Interface magnetism in Permalloy/Cu multilayers: Ferromagnetic-resonance study

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Ferromagnetic resonance has been applied to study sputtered $[\text{Permalloy/Cu}]_{100}$ multilayers with Permalloy (Py) and Cu layers thicknesses 5 Å$<d_{\text{Py}}<$ 40 Å and 8 Å$<d_{\text{Cu}}<$ 40 Å, respectively. The effective magnetization is analyzed in the vicinity of nominal critical thicknesses of Py layers, $2\Delta d$ = 4.9, 7.6, and 8.3 Å, at 77, 293, and 400 K, respectively. From its behavior and the temperature measurements of ferromagnetic resonance (FMR) spectra we argue about the magnetic structure of a statistically averaged interface between Py and Cu: At room temperature, about 0.5 monolayer (ML) (1 ML = 1.8 Å) may be magnetically inactive due to spin wave excitations in ultrathin films and about 1.5–2 monolayers—due to intermixing resulting from roughness. Trapezoidal-like magnetization profiles in ultrathin Py layers 4–7 monolayers thick are discussed in terms of percolation of magnetic clusters within a rough interface. The FMR linewidth depends on the thickness of Py layer as d_{Py}^{-2}. [S0163-1829(98)00510-4]

I. INTRODUCTION

Magnetic films, and multilayers in particular, reveal a broad range of magnetic properties which depend both on thickness and the growth conditions.[1] In connection with the interest in the giant magnetoresistance (GMR) and efforts directed to its optimization, much of the recent work has been aimed at understanding the role of interfaces with nonmagnetic spacers: their roughness and intermixing.[2] The effects related to the presence of interfaces are very subtle and the corresponding microstructural changes are complex and dependent on the preparation details.[3]

Since in multilayers interface effects show a tendency to accumulate with the increasing number of repetitions, static magnetization measurements are often used to get statistically averaged information on them provided that an appropriate analysis of the dependence of magnetic properties on magnetic layer thickness is performed. In particular, from an intercept of the magnetic moment $\mathcal{M}_s$ vs d_{Py}, a quality of interface and a degree of interdiffusion have been deduced.[4–9]

In this work we have extensively applied the ferromagnetic resonance (FMR) technique to elucidate the magnetic behavior of the Permalloy (Py = $Ni_{83}Fe_{17}$) layers stacked in Py/Cu multilayers which have been frequently used for constructing magnetic structures with a fairly large GMR effect.[9–11] The ferromagnetic resonance method has already proved useful for providing information on magnetic anisotropies even in the monolayer thickness range,[12] for studying the critical behavior at the Curie temperature,[13] and for determining the magnetic moment of ultrathin magnetic films.[14] Stress is put on a thickness dependence of the effective magnetization $4\pi M_{\text{eff}}$ in the thickness range 4 Å $<d_{\text{Py}}<$ 15 Å where the FMR response weakens and eventually ceases at a critical thickness. In this range of Py thickness, the magnetic properties are substantially influenced by the presence of interfaces.

Our measurements were performed on a large number of samples in which the thickness of an individual layer was determined with the highest possible accuracy, which enabled us to have better confidence and a better data statistics. The FMR data are discussed in the framework of a realistic model which focuses on a relation between microstructural characteristics of interfaces and thermal fluctuations of discontinuous magnetic structures below a percolation threshold or due to the spin wave excitations in ultrathin magnetic layers.

II. EXPERIMENTAL PROCEDURE

The samples were produced by a double face-to-face sputtering method[11] on Si (100) substrates with a 200-Å-thick Cu buffer. Substrates were wet-chemically etched with hydrofluoric acid in order to promote epitaxial growth of a Cu(100) seed layer. Several series of $[\text{Py/Cu}]_{100}$ (i.e., consisting of 100 repetitions) multilayers were prepared with d_{Cu} and d_{Py} covering a broad range (Fig. 1, inset) of individual Py and Cu layer thicknesses. The thicknesses were later controlled by analysis of low-diffraction scans and an x-ray fluorescence method.[15] The results obtained were in good (better than 95%) agreement with each other. The clear observation of peaks associated with the superperiodicity at large and small angles indicated a good structural quality of our samples which shows a dominating (100) texture.[11,16] GMR measurements on the same set of samples revealed the presence of two ranges with a relatively weak antiparallel coupling centered at ~10 and 21 Å, respectively.[16] The FMR data were taken at the X band (9.4 and 9.08 GHz) with the external magnetic field parallel and perpendicular to the surface of the films. Magnetic field modulation was employed so that the detected signal was proportional to the field derivative of the absorbed power in a TE102 rectangular cavity. Most data were taken at 77 K, 293 K, and 400 K. For some of the samples the temperature dependence of the resonance spectra was measured from 77 K up to 800 K in a quartz Dewar with flowing nitrogen gas.

