

Magnetic properties of Fe/Ni bilayers on Cu(100)

Xiangdong Liu*

*ISG3, Forschungszentrum Jülich, D-52428 Jülich, Germany
and I. Physikalisches Institut der RWTH Aachen, D-52056 Aachen, Germany*

Matthias Wuttig

*I. Physikalisches Institut der RWTH Aachen, D-52056 Aachen, Germany
and ISG3, Forschungszentrum Jülich, D-52428 Jülich, Germany*

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Fe/Ni bilayers with various Fe and Ni thicknesses and different deposition sequences have been prepared by molecular beam epitaxy on Cu(100) substrates. Their magnetic properties have been investigated using the magneto-optic Kerr effect. To study magnetic coupling phenomena in the bilayer, the temperature dependence of the magnetic properties has been studied. It is found that the Ni thickness necessary for the bilayer to start to show perpendicular magnetization is consistently larger than that for Ni films on Cu(100). Magnetic live layers have been observed at room temperature at the Ni-Fe interface for bilayers that include a 9-monolayer Fe film. In addition, such bilayers show peculiar temperature-dependent phenomena. Possible mechanisms underlying the temperature dependence of the remanent magnetization and the coercivity are discussed.

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I. INTRODUCTION

In the last decade a number of new and exciting coupling phenomena and related effects such as oscillatory coupling, giant magnetoresistance (GMR) and tunneling magnetoresistance (TMR) have been discovered in ultrathin magnetic films.¹⁻³ This has led to very promising applications such as spin valves⁴ and sensitive read heads.⁵ On the other hand, in the last two decades a large number of metastable structures with novel and unusual magnetic properties has been stabilized by epitaxial growth.^{6,7} It is interesting to exploit such metastable structures to tailor the magnetic coupling between magnetic films. To determine the potential to tailor the magnetic coupling we have studied the magnetic properties of Fe/Ni bilayers on Cu(100). This system was chosen since both Fe and Ni grow epitaxially on Cu(100) and show interesting structural and magnetic properties as a function of temperature and film thickness.

Iron usually crystallizes in the bcc phase at room temperature, while the fcc γ phase only exists in the temperature range between 1184 K and 1665 K. However, microcrystalline particles of γ iron can precipitate from solid solution in copper and retain their structure at room temperature.^{8,9} Molecular beam epitaxy (MBE) provides another approach to stabilize fcc iron at room temperature. Ultrathin iron films grown on Cu(100) exhibit a rich variety of structural and magnetic phases. For the room temperature grown ultrathin films, two different fcc iron phases are stabilized on Cu(100).^{7,10,31,12} The first one exists for iron layer thicknesses up to 5 monolayers (ML). This iron phase is characterized by a ferromagnetic coupling and an enlarged atomic volume of 12.1 \AA^3 . For iron films on Cu(100) with thicknesses between 5 and 11 ML, only the first two layers couple ferromagnetically and show an atomic volume of 12.1 \AA^3 . The rest of the film has an atomic volume of 11.4 \AA^3 and presumably shows antiferromagnetic coupling at low temperature.^{11,13,14} All films below 11 ML show a perpen-

dicular magnetization. The transition to the stable bcc ground state of iron is observed¹⁵⁻¹⁷ above 11 ML and is accompanied by a switching of the magnetic anisotropy to in-plane orientation.

Ultrathin Ni films grown on Cu(100) also show a peculiar behavior regarding the magnetic anisotropy.^{18,19} For a film thickness between 7 ML and 9 ML, a sharp transition from in-plane to out-of-plane spin reorientation is observed. At much larger film thicknesses between 37 ML and 70 ML, the preferential magnetization direction gradually rotates back to the film plane. Hence in the range from 9 ML to 37 ML, the films show a perpendicular easy axis. Using ferromagnetic resonance, Schulz and Baberschke found that this unusual behavior results from the competition between the perpendicular magnetoelastic volume anisotropy due to mismatch-induced strain, and the sum of shape anisotropy plus surface and interface anisotropy.²⁰ The latter favors in-plane magnetization. Therefore, when the thickness increases beyond the pseudomorphic regime, strain relief by dislocation formation begins and the perpendicular anisotropy is gradually reduced. At last, the easy axis returns back to in-plane orientation.

