spectra recorded at 4.2 K from Co alloys with Mn, Cr, Ru and Cu and impurity concentrations of 0.2, 0.6 and 1.0 at.%.

0.6 and 1 at.%. In addition to the main Co line, two satellites of about equal intensity are clearly visible in each spectrum. The intensity of the satellites increases with the impurity concentration and their positions are constant for each alloy, and characteristic for a given impurity. Similarly to the case of more concentrated alloys [6], the satellite separation varies from one alloy to another. The frequency separation of the two satellites from the main line for all of the alloys studied is given in table 2.

At such a low level of doping, the probability of Co having two alien neighbours in the first coordination zone is below 0.5%, so the impurities may be regarded as completely isolated and both satellites can be reasonably linked to the first-neighbour shell only. In all of the spectra presented in figure 4 the two satellites have the same intensity, indicating that the two types of site are equally populated with impurity atoms. Following Kawakami [7], one satellite would be associated with impurity atoms located in the hexagonal plane of the observed Co nucleus (six sites), and the other with impurities occupying the out-of-plane sites (six sites). The satellite positions in **CoMn** and **CoCr** alloys are in good agreement with Kawakami's previous results. **CoRu** hcp alloys have not been previously studied by means of NMR for such low Ru concentrations and this is the first report of the presence of two satellites for those samples. In the case of **CoCu** alloy we observe, just like for all of the other samples, two resolved satellites on the low-frequency side of the main line, whereas in [7] only one low-frequency satellite was observed at 209 MHz—the frequency corresponding to the closest satellite to the main line. For **CoCu** there was also a satellite reported in [7] above the main line at 224.4 MHz without a clear assignment being given; it is not resolved in our new experiment. In fact, stacking faults in the structure give rise to lines in the same frequency range [6, 14]. Moreover, this frequency is very close to that

of the Co peak arising from the domain walls, which may suggest that this is a residual Co line from the NMR signal for pinned domain walls.

For alloys with larger impurity concentrations the resolution of the two satellites due to the hcp structure is lost. Indeed, with increasing impurity content the two satellites broaden and eventually merge into a single, asymmetric satellite [6]. For CoCr alloys the position of this broad line (table 2) is close to that of the centre of gravity of the twin satellites, but this is not the case for CoRu alloys.

The spectra presented in figure 4 reveal also some structure of unknown origin at about 230 MHz, which is especially pronounced for alloys with low concentration. Since its intensity decreases with increasing alloy concentration, we can exclude the possibility that it is a satellite due to the impurity. It is possible that this line is similar to the in-plane NMR signal at 229 MHz, which is also sometimes observed for pure hcp Co, as discussed in the section above.

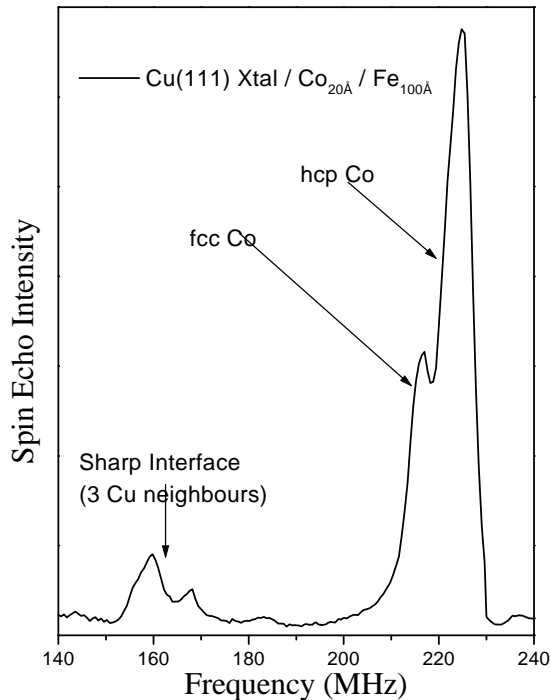
### 3.3. Analysis of the interface region in Co layers with the hcp structure

This anisotropic character of the interaction of the impurity with Co is obviously expected to manifest itself also in hcp-Co-based layered systems. However, in layered systems, the distribution of impurity NN is not isotropic: atomic configurations with impurities out of the plane are favoured, which should allow one to assign the two satellites to the two inequivalent NN positions.