To determine the effective magnetization

$$4\pi M_{\text{eff}}=4\pi M_s-2K_U/M_s, \tag{1}$$

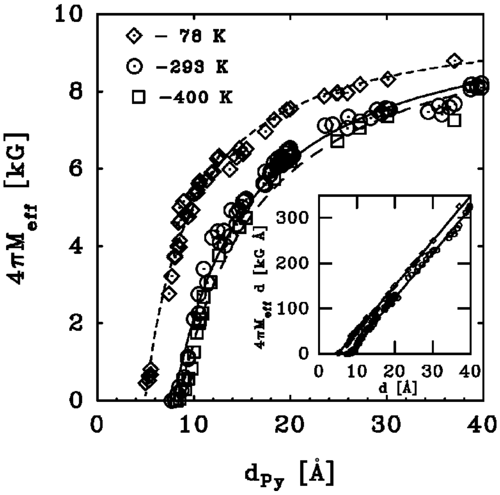

FIG. 1. Dependence of the effective magnetization on the Py layer thickness for 77, 293, and 400 K. Lines are least squares fits to the data points according to Eq. (5).

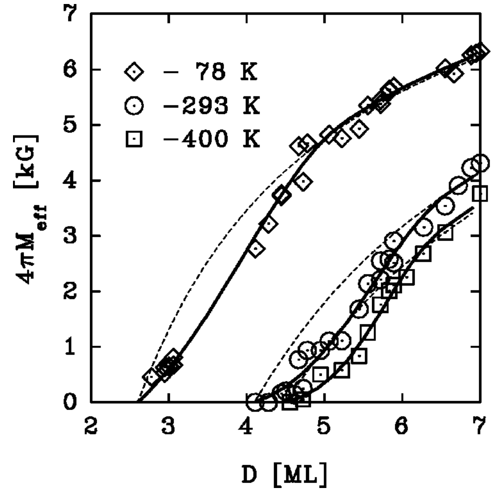

FIG. 2. Dependence of the effective magnetization on the Py layer thickness (in ML) for 77, 293, and 400 K in the vicinity of the critical thickness. Dashed lines are plots of Eq. (5) with $4\pi M_{\rm eff}(\infty)=10$ kG and $2\Delta D=2.6$, 4.2, and 4.6 ML. Solid lines are fits according to Eq. (5) with $4\pi M(D)$ described by Eqs. (8) and (9).

where $M_{\rm eff}=M_{\rm eff}(d_{\rm Py})$, we use the resonance conditions for a thin film[17] for a magnetic field (H_P) applied perpendicular to the film plane,

$$\left(\frac{\omega}{\gamma}\right)=H_P-4\pi M_{\rm eff}, \tag{2}$$

and for the magnetic field (H_R) in the film plane,

$$\left(\frac{\omega}{\gamma}\right)^2=H_R(H_R+4\pi M_{\rm eff}), \tag{3}$$

where $\omega/2\pi$ is the microwave frequency, $4\pi M_s$ is the saturation magnetization, and K_U is the uniaxial anisotropy which may contain surface and volume terms.[18] To compare $4\pi M_s$ with $4\pi M_{\rm eff}$, we measured FMR absorption intensities at 293 K for several samples. An Fe-doped MgO crystal, placed into the cavity, served as a reference for absolute calibration of the FMR intensity I, which is proportional to the total magnetic moment $\mathcal{M}_s$ of the specimen,[14]

$$I\propto\mathcal{M}_s\frac{H+4\pi M_{\rm eff}}{2H+4\pi M_{\rm eff}}. \tag{4}$$

The absolute FMR intensity was determined by numerical integration of the field derivative of absorption.