Hence both systems are characterized by a number of different magnetic states with comparable energy. In such a situation the vicinity of a magnetic interface or simple temperature changes can already alter the magnetic ground state. This is an ideal situation to tailor or modify magnetic coupling phenomena. Therefore it is appealing to prepare Fe/Ni bilayers on Cu(100) to explore the interaction of the two magnetic layers through interfacial coupling. Interestingly enough, in a conclusive study by O'Brien and Tonner for Fe films on 15 ML Ni on Cu(100), a magnetic behavior of the Fe layer has been found that closely resembles the behavior of Fe/Cu(100) in a number of aspects.²¹ Fe films grown on Ni on Cu(100) show practically the same evolution of magnetic phases with Fe film thickness that was previously observed on Cu(100). Below 5 ML, the Fe film on Ni/Cu(100)

is ferromagnetic. Between 5 and 11 ML, Fe films at room temperature show a ferromagnetic live Fe layer at the Fe/Ni interface. In contrast, magnetic live layers are only located at the surface in Fe/Cu(100) films and have a Curie temperature of about 273 K. Evidence for a magnetic live layer at the Fe film surface for Fe/Ni/Cu(100) comes from measurements at low temperature.²² The temperature dependence of the saturation magnetization for 5.3 ML Fe on 7 ML Ni indicates that a live layer exists both at the surface as well as at the interface. Support for the surface live layer comes from the low-energy electron diffraction (LEED) observation of a (2×1) surface reconstruction,²³ which is indicative for an enlarged interlayer spacing and hence ferromagnetic coupling. A similar behavior is also observed for Fe/Co bilayers where the same sequence of crystallographic phases is observed with increasing film thickness.^{21,24}

In most of the previous studies, the main focus was the evolution of structure and magnetic phases of the Fe films with increasing film thickness. Less attention was devoted to other aspects, such as the evolution of the magnetic anisotropy of the bilayer upon changes of the thickness of both Fe and Ni layers. Furthermore, many previous measurements were carried out at room temperature only. Considerable insight into the coupling phenomena of metastable structures is expected from measurements of the temperature dependence of the magnetic properties. For example, an interesting temperature dependence of the magnetization was observed for 5.3-ML Fe/7-ML Ni/Cu(100).²² These data imply that the surface Fe live layer and the interface Fe live layer couple with each other and that the coupling strongly depends upon temperature. More interestingly, exchange biasing was found at low temperature in the Fe/Ni/Cu(100) system within the Fe thickness range, where the (4×1) and (2×1) superstructure coexist.²⁵ To obtain a deeper understanding of interface coupling in Fe/Ni bilayers, we have investigated the magnetic properties of a series of Fe/Ni bilayers with different thicknesses and different deposition sequence for the two elements. Our results show a surprising variety of coupling phenomena that can be attributed to a number of competing contributions to the magnetic anisotropy.

The remaining part of this paper is organized as follows: In Sec. II, the experimental approach is briefly described. The main experimental results are present in Sec. III. Several specific issues are discussed in Sec. IV, while a summary is given in Sec. V.

II. EXPERIMENT

Both, Ni and Fe films were prepared and analyzed in an ultrahigh vacuum chamber that has already been described elsewhere.²⁶ The chamber has a base pressure of 6×10^{-9} Pa and is equipped with a LEED and medium-energy electron diffraction (MEED) system, a cylindrical mirror analyzer (CMA) for Auger analysis and a pair of Helmholtz coils to apply magnetic fields up to 1000 Oe. The substrate was a polished Cu(100) single crystal, approximately 7 mm in diameter and 2.5 mm thick. The sample was cleaned by Ar-ion sputtering with an ion energy of 2 keV at room temperature. Subsequently the Cu crystal was annealed at 900 K for 5 min

to reduce the sputter damage. High-purity Ni (99.98%) and Fe (99.99%) was evaporated from thin disks of 10 mm diameter and 0.2 mm thickness, heated by the radiation of a tungsten filament. A deposition rate of about 0.3 ML/min was chosen for both Ni and Fe. During evaporation, the pressure did not exceed 3×10^{-8} Pa. The growth of the films was monitored by MEED, employing an electron energy of 3 keV at an angle of incidence of 5° against the surface. All films are deposited at room temperature. In order to avoid or at least reduce interface mixing, the second film was grown after the first film had been deposited without subsequent annealing. LEED was used to determine the crystalline order of the substrate and the film. The evaporation rate of both Fe and Ni was calibrated using MEED. For the growth of Ni on Cu(100), clear MEED intensity oscillations can be observed up to at least 15 ML, which enables a precise thickness determination to within 2%. The growth of Fe films on Cu(100) has already been investigated in considerable detail.⁷ Regular intensity oscillations are observed for film thicknesses between 5 and 11 ML. Again, these oscillations can be exploited to determine the film thickness to within 4%.

