NMR study of the strain in Co-based multilayers

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The strain in Co layers of multilayers with largely different structural mismatch (Co/Ni, Co/Ag, and Co/Cu) has been measured with nuclear magnetic resonance and compared with a model for strain in multilayers that is based on the model of Van der Merwe and Jesser. For the Co/Ni multilayers, the measured strain depends on both the Co and the Ni layer thicknesses over a large range and follows the behavior expected from the model in the coherent regime. The strain in the Co/Ag multilayers is proportional to $1/t_{\mathrm{Co}}$ and independent of the thickness of the Ag layers. This behavior resembles that for the incoherent regime. For Co/Cu multilayers the results indicate that the multilayers are in the transition region from coherent to incoherent behavior.

I. INTRODUCTION

Magnetic multilayers have been studied intensively in recent years because of their interesting magnetic properties like perpendicular anisotropy, exchange coupling, and giant magnetoresistance. The artificial nature of these systems offers the possibility to tailor these properties by choosing the appropriate structure and composition. Apart from this it has been found, or at least predicted, that the aforementioned properties can depend also on microstructure, interface topology (roughness), and strain.[1,2] Since interfaces and layers are buried in the multilayer structure, information on these structural data is generally hard to obtain. In earlier publications, it has been documented that in this respect NMR may be a very useful tool.[3,4]

In the present paper, we will focus on strain in multilayers. Because the hyperfine field, which is measured with NMR, depends on the atomic volume,[5] a change of the lattice parameters will result in a change of the hyperfine field. This enables the determination of the strain inside multilayers.[3,4]

We will present data on the strain in two limiting cases, with small (Co/Ni, $\eta = 0.6\%$) and large (Co/Ag, $\eta = -14\%$) mismatch between the adjacent layers, respectively, and compare the results with a strain model that is based on the model of Van der Merwe and Jesser.[6] Moreover, we will compare the results with earlier reports on Co/Cu multilayers ($\eta = -2\%$).

II. STRAIN IN MULTILAYERS, THEORETICAL MODEL

In multilayered samples strain is a common feature and can be caused by the mismatch between the substrate or base layer and the actual multilayer, and by the mismatch at the interfaces. In this paper, we will only regard strain caused by the mismatch at the interfaces, because the contribution of the mismatch between the substrate and the first layer of the multilayers is expected to be much smaller.[7] The mismatch η at the interfaces of a Co/x multilayer is defined by

$$\eta = \frac{a_{\mathrm{Co}} - a_x}{\frac{1}{2}(a_{\mathrm{Co}} + a_x)} , \tag{1}$$

where a denotes the lattice constant. This mismatch may be relaxed by a homogeneous strain in the layers or through the introduction of interfacial dislocations. In the first case, one layer is compressed and the other is stretched in such a way that both layers have the same in plane lattice parameter. In this case, the layers are called coherent. In the second case, the lattice registry of the layers is lost and the layers are called incoherent. Because the strain energy is proportional to the volume and the dislocation energy is proportional to the area, a critical thickness (t_c) will exist below which coherent behavior is expected and above which incoherent behavior is expected. The critical thickness and the strain in multilayers can be obtained from a minimization of the total energy, which contains elastic and dislocation energies.

To obtain the strain we extended the calculations of Van der Merwe and Jesser,[6] based on a model which was originally developed to calculate the critical thickness in multilayers. The model uses a succession of parabolic arcs to describe the periodic interaction potential between atoms across the interface. It is assumed that the interfaces are perfectly flat and that the elastic properties of the materials are isotropic. The strain and the critical thicknesses depend on the relative magnitude of the elastic constants, the thicknesses of the layers, and the mismatch. More details about the model can be found in the original papers.[6]

In Fig. 1(a) the strain in the Co layer, calculated in the framework of the model of Van der Merwe and Jesser, is shown as a function of the layer thickness of the other element (t_x) at a constant Co layer thickness. If t_x increases from zero the strain in the Co layers in-