III. RESULTS

Figure 1 shows the dependence of $4\pi M_{\rm eff}$ on $d_{\rm Py}$ at 77, 293, and 400 K. Almost no dependence on $d_{\rm Cu}$ was detected except a very narrow range below $\sim$9 Å where pinholes through Cu layers enhance the effective magnetization, but this effect is not substantial for the present discussion. The curves which fit the experimental data points correspond to a frequently used formula

$$4\pi M_{\rm eff}=4\pi M_{\rm eff}(\infty)\left(1-\frac{2\,\Delta d}{d_{\rm Py}}\right), \tag{5}$$

which expresses an effect of the reduced magnetization on the two interfaces between Cu and Py. The inset shows linearized plots of Eq. (5) for 77 and 293 K. The slopes of the plots give a bulk value of $4\pi M_{\rm eff}(\infty)$ equal to 10 ± 0.05 kG for each temperature. $4\pi M_s$ determined with a vibrating-sample magnetometer (VSM) for 15 Å$\leqslant d_{\rm Py}\leqslant$ 40 Å gives nearly the same value of 10.8 ± 0.8 kG. The offset $2\Delta d$ from the origin, attributed to nonmagnetic layers at the Py/Cu interfaces, varies from 4.9 Å to 7.6 Å and 8.3 Å for 77, 293, and 400 K, respectively. Assuming that 1 monolayer (ML) of Permalloy with (100) texture is 1.8 Å thick, $\Delta D^{77}=1.3$ ML, $\Delta D^{293}=2.1$ ML, and $\Delta D^{400}=2.3$ ML for a single average interface.

The behavior of the effective magnetization in a region where ferromagnetism sets on may provide some additional information on the morphology of the average Py layer in the Py/Cu multilayers. The important point is that $M_{\rm eff}$ (or M_s) is proportional to a derivative of the magnetic moment with respect to the thickness, and therefore it is more sensitive to small departures from the normal behavior expressed by Eq. (5) (see Fig. 1, inset). In Fig. 2 we plot $4\pi M_{\rm eff}$ vs D (the nominal thickness in ML) in the vicinity of the critical thickness at each temperature of measurements. The departures from the dependences predicted by Eq. (5) (dashed lines) is clearly seen in the thickness range from 3 to 5 ML for data taken at 77 K and extends to about 6 ML for 293 and 400 K.

All these features of the magnetization behavior for $D\leqslant6$ ML will be tempting to relate in the discussion to a transition from continuous layers to clusters or spin blocks when D decreases. It is, however, also possible that such an anomaly may be induced by the interplay of the uniaxial anisotropy K_U (of a surface origin, in this case) and the shape anisotropy, yielding a thickness range in which the two contributions [see Eq. (1)] balance each other. To confirm the above possibility, we measured the FMR intensity for several samples and, according to Eq. (4), we evaluated the dependence of the magnetic moment $\mathcal{M}_s$ on D. Since the correlation between $\mathcal{M}_s$ and $4\pi M_{\rm eff}D$ is surprisingly good (Fig. 3), we believe that $4\pi M_{\rm eff}=4\pi M_s$ for Py/Cu multilayers at room temperature. Therefore, at least at room tem-

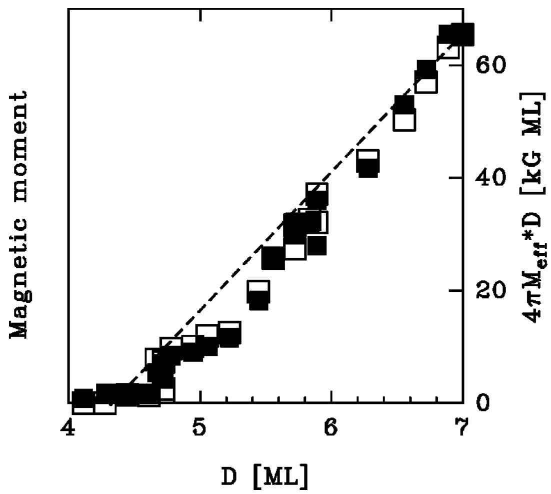

FIG. 3. Magnetic moment $\mathcal{M}_s$ (open squares) and the product of effective magnetization and thickness $4\pi M_{\mathrm{eff}} D$ (solid squares) as a function of the nominal Py thickness at 293 K.

perature, the effective magnetization seems to be solely due to the shape of the Py layers down to a few ML. The same conclusion has been drawn by Smits *et al.*,[19] for Py/Cu multilayers prepared by ion beam sputtering. Unfortunately, a drift in the microwave cavity coupling did not allow us to measure the FMR intensity at 77 K.