The situation is less favorable when a second film is deposited, i.e., when Fe is deposited on a Ni film or vice versa. Then, weaker and short-lived oscillations are observed only, which most likely have to be attributed to the rougher surface formed after deposition of the first film. As a consequence, a larger error bar of roughly 10% exists in the film thickness determination from the MEED data for the second film. Presumably, however, this error bar overestimates the true error, since we have used parameters for the evaporation source identical to previous runs, where Fe and Ni had been deposited separately, and the deposition rate could be determined with high precision.

Magnetic properties of the films were characterized using the magneto-optic Kerr effect (MOKE). Hysteresis loops were always recorded in polar geometry, for which the magnetic field is applied perpendicular to the film plane and the magnetization is measured along the field direction. The light source was a He-Ne laser with a wavelength of 632.8 nm. The Kerr effect was measured employing a null ellipsometer with polarizer-sample-compensator-analyzer arrangement.

III. RESULTS

A. Ni/Fe bilayers on Cu(100)

Two typical Fe layer thicknesses were chosen for this deposition sequence: 3 ML and 9 ML. The LEED pattern shows a (5×1) superstructure at low temperature for 3 ML Fe on Cu(100), while a (2×1) reconstruction is observed for 9 ML Fe/Cu(100). These LEED observations are consistent with previous studies.^{26,10} Many of the conclusions in this section are based upon the temperature dependence of the magnetic properties of Fe-Ni bilayers on Cu(100). To clearly distinguish the new features resulting from the coupling of Fe and Ni layers, we first examine how the magnetic properties of Fe films on Cu(100) change with temperature. The key magnetic parameters such as remanent magnetization (M_R), saturation magnetization (M_S), and coercivity (H_C)

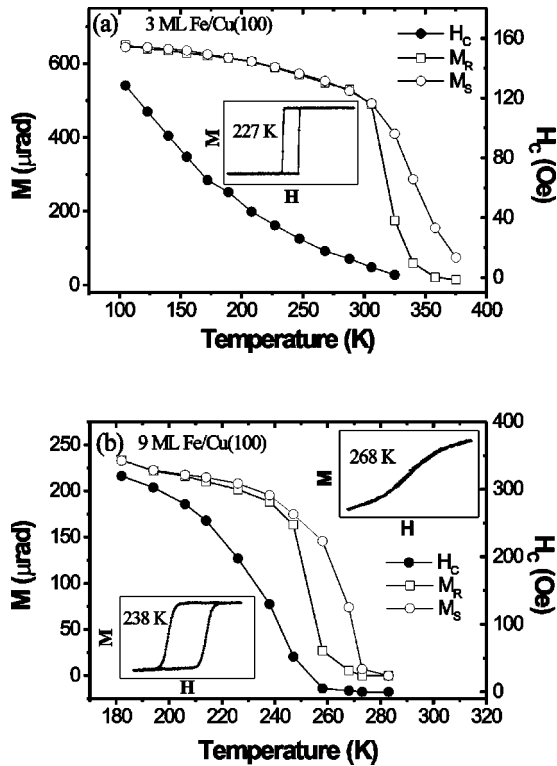


FIG. 1. Saturated magnetization (M_S), remanent magnetization (M_R), and coercivity (H_C) of (a) 3-ML Fe film on Cu(100) and (b) 9-ML Fe/Cu(100) as a function of temperature. The insets show the hysteresis loops at the denoted temperature.

are plotted in Fig. 1. The 3-ML-thick Fe film shows considerable M_S even at 370 K. This is in agreement with a previous report that a 3-ML Fe film has a Curie temperature of 390 ± 30 K.²⁷ A rapid decrease in M_S is observed around 270 K for the 9.2-ML Fe/Cu(100). Here a smaller overall magnetization as compared with the 3-ML film is observed. A vast number of studies confirms that for Fe films with a thickness between 6 and 11 ML, the film surface exhibits ferromagnetic ordering (magnetic live layer) below around 250–270 K.^{7,28,29} The remaining inner part is believed to be paramagnetic at room temperature and antiferromagnetic at low temperature.^{31,30,11} The existence of a surface magnetic live layer explains both the smaller magnetization and lower Curie temperature of the 9 ML Fe film compared with the 3 ML Fe film.