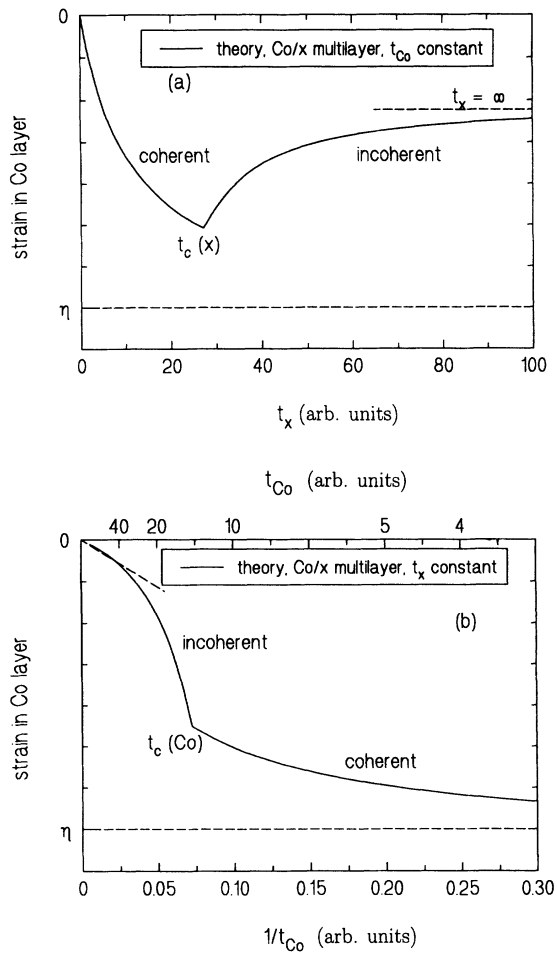

FIG. 1. Typical behavior of the strain in the Co layers of Co/x multilayers ($a_{Co} < a_x$) calculated using the model of Van der Merwe and Jesser. In (a) the strain is shown for multilayers with a constant Co layer thickness and a variable thickness of element x, in (b) t_{Co} was variable and t_x was constant. The exact values of the strain and the critical thickness of the Co layers of a specific multilayer composition depend on the elastic constants, the mismatch, and the thickness of the other layer. For further explanation see the text.

creases monotonously (coherent regime) until, at the critical thickness of element $x(t_c(x))$, dislocations are formed at the interfaces and the strain is partially relaxed by dislocations. If t_x is further increased, the strain decreases because of the increasing number of dislocations (incoherent regime). If t_x is much larger than the critical thickness, the strain in the Co layer is almost independent of t_x. The magnitude of the strain in this regime ($t_x \to \infty$) depends on the thickness of the Co layer: the thinner the Co layer the larger the strain. Figure 1(b) shows the dependence of the strain in the Co layer on the thickness of the Co layer (t_{Co}) if t_x is constant. For very thin Co layers the strain in the Co layers is equal to the lattice mismatch. For increasing t_{Co} the strain in the Co layer decreases because now the other layer is also strained. If the Co layer becomes thicker than the critical Co thickness (t_c (Co)) the strain is further decreased and with a steeper slope because now part of the mismatch is accommodated by dislocations. Of course, for very thick Co layers, the strain is zero. In the range $t_{Co} \gg t_c$ (Co), the dependence of the strain (ϵ) on the Co thickness is usually approximated by $\epsilon \sim 1/t_{Co}$,[1] which is shown in Fig. 1(b) by a straight line.

Now we will consider in more detail the two series of multilayers that were investigated with NMR. The Co/Ni multilayers have a very small mismatch (0.6%), and hence the critical thicknesses are large [t_c (Co,Ni) > 100 Å] and the layers are expected to be coherent. For this situation the total mismatch is accommodated by elastic strain in the layers. For a fcc [111] multilayer the relation between the in plane Co strain (ϵ_{Co}), the thicknesses, and the elastic constants can be approximated by (coherent regime Fig. 1):

$$\epsilon_{Co} = \frac{-\eta}{1 + q\frac{t_{Co}}{t_{Ni}}}, \tag{2}$$

with q the ratio between the shear moduli of Co and Ni, $q = G_{Co}/G_{Ni}$.

For Co/Ag the mismatch is much larger (−14%). Consequently, the critical thicknesses are very small [t_c (Co,Ag) < 6 Å] and the multilayers are expected to be incoherent. In Fig. 1 it can be seen that for thicknesses much larger than the critical thickness the strain in the Co layer is expected to be proportional to $1/t_{Co}$ and independent of the thickness of the Ag layer.