The dependence of the FMR linewidth ΔH_{pp} on Py layer thickness (Fig. 4) may also serve as a representative example of cooperative effects characteristic of magnetization fluctuations due to the discontinuous nature of Py layers and the increasing role of defects with decreasing the thickness of Py down to several monolayers. The linewidth is approximately independent of Cu thickness, except the narrow regions where antiparallel coupling occurs. The effect of exchange coupling on ΔH_{pp} is, however, more clearly seen for rather thick Py layers with $d_{\mathrm{Py}} \gtrsim 30$ Å. For smaller d_{Py}, even small fluctuations in Py thickness can substantially destroy this effect. The experimental data can be nicely fitted by the formula

$$\Delta H_{pp} = \Delta H_0 + \frac{A}{d_{\mathrm{Py}}^2}, \tag{6}$$

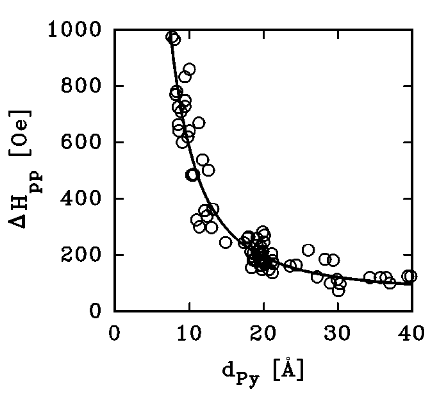

FIG. 4. Peak-to-peak linewidth (at the perpendicular configuration) vs d_{Py}. The solid line represents a fit according to Eq. (6).

with $\Delta H_0 = 64$ Oe and $A \approx 5 \times 10^4$ OeÅ^2 but we have no clear idea of its physical origin. Generally, the FMR linewidth may be decomposed into two terms[14]

$$\Delta H_{pp} = \Delta H_0 + 1.16 \frac{\omega}{\gamma} \frac{G}{\gamma M_s}, \tag{7}$$

where the first term describes the role of magnetic inhomogeneities and the second determines the role of viscous damping. Since both ΔH_0 and G were found to be dependent on thickness,[20] we may only speculate that both terms are involved in the apparent d_{Py}^{-2} dependence.

IV. DISCUSSION

The effects related to the presence of interfaces are shared in the total magnetic behavior of multilayers in inverse proportion to the thickness of the magnetic layer [see Eq. (5)]. Therefore, this simple relation is often used in evaluating the thickness of magnetically inactive layers at interfaces. Depending on the technological peculiarities used for preparing Py/Cu multilayers or spin valves, the thickness of a magnetically inactive layer Δd estimated from magnetic measurements[5–7,9] lies within a wide range of 1.5–10 Å. Our estimation of $\Delta d = 3.8$ Å lies in the middle of this range. Such a remarkable scattering in Δd values seems to be worth explaining from the point of view of possible origins. It is plausible that two main sources are responsible for the interface magnetism in the Py/Cu multilayers: (i) magnetization fluctuations due to size effects of ultrathin Py layers and (ii) interdiffusion between Py and Cu during deposition or intermixing related to interfacial roughness. It is worth noticing that interdiffusion at distances of about a few ML, regarded as local spreading of one component into another, is actually indistinguishable from roughness.

A. Magnetization fluctuations

According to the spin-wave theory, in ultrathin films an enhanced thermal decrease in magnetic order is observed.[21] To estimate the size effects related to the spin-wave excitations we present in Fig. 5 the dependence of the thickness of the magnetically inactive region on the reduced temperature $T/T_C(\infty)$, where $T_C(\infty)$ is the Curie temperature of bulk Permalloy equal to 850 K. The straight line approximating roughly the trend of our data is shifted about 3 ML above that calculated from the spin-wave theory[22] and the experimental points for $Ni_{48}Fe_{52}$ oligatomic films taken from Ref. 23. Hence, the presence of magnetically inactive interfaces in our Py/Cu structures may be mainly attributed to intermixing at interfaces ($\sim$ 1–2 ML per single interface) and partially to the magnetization fluctuations (0.5 ML per single interface) due to the spin-wave excitations. At room temperature, the thickness of magnetically active Py layer is $D_m^* = D - 2(\Delta D - 0.5\mathrm{ML})$, where D is the nominal thickness of a Py layer; $2\Delta D$ is the nominal critical thickness and 2×0.5 ML accounts for the spin fluctuations at room temperature.