A first attempt has been made for the CoCu system. Figure 5 presents the NMR spectrum recorded from a 20 Å layer of Co grown on a single crystal of Cu and capped with Fe. In the frequency range for bulk Co (215–230 MHz) the NMR spectrum exhibits a main peak at about 225.2 MHz, originating from hcp Co with the magnetization in the hexagonal plane; the smaller peak at 216.4 MHz originates from a minority fcc phase which could not be avoided. These two values are about 1 or 2 MHz lower than the bulk Co ones because of the tensile stress due to the Cu substrate [15]. The low-intensity structures on the low-frequency side of the main line correspond to the interface with Cu, and those on the high-frequency side (partially shown) to the interface with Fe. The resolved peaks at 160 and 168 MHz are fingerprints of a very sharp Co/Cu interface corresponding respectively to the hcp and fcc grains as shown by their relative intensity. The very weak background intensity comes from more diffuse interface regions. Co atoms located at the sharp interface have nine Co NN (six in their own atomic plane and three in the adjacent plane on the side of the Co) and three Cu atoms in the other adjacent plane. As far as the fcc phase is concerned the satellite position at 168 MHz corresponds to a shift of 48 MHz from the main fcc line, i.e. a shift of 16 MHz per Cu NN in perfect agreement with measurements for bulk alloys [6] and all fcc Co/Cu multilayers [16]. The frequency difference between the hcp main line and the sharp interface peak is 65 MHz. This means that each Cu NN in the adjacent plane shifts the NMR frequency of hcp Co by about 22 MHz. Considering the two shifts observed for the hcp alloys (17 and 10 MHz) found in the hcp alloys, one can assign with confidence the more distant satellite for hcp CoCu alloys to Cu neighbours located out of the plane—the closest satellite to the main line would then correspond to Cu neighbours located in the plane.

A similar attempt to make a satellite assignment has been undertaken for the Co/Cr and Co/Ru multilayers. These multilayers exhibit rather diffuse interfaces over about five atomic layers. For Co/Ru multilayers, both components are hcp for any thickness [17]. For Co/Cr multilayers, Co is hcp only for thin Cr layers (less than 10 Å thick) [18]. Such thin Cr layers are alloyed to the core but, unlike the case for homogeneous bulk alloys,



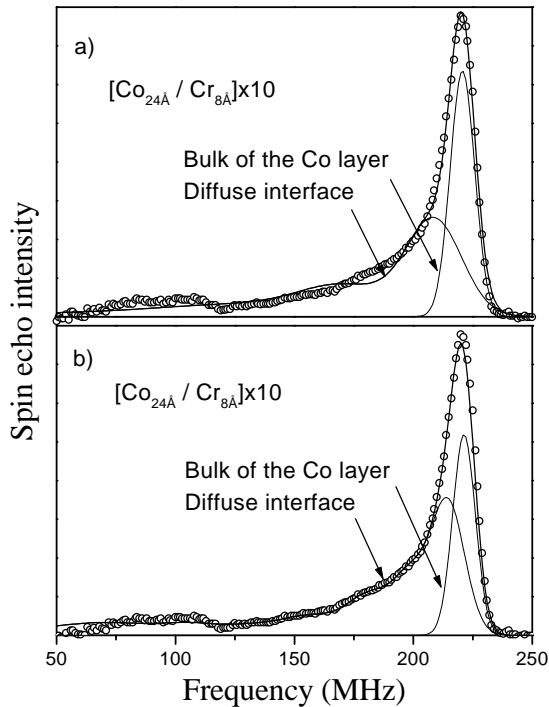


**Figure 5.** The zero-field  $^{59}\text{Co}$  NMR spectrum recorded at 1.6 K for the layered structure ( $(111)$  Cu single crystal/ $\text{Co}_{20\text{\AA}}/\text{Fe}_{100\text{\AA}}$ ). The crystallographic structure of Co is mostly hcp with  $(001)$  growth orientation and magnetization in the hexagonal plane (the line at 225 MHz); a minority fcc phase (the line at 216 MHz) is also observed. The interface lines at 160 and 168 MHz correspond to perfectly sharp interfaces of hcp and fcc Co grains, respectively, with copper.

there is a steep concentration gradient over the five mixed layers. In figure 6 we present the experimental NMR spectrum recorded from the  $\text{Co}_{24\text{\AA}}/\text{Cr}_{8\text{\AA}}$  multilayer and two fits obtained using a model of a diffuse interface with a concentration profile [16]. The fit presented in figure 6(a) has been performed using a satellite spacing of 32 MHz, which is the average value obtained from the analysis of more concentrated alloys. This fit does not reproduce the experimental spectrum in a satisfactory way. In fact, we have shown in [18] that a much better fit can be obtained by letting the satellite spacing be free in the fitting procedure—then this parameter was found to take a rather lower value (20 MHz). In this work we have used the two fixed values of satellite spacing obtained in the section above (i.e. 20 MHz and 41 MHz) and tried to model the spectra with the two possible assignments.