III. EXPERIMENT

The [111] fcc Co/Ni multilayers were prepared at the Philips Research Laboratories with molecular beam epitaxy (MBE). The multilayers were deposited at room temperature on oxidized silicon substrates with a base layer of 300 Å Au. The deposition rate was between 0.5 Å/s and 1 Å/s. X-ray diffractometry in the θ-2θ scan mode showed [111] texture and confirmed the superlattice modulations. The Co/Ag multilayers were made by magnetron sputtering on Si [100] at the Michigan State University. The deposition rate was 4 Å/s for Co and 8 Å/s for Ag. X-ray diffractometry confirmed the superlattice modulations and showed [111] texture.[8]

The NMR experiments were performed partially with an incoherent and partially with a coherent spin echo spectrometer at a temperature of 1.4 K. Magnetic fields, an order of magnitude larger than the saturation field, were applied parallel to the film plane in the easy direction of the magnetization. The hyperfine field B_{hf} was obtained from the resonance field B_r and the frequency f using the relation $2\pi f = \gamma(B_{hf} - B_r)$, where γ is the ^{59}Co nuclear gyromagnetic ratio ($\gamma/2\pi = 10.054$ MHz/T).

The possibility to study the strain by NMR arises from the dependence of the hyperfine field on the atomic distances. For Co the following relation between the relative change in hyperfine field and the relative change of the atomic volume has been reported (at room temperature and under isotropic pressure[5]):

$$\frac{\Delta B_{\rm hf}}{B_{\rm hf}} = -1.12\frac{\Delta V}{V} \ . \tag{3}$$

For isotropic volume changes this $\Delta V/V$ equals 3ϵ. However, for multilayers the volume changes are generally not isotropic because the in plane lattice contractions are often accompanied by perpendicular lattice expansions. This tetragonal distortion has been shown by low energy electron diffraction experiments on Co and Ni deposited on a Cu single crystalline substrate.[9] The change in volume due to a certain in plane strain can be estimated using Poisson's ratio σ. If the strain in the interface plane equals ϵ the strain perpendicular to this plane will be $(2\epsilon\sigma)/(\sigma - 1)$. Now the relation between the total change in volume and the strain will be

$$\frac{\Delta V}{V} = 2\epsilon\frac{1-2\sigma}{1-\sigma} \ . \tag{4}$$

In the case of coherent multilayers with tetragonal distortion, one can combine Eqs. (2), (3), and (4) to get the dependence of the hyperfine field on the thicknesses of the layers (using $\sigma_{\rm Co} = 0.31$)

$$\frac{\Delta B_{\rm hf}}{B_{\rm hf}} = 1.2 \times \frac{\eta}{1 + q\frac{t_{\rm Co}}{t_{\rm x}}} \ . \tag{5}$$

IV. RESULTS

A. Co/Ni multilayers

A typical example of the NMR spectra of the Co/Ni multilayers is presented in Fig. 2. The figure shows the spin echo intensity as a function of $B_{\rm hf}$ for a 25 × (12 Å Co + 60 Å Ni) multilayer. The main line in the spectrum (arising from Co atoms in the middle of the Co layers) is close to the value of bulk fcc Co (21.54 T, measured for a 1000 Å thick Co layer). The difference between the bulk value and the measured position is caused by strain in the Co layers. Because there is practically no intensity at the high field side of the main line, the amount of hcp Co and stacking faults must be very small.[3] The shoulder on the low-field side of the main line probably arises from Co nuclei with one or more Ni atoms in the next nearest-neighbor shell and no Ni atoms in the nearest-neighbor shell. The intensity for $B_{\rm hf} < 21.5$ T arises from Co atoms at the interfaces.[3]

In Fig. 3 the position of the main line is plotted as a function of the ratio of the Ni and Co layer thickness ($t_{\rm Ni}/t_{\rm Co}$). The experimental results for the (X Co + 42 Å Ni) multilayers are represented by circles, the data on the (12 Å Co + X Ni) series are denoted by squares. The bulk value $B_{\rm hf} = 21.54$ T measured for a 1000 Å thick Co film on the same substrate is also denoted. The figure shows that the strain in the Co layers is compressive (if the Co layer thickness is decreased or if the Ni layer thickness is increased the main line shifts to higher fields) and depends strongly on both the Co and the Ni layer thickness. It also appears that the shift of the hyperfine field of the two series is approximately the same for a specific thickness ratio, as is expected for coherent multilayers [see Eq. (5)]. The maximum shift of the hyperfine field is 0.17 T for the sample (10 Å Co + 100 Å Ni). Assuming tetragonal distortion of the Co lattice, this shift corresponds, according to Eqs. (3) and (4), to a $\Delta V/V$ of about -0.7% and an in plane Co strain of approximately -0.6%. It has to be noted that this experimental number agrees very well with the lattice mismatch between Co and Ni, showing that in this limit the thin Co layers adopt the Ni spacing, as is expected in the coherent regime.