For four samples with nominal Py thickness 7.4 Å (4.1 ML), 8.9 Å (5.0 ML), 12.1 Å (6.7 ML), and 23.5 Å (13 ML) we performed temperature measurements of FMR spectra to estimate how the Curie temperature $T_C(D)$ depends on thickness D_m^*. A method proposed by Li *et al.*[13] was applied.

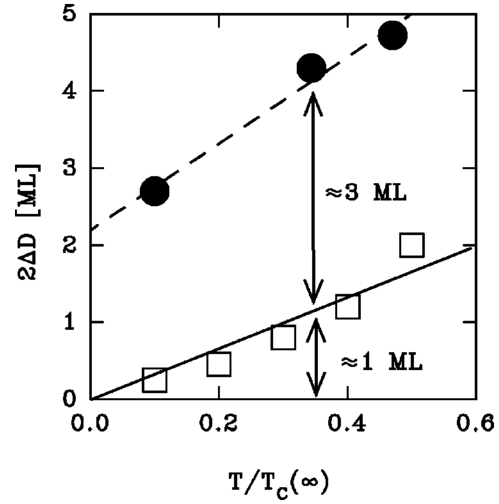

FIG. 5. Temperature dependence of the number of magnetically inactive Py monolayers $2\Delta D$ (solid circles). Experimental points (solid circles) for Py layers are compared with Gradmann's results (open squares) (Ref. 21) and with the spin-wave theory (solid line) (Ref. 22).

Figure 6 shows the reduced Curie temperatures versus thickness D_m^* of a magnetically active layer for Py/Cu multilayers together with relevant experimental data taken from Ref. 1. Our experimental data lie well below the data which were shown to obey the spin-wave theory (see Ref. 1, for details). Such a discrepancy with the spin-wave theory suggests other significant sources determining the magnetic behavior of the Py/Cu multilayers in the ultrathin Py thickness range, but for the best samples[5,9] with $\Delta D \approx 1$ ML the enhanced thermal decrease of magnetic order, combined with changes in the electronic band structure due to proximity effects, may be an important origin of magnetically inactive regions at the interfaces and may have some relations to a Langevin-like magnetoresistance in multilayers with magnetically smooth interfaces.[24]

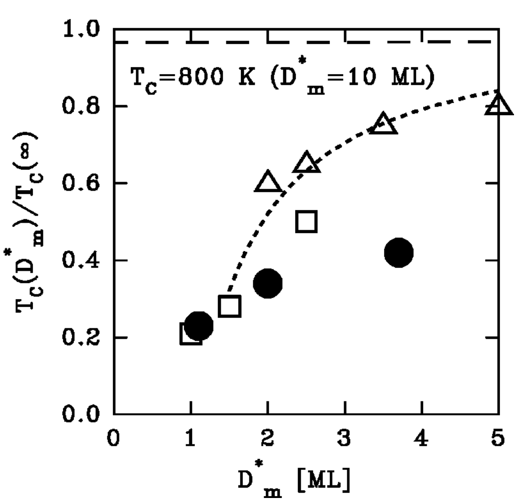

FIG. 6. Reduced Curie temperature of Py layers vs the number of magnetically active atomic layers. Our data (solid circles) are compared with the relevant data for Cu/NiFe/Cu (open squares) and Cu/Ni (open triangles) taken from Ref. 1 and the spin-wave theory (dotted line; see Ref. 1 for details).

TABLE I. A set of parameters of Eqs. (9) and (10) obtained from the least squares fitting to the experimental data in Fig. 2. 1 ML=1.8 Å.