A good fit can only be obtained with the spacing of 20 MHz for the out-of-plane NN and 41 MHz for the in-plane ones (see figure 6(b)). This result is consistent with the free fit in [18]: indeed, most interfacial Co atoms have NN impurities located out of the plane, towards the Cr layer, so the spacing of 20 MHz found using a single spacing in the fit is quite logical.

Similarly, in the case of Co/Ru multilayers, we can only obtain a good fit using the smaller spacing (16.4 MHz) for the out-of-plane NN and the larger one (26.7 MHz) for the in-plane NN. The best fit obtained using a single shift yields a value of 15 MHz instead of the average value of 25 MHz observed for concentrated alloys which, again, corroborates the assignment.



**Figure 6.** The  $^{59}\text{Co}$  NMR spectrum recorded at 1.6 K for a  $[\text{Co}_{24\text{\AA}}/\text{Cr}_{8\text{\AA}}] \times 10$  multilayer and its decomposition, using a model of a diffuse interface with a concentration gradient. (a) The fit assumes a single, average, satellite spacing of 32 MHz as found in [4] for concentrated alloys. (b) The fit is carried out with two values of the satellite spacing: 20 MHz and 41 MHz, as found in this work for dilute alloys.

In this way, studies of multilayers make it possible, in some favourable cases, to assign the twin satellites to each type of site in the first-neighbour shell. Because of the complicated nature of Co/Mn multilayers, we cannot draw any conclusion from their study. However, comparison of the spectra observed for the dilute alloys (figure 4) strongly suggests a similar assignment for CoMn to that for CoCr and CoRu alloys. Indeed, for the three systems, the closer satellite is obviously broader than the distant one. The reverse assignment in the case of Cu impurities is also supported by this comparison: the close satellite, despite the fact that it is superimposed on the tail of the main Co peak, does indeed look narrower than the distant one.

It is puzzling that, whereas for Cr, Ru and, most probably, Mn, impurity neighbours in the plane depress the hyperfine field of Co more than out-of-plane ones, it seems to be the other way around for Cu. Because of the subtle origin of the hyperfine field, one can only speculate as regards the possible reasons for the opposite behaviours of Cu and the other transition metals. The explanation is left to band-structure calculations which, unfortunately, are rather scarce for non-cubic systems. As far as experiment is concerned, there is a possibility that the observation in our Co/Cu structure could lead to an erroneous assignment. Indeed, the thorough EXAFS study by Magnan and co-workers [19], who also succeeded in growing hcp Co on a Cu single crystal, has shown that the two first Co atomic planes may still be stacked in a fcc structure, together with the last Cu plane. For our sample we can distinguish the satellites corresponding to the interface between the Cu and

fcc Co grains or the Cu and hcp Co grains, but we cannot prove that the Co planes are stacked in a hcp structure at the interface between the Cu and the hcp Co grains.

#### 4. Conclusions

The present study is a refinement of our previous work concerning the influence of alien atoms on the hyperfine field of Co, carried out to the limit of dilute hcp alloys and two-dimensional hcp structures. It confirms and extends the previous report by Kawakami of an anisotropic contribution of the first impurity neighbours to the hyperfine field of hcp Co. A pair of low-frequency satellites have been observed for all of the alloys studied. The satellite frequency shift for **CoCr** and **CoMn** alloys is in agreement with the ones observed by Kawakami. In the case of **CoCu**, a satellite additional to the one already reported has been observed. The twin satellites for **CoRu** alloys have not been reported before. The study of two-dimensional alloys present at the interfaces of the corresponding multilayers show that the impurity atoms located in the same hexagonal plane as the probed Co nucleus give rise to the more distant satellite for **CoCr** and **CoRu** alloys: these in-plane neighbours depress the Co hyperfine field more than out-of-plane impurities. Comparison with the results obtained for the alloys **CoCr** and **CoRu** suggests a similar conclusion for **CoMn** alloys. As to **CoCu** systems, the opposite conclusion is reached, although there is some ambiguity in the analysis of Co/Cu multilayers because the hcp Co layer might have stacking faults in the vicinity of the interface with Cu.

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