The dashed line in Fig. 3 is a fit of the data of the 2 Co/Ni series with Eq. (5), which is expected to hold in the coherent regime. This fit results in $\eta = (1.0 \pm 0.1)\%$ and $q = 1.9 \pm 0.6$. Although both values are larger than the values reported in the literature: $\eta = 0.6\%$ and $q = 0.96$,[10] the overall agreement is rather good, cor-

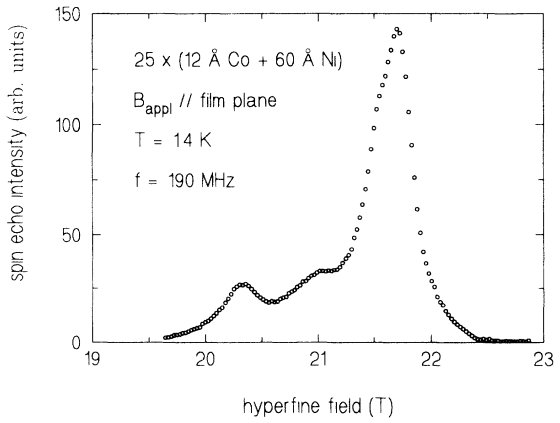

FIG. 2. NMR spectrum of a 25 × (12 Å Co + 60 Å Ni) multilayer. The spectrum was recorded at 190 MHz with the field applied parallel to the film plane and at a temperature of 1.4 K. The spectrum is corrected for receiving enhancement. The intensity for $B_{\rm hf} < 21.5$ T arises from Co atoms at the interfaces.

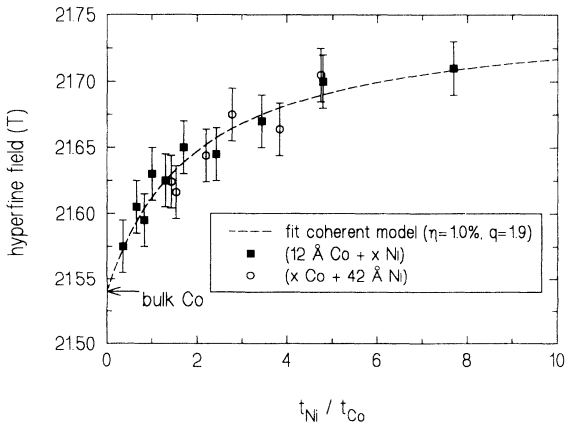

FIG. 3. The hyperfine field as a function of the ratio of the Ni and Co layer thickness for the two series of Co/Ni multilayers. The data of the series (x Co + 42 Å Ni) are from Ref. 3. At 21.54 T the reference bulk Co hyperfine field is denoted. The fit (dashed curve) is based on the coherent model.

roborating the indication that the Co/Ni multilayers are coherent. The deviations of η and q may be caused by the assumption that was made by using Eq. (3), which is only valid for isotropic volume changes. Another explanation for the deviations is the fact that the literature values for η and q, and the constant in Eq. (5) are bulk values at room temperature, whereas the measurements were performed on multilayers at 1.4 K.

B. Co/Ag multilayers

In Fig. 4 a typical example of the NMR spectra of the Co/Ag multilayers is given. The intensity for $B_{\rm hf} < 19$ T arises from Co at the interfaces. The main line (around $B_{\rm hf} = 21.2$ T) is much broader ($\sim$ 3 times) than the main line of the Co/Ni multilayers and is a mixture of fcc Co, hcp Co, and stacking faults.[8] In such a situation, a shift of the main line can, in principle, be caused by two effects: strain and a change in the relative amounts of the different structures of Co. Because the shape of the main line of all Co/Ag spectra was the same, except for the sample (100 Å Co + 20 Å Ag), the relative amount of fcc Co, hcp Co, and stacking faults must be constant for the samples.