Both 3- and 9-ML films show a perpendicular magnetic anisotropy, but a close look at the hysteresis reveals subtle differences (see the insets in Fig. 1). For the 3 ML thick film, an ideal rectangular hysteresis loop is observed. The exact coincidence of remanent and saturation magnetization is indicative of a stable single-domain state. The magnetization reversal process is dominated either by a coherent rotation of all spins or by rapid motion of domain walls over long distances once a few areas with reversed magnetization have been nucleated.³² The hysteresis loop of a 9-ML-thick Fe film, in contrast, is characterized by an inclined slope instead of a vertical slope. Similar loops have been observed for Au/Co/Au sandwiches. For this system it was found using Faraday-rotation microscopy³² that the magnetization rever-

sal is dominated by the formation of the nuclei. The process has been ascribed to the random appearance of nucleation centers in the sample and irregular domain growth of these nuclei over short length scales. Such a mechanism gives rise to a nonrectangular hysteresis because of the local variation of the nucleation and propagation fields, which provides a distribution of coercive fields throughout the sample.

For both films, a considerable difference between remanent and saturation magnetization is observed when the temperature approaches the Curie temperature. Above a certain temperature, which is considerably smaller than the corresponding Curie temperature, the hysteresis loop vanishes. The same phenomenon has also been observed in Ni/Cu(100) films, which also show a perpendicular easy axis.³³ Three different explanations have been put forward to account for the disappearance of the hysteresis loop. In the first model this was explained by a transition from a single-domain state to a multidomain state with perpendicular magnetization component at a certain temperature below T_c .³³ An alternative mechanism was proposed by Jensen and Bennemann.³⁵ They took the entropy of magnetization into account and deduced that above a temperature below the Curie temperature, the magnetization in the presence of a perpendicular anisotropy is aligned completely parallel to the surface plane. A third theoretical explanation for this reorientation of the magnetization was given by Pescia and Pokrovsky.³⁴

We now want to discuss the magnetic properties of the Ni/Fe/Cu(100) bilayers. The first case we present consists of Ni films with different thickness on 3-ML Fe on Cu(100). A compilation of hysteresis loops at various temperatures can be found in Fig. 2. The 14-ML Ni/3-ML Fe/Cu(100) sample is characterized by well-defined rectangular hysteresis loops in the whole measured temperature range. This result reveals that this film possesses a perpendicular easy axis and a stable single domain state in the whole temperature range measured.

The temperature dependence of the magnetization of the four films, which was measured in a field of 380 Oe perpendicular to the film plane, is displayed in Fig. 3. We denote this magnetization value as “measured magnetization.” For a perfect square-shaped hysteresis loop the measured magnetization is identical to both the saturation and remanent magnetization. For other hysteresis loops, like the ones characteristic for 4.7, 8, and 11 ML on 3-ML Fe on Cu(100), the magnetization measured at 380 Oe is much larger than the remanent magnetization, showing that the magnetization is only saturated at very high fields for these samples. The change of the magnetization with temperature measured at a field of 380 Oe for the 14-ML Ni/3-ML Fe bilayer on Cu(100) behaves as expected (Fig. 3). The magnetization increases with decreasing temperature.

The situation is more complex for the 11-ML Ni/3-ML Fe/Cu(100) sample. An interesting evolution of the hysteresis loop can be found with decreasing temperature (Fig. 2). Below the Curie temperature of 390 K for 3-ML Fe films on Cu(100) but above 300 K, no coercive behavior is observed. In this temperature range, the magnetization measured at 380 Oe increases dramatically with decreasing temperature. Dis-