T [K]	$2\Delta D$ [ML]	σ [ML]	w [ML]
77	2.6	5.1	1.7
293	4.2	5.8	0.8
400	4.5	5.8	0.4

B. Intermixing at interfaces

The departure from regular behavior of magnetization (see Fig. 2) expressed by Eq. (5) can be attributed to a magnetic inhomogeneity across an averaged Py layer of thickness range $2\Delta d \leqslant d \leqslant \sigma$, where σ is the characteristic thickness of the intermixed region. For $d \geqslant \sigma$ each additional monolayer contributes to the total magnetization of Py layers and a continuous growth mode is achieved. A simple model of the shape anisotropy of heterogeneous magnetic structures[25] seems to be useful in a quantitative description of the magnetic behavior in the ill-defined region $2\Delta d \leqslant d \leqslant \sigma$. According to this model, the effective magnetization of a film with roughness of the order of σ at each interface can be expressed by

$$4\pi M_{\rm eff} = 4\pi M(d)\left\{1 - \frac{2[3\sigma\epsilon f(1-f)]}{d}\right\}, \qquad (8)$$

where the term in square brackets has the meaning of Δd, $\epsilon f(1-f)$ is a dimensionless factor describing peculiarities of the lateral geometry of roughness, f is a packing factor of the roughness elements, and ϵ is the ellipticity factor of the demagnetization tensor of an individual element forming roughness (see Ref. 25 for details). Since $0.5 < \epsilon < 1$ ($\epsilon = 0.5$ for a needlelike roughness, 1 for a flat island) and $0 < f < 1$, a rough estimate of $4\pi M_{\rm eff}$ is given by $4\pi M(d)(1-\sigma/2d)$, and hence, $2\Delta d \gtrsim \sigma/2$ for a needlelike roughness. Equations (5) and (8) have the same physical meaning provided $4\pi M(d)$ (i.e., a magnetization profile) is known.

The lack of detailed knowledge of the spatial dependence of any inhomogeneity across the average Py layer thickness makes it difficult to make complete estimates of its effect on resonance. We may, however, choose an adequate magnetization profile $4\pi M(d)$ that accounts satisfactorily for the experimentally observed departure from regular behavior. It turns out that the quality of fitting to the experimental data in Fig. 2 depends fairly sensitively on the magnetization profile at the interface region. The best fitting results (depicted by the solid lines in Fig. 2) have been achieved with the assumption of trapezoidal-like magnetization profiles with a set of parameters juxtaposed in Table I.

$$4\pi M(d) = \begin{cases} 0 \quad \text{for} \quad d < 2\Delta d, \\ 4\pi M_s g \quad \text{for} \quad 2\Delta d \leqslant d \leqslant \sigma, \\ 4\pi M_s \quad \text{for} \quad d > \sigma, \end{cases} \qquad (9)$$

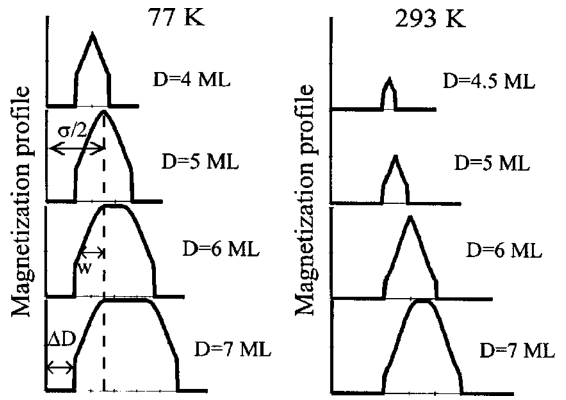

FIG. 7. Shapes of magnetization profiles calculated according to Eqs. (9) and (10) with the set of parameters from Table I.

with

$$g=\exp\left[-\frac{1}{2}\left(\frac{d-\sigma}{w}\right)^2\right], \tag{10}$$

where the Gaussian distribution describes a trapezoidal-like profile. The meaning of σ, Δd, and w is shown in Fig. 7. It is seen from Table I that the characteristic thickness σ of roughness is almost independent of temperature while $2\Delta d$ grows with temperature and the width of distribution function w decreases. In Fig. 7 we present some magnetization profiles of Py layers expressed by Eqs. (9) and (10) for various nominal thicknesses D in ML at 77 and 293 K, respectively. It is seen that at 77 K the central monolayer of a 5-ML-thick Py layer is nearly continuous, while at 293 K a central continuous Py monolayer is expected for a D = 6–7 ML thick Py layer. Such a shape accounts, for example, for a kink in the $4\pi M_{\mathrm{eff}}$ vs D plot at 77 K far much better than the more smeared diffusionlike profiles expressed by the error function. In the next section we will give some physical reasons supporting our model of the magnetic behavior of the rough interfaces.