Figure 5 shows the variations of the average hyperfine field of the main line for Co/Ag multilayers with different Co as well as varying Ag thicknesses. Unlike the Co/Ni multilayers, shown above, for Co/Ag the strain cannot be uniquely described by the ratio of the thicknesses: $t_{\rm Ag}/t_{\rm Co}$. Instead it is observed that the series with varying Co layer thickness behaves completely different from the series with varying Ag layer thickness. For the first series, the shift of the hyperfine field is proportional to $1/t_{\rm Co}$, whereas in the latter series the variation of the Ag layer thickness does not alter the position of the main line and, thus, the strain appears to be independent of $t_{\rm Ag}$. This behavior is exactly what would be expected for a completely incoherent system.

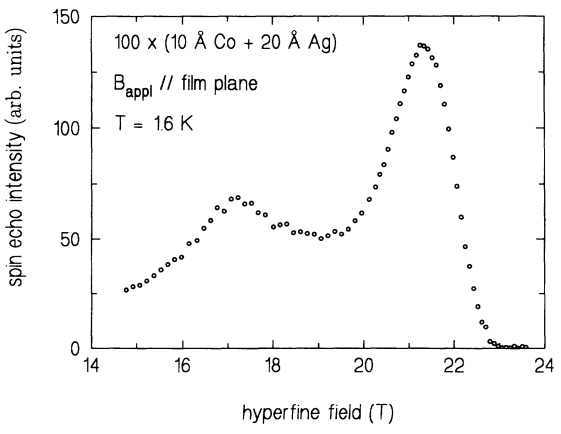

FIG. 4. NMR spectrum of a 100 × (10 Å Co + 20 Å Ag) multilayer. The spectrum was measured using several field sweeps at different frequencies. The magnetic field was applied parallel to the film plane, the temperature was 1.6 K. The spectrum is corrected for enhancement. The line at about 17 T arises from Co atoms at the interfaces.

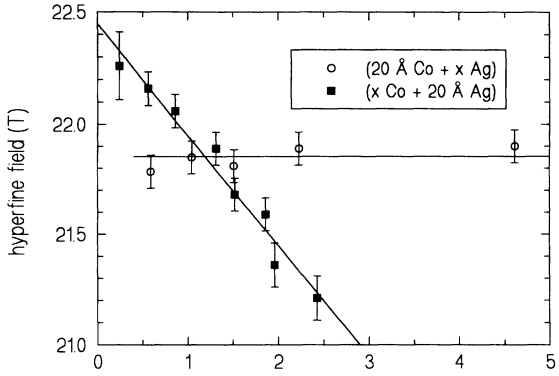

FIG. 5. The hyperfine field as a function of the ratio of the Ag and Co layer thickness for the two series of Co/Ag multilayers. The solid lines are guides to the eye.

Figure 5 also shows that, in contrast to Co/Ni, $B_{\rm hf}$ shifts to lower fields with decreasing Co thickness, showing that the strain in the Co layers is tensile in the Co/Ag multilayer system, in accordance with the sign of the mismatch.

The magnitude of the shift of the hyperfine field is much larger than for the Co/Ni multilayers. The sample with (10 Å Co + 20 Å Ag) is shifted over −1.2 T with respect to the extrapolated "bulk" hyperfine field. This shift corresponds [see Eq. (3)] to a Co volume change of about 5%. Assuming isotropic volume changes, the in plane Co strain would be about 2%. If the Co lattice is tetragonally deformed the in plane Co strain would be about 4% [see Eq. (4)].

C. Co/Cu multilayers

Figure 6 shows earlier data[11] on the variation of the hyperfine field of two series of Co/Cu multilayers, (x Å Co + 21 Å Cu) and (x Å Co + 42 Å Cu), as a function of $1/t_{\rm Co}$. This figure shows that the shifts of the hyper-

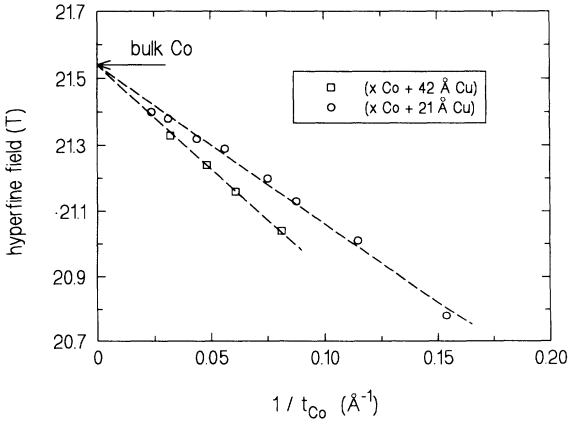

FIG. 6. The hyperfine field as a function of $1/t_{\rm Co}$ for two series of Co/Cu multilayers (data from Ref. 11). The dashed lines represent least squares fits of a straight line to the data.

fine field are proportional to $1/t_{Co}$, which might indicate that the Co/Cu multilayers are incoherent. However, the fact that the shift of the series with $t_{Cu} = 42$ Å is larger than for $t_{Cu} = 21$ Å suggests [see Fig. 1(a)] that either the layers are coherent or that the critical thickness of the Cu layer is between 21 and 42 Å. A fit of the data on the Co/Cu multilayers with the coherent model results in values of q larger than 100, which is completely unphysical.

One might therefore conclude that the Co/Cu multilayers with a mismatch of $\eta = -2\%$ cannot be considered as a limiting case where the thicknesses of the layers are much smaller (like Co/Ni) or much larger (Co/Ag) then the critical thicknesses.

V. DISCUSSION

Before the results of the measurements of the strain will be discussed, we would like to remark that, in principle, a shift of the hyperfine field as a function of the layer thickness might also be the result of changes in the magnetization of the sample. However, because the temperature during the measurements was much lower than the Curie temperature (2 K compared to a Curie temperature of about 1390 K) changes of the magnetization are not very likely. The fact that the shifts are to higher hyperfine fields for Co/Ni and to lower hyperfine fields for Co/Cu (if t_{Co} decreases), and the fact that the shift of the Co/Cu multilayers depends on the thickness of the Cu layer, demonstrates that a reduction of the saturation for thinner Co layers is very likely a minor effect, which, however, cannot be fully excluded.

The qualitative behavior of the strain in the Co/Ag multilayers resembles that expected for the incoherent regime of the model. The magnitude of the strain in the Co/Ag multilayers is comparable to that reported for other related large mismatch systems such as Co/Pt,[12] Co/Au,[13] and Ni/Ag.[14] In this respect the initial results of an extended x-ray absorption fine structure study on sputtered Co/Ag multilayers by Foiles *et al.*[15] are puzzling. Their measurements indicate that for a series of (x Å Co + 35 Å Ag) the Co lattice parameter is equal to that of bulk Co and does not depend on the Co thickness. Reflection high energy electron diffraction measurements of Kingetsu and Sakai[16] show that their MBE grown Co/Ag multilayers are completely incoherent and that the layers are nearly strain free. On the contrary x-ray diffraction measurements on MBE grown Co/Ag multilayers by Araki[17] show that the lattice constant of Co is about 2% larger than in bulk Co. These differences in experimental results on the same Co/Ag multilayer system may be caused by differences in roughness. Rough interfaces may lock the two structures and prevent an abrupt complete incoherent behavior at the interfaces.

X-ray scattering measurements with the scattering vector lying in the film plane by Lee *et al.*[13] on Co/Cu multilayers with a Cu layer thickness of 25 Å and a Co layer thickness between 5 and 40 Å showed that the Co/Cu multilayers were coherent for all Co layer thicknesses. The strain of the Co layer varied between about 0.8% for the sample with 40 Å Co up to almost 2% for the thinnest Co layers. The maximum shift of the hyperfine field of the series with $t_{Cu} = 21$ Å that we measured was 0.75 T. This corresponds to a volume change of about 3%. If we assume tetragonal deformation and use the bulk Poisson constant, the in plane Co strain would be about 2.8%, which is larger than the lattice mismatch and, thus, not very probable. In a recent study on the structure of UHV evaporated Co/Cu multilayers by Pizzini *et al.*,[18] it appeared that the in plane lattice constant of Co was expanded, but that in the direction perpendicular to the Co/Cu interfaces the Co nearest-neighbor distance was close to the bulk value. In this case, the volume change equals 2ϵ and the NMR measurements indicate an in plane Co strain of 1.8% for the sample (6 Å Co + 21 Å Cu). This value agrees very well with the results from Lee *et al.*[13]

Two comments can be made about the information NMR can provide about the strain in multilayers. In the analysis of the strain we have used the shift of the bulk NMR signal (nuclei which are surrounded by 12 Co nearest-neighbor atoms). In general, the NMR spectra also contain contributions from nuclei situated at or near