C. Percolation at interfaces

It has been shown from the discussion of our experimental results that there is a direct interplay between the interface morphology and magnetic properties of ultrathin magnetic layers stacked in magnetic multilayers. In this thickness range, the concept of a surface shape anisotropy does not require strong localization. In most cases, the interfaces are neither sharp nor flat because of interdiffusion and roughness, the latter presenting a locally sharp compositional boundary whose depth varies irregularly in the plane of the structure (Fig. 8). Therefore, the surface shape anisotropy may arise as well from the extended (of a few ML) inhomogeneities in the volume of an ultrathin film.

The main features of the interface magnetism of real multilayers, including the effects related to magnetization fluctuations, can be described by a simple, intuitive model which is based on some concepts of percolation theory. Let us consider a single interface between a magnetic (M) and a nonmagnetic (NM) layer (Fig. 8). Its roughness σ, generated randomly with a computer program, is defined as the standard deviation from a mean surface. Moving across the interface, we find that slightly below the percolation threshold at $\gtrsim\sigma/2$ there are finite clusters only. Depending on the temperature, they may or may not contribute to the total magnetization. Above the percolation threshold, an infinite cluster forms and provides the main contribution to the magnetization, resulting in its fast growth and, hence, a trapezoidal-like magnetization profile. At low temperatures, finite clusters contribute more to the total magnetization since the thermally activated magnetization fluctuations are almost frozen. At room temperature, a superparamagnetic behavior of Py/Cu multilayers with $d_{\mathrm{Py}}<10$ Å has been observed.[16] Therefore, temperature plays the role of a scaling factor which moves up the percolation threshold, which in effect results in a shifting of the critical thickness.

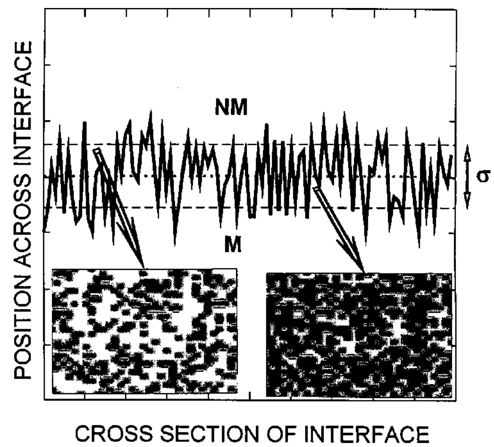

FIG. 8. A schematic sketch showing the cross section of an interface between magnetic (M) and nonmagnetic (NM) layers. Insets show the calculated pattern of finite magnetic clusters (shaded regions) below the percolation threshold (left) and an infinite magnetic cluster above percolation threshold (right).

The percolation probability defined as the ratio of the largest cluster volume to the entire volume may be regarded as a measure of magnetization.[26] In Fig. 9 we plot the percolation probability vs the height measured across the interface. From a comparison of Figs. 7 and 8, it seems that there is a qualitative agreement between the shape of magnetization profiles at a single interface expressed by Eqs. (9) and (10) and that predicted by the percolation model.

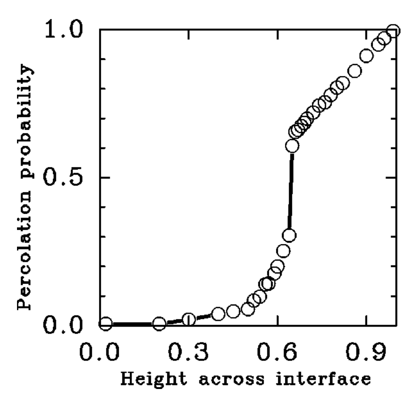

FIG. 9. Site percolation probability as a function of height across the interface shown in Fig. 8.

V. SUMMARY

A coherent, statistically averaged picture of the magnetic structure in Py/Cu multilayers can be provided from the FMR data taken on a large number of samples. It accounts for possible magnetization fluctuations due to spin-wave excitations in ultrathin magnetic layers and magnetization fluctuations of finite clusters formed at the interfaces as well as explaining qualitatively the presence of an ill-defined thickness range in the vicinity of the percolation threshold.

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