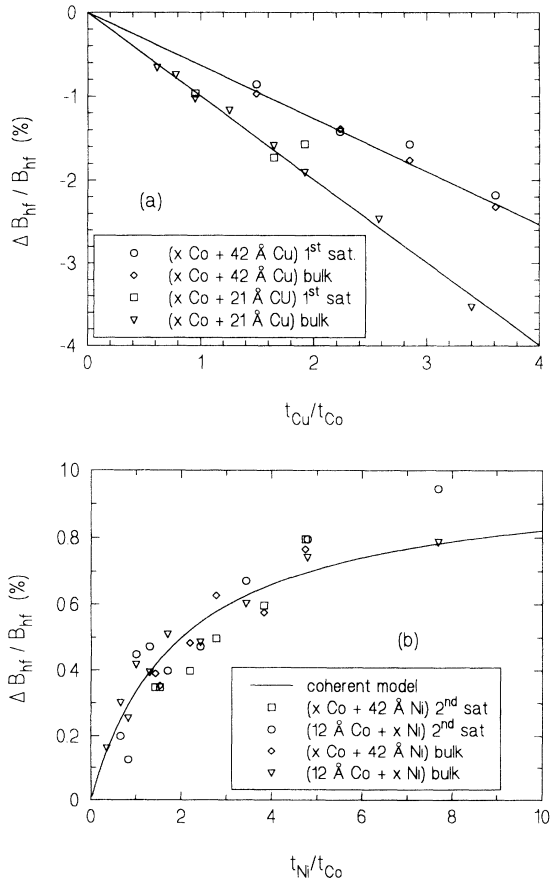

FIG. 7. Comparison of the relative shift of the hyperfine field of bulk Co and Co at the interfaces for Co/Cu (a) and Co/Ni (b) multilayers. ΔB_{hf} is the difference between the measured B_{hf} and the reference B_{hf}. The bulk reference was 21.54 T, the reference of the interface signal was 19.69 T for Co/Cu and 20.11 T for Co/Ni.

the interface (see Figs. 2 and 4). The hyperfine field of these nuclei is decreased because of the reduction of the number of nearest-neighbor Co atoms. Apart from the fact that these spectra contain information on the interface topology,[11] they also monitor the *local* strain. In Fig. 7 we have plotted the thickness dependence of the relative shift in the position of these interface signals together with those from the bulk for the Co/Cu [Fig. 7(a)] and Co/Ni [Fig. 7(b)] multilayers. For the Co/Ag multilayers a comparison of the strain in the bulk and at the interface is less reliable because the lines were rather broad, due to the mixture of phases of the Co in Co/Ag multilayers, and because the position of the satellite line could only be determined for small Co thicknesses ($t_{Co} < 20$ Å). Figure 7 clearly shows that for Co/Cu as well as Co/Ni the thickness dependence of B_{hf} is very similar for the interface and bulk nuclei. If one assumes that the phenomenological relation Eq. (3) also holds for defect surroundings, such as encountered at the interface, the present result indicates that the strain in Co/Ni and Co/Cu is uniform. This observation supports the *a priori* implementation of uniform strain in theoretical models.

Another source of information of the strain in the layers is the linewidth of the bulk NMR lines. For uniform strain the linewidth of the bulk NMR line should be independent of the Co thickness. When a gradient in the strain would exist a broadening of the line would be expected, since the strain induced shift is of the same order as the linewidth. This broadening will become more dominating for decreasing Co layer thickness. For Co/Cu and Co/Ag a strain gradient would result in a tail of the main line at the low-field side, whereas for Co/Ni a tail is expected at the high-field side. If a strain gradient is present, the strain of the interface atoms will always be larger than the strain of the bulk atoms. For the Co/Ni and the Co/Cu multilayers this behavior was clearly not observed. For Co/Ag it is not possible to exclude this kind of strain.

We would like to conclude with some comments on the model of Van der Merwe and Jesser that we used. In the model it is, among other things, assumed that the interfaces are perfectly smooth and that the average of the bulk values of the elastic constants (shear modulus and Poisson's ratio) may be used at the interfaces. Both assumptions do very likely not hold for real multilayers. To make more reliable calculations of the strain and the critical thickness more knowledge is required about the influence of roughness, about the elastic constants at the interfaces and about the atomic bonding energies between the two elements across the interface.[16]

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