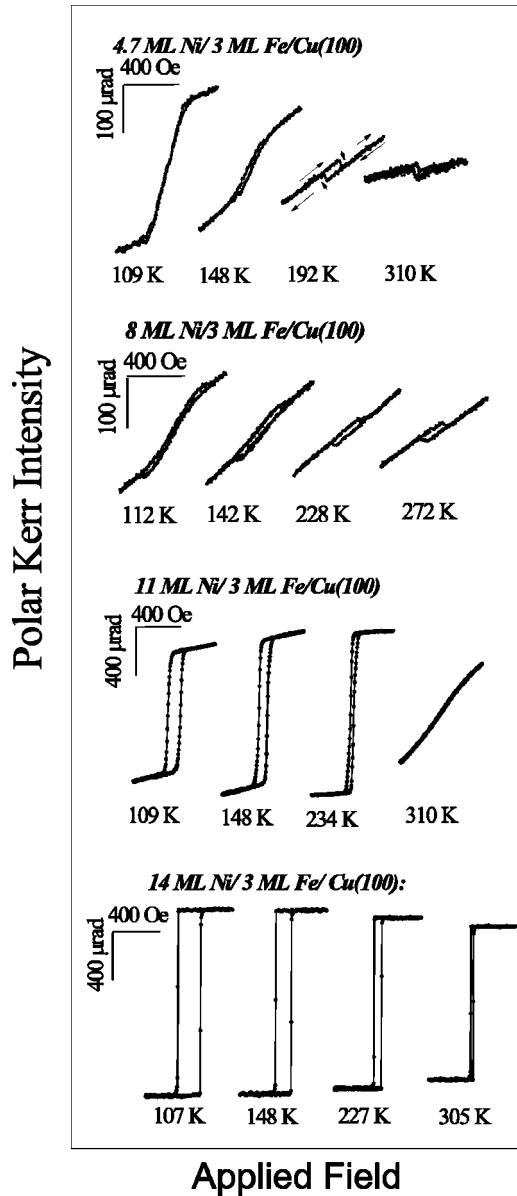


FIG. 2. A compilation of hysteresis loops (for a series of samples with different Ni film thickness on 3-ML Fe/Cu(100)). For 4.7-ML Ni/3-ML Fe/Cu(100) and 8-ML Ni/3-ML Fe/Cu(100) inverted hysteresis loops are observed. The arrows indicate the field sweeping direction.

cernible hysteresis loops begin to appear at 298 K. In this temperature regime, the difference between the remanent magnetization and the measured value at 380 Oe field is rather small. The magnetization shows a temperature dependence similar to the 14-ML Ni/3-ML Fe/Cu(100) sample. However, if the temperature is reduced below 170 K, the hysteresis loops become more and more canted. Hence, the ratio of the remanent to the measured magnetization gets smaller with decreasing temperature. In addition, the measured magnetization also drops with decreasing temperature. The drastic increase of the field necessary to reach saturation implies that the easy axis gradually deviates from the perpendicular direction with decreasing temperature. The disappearance of the hysteresis loop and the reduction of measured

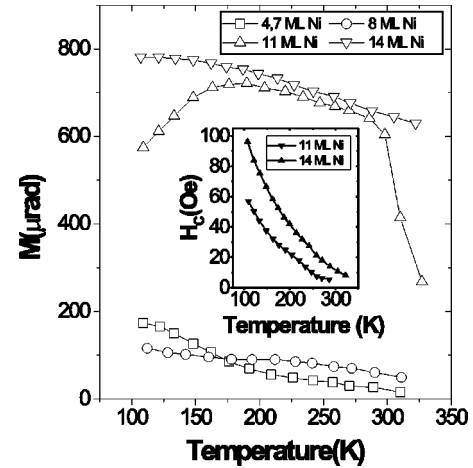


FIG. 3. Temperature dependence of the measured magnetization at 380 Oe of a series of samples with different Ni film thickness on 3-ML Fe on Cu(100). The inset shows how the coercive field changes for the samples with 11- and 14-ML Ni on 3-ML Fe/Cu(100).

magnetization at high temperature for 11-ML Ni/3-ML Fe/Cu(100) can be explained using the similarity with the high-temperature behavior of 3-ML and 9-ML Fe on Cu(100).^{35,34} The observation can be attributed to the transition of the easy axis from a nearly perpendicular direction to the in-plane direction, because a film with an in-plane magnetization has a larger entropy of magnetization. At high temperature the contribution of the entropy to the Gibbs enthalpy becomes more important and leads to a rotation of the magnetic anisotropy. Similar observations have been reported for several other thin film systems.^{36,37} The rotation of magnetization direction from the film normal at low temperature can be attributed to the competition between several contributions to the anisotropy that have different temperature dependences. In the Ni/Cu(100) system, the same mechanism leads to a gradual rotation of the perpendicular direction below a critical temperature for film thicknesses around 7 ML.^{38,39} The rich variation in preferential magnetization shows that the anisotropies favoring perpendicular magnetization and those favoring in-plane magnetization are very similar in magnitude. Hence the anisotropy field⁴⁰ is almost zero. This is quite surprising, considering the fact that the spin reorientation transition thickness to perpendicular is 7–9 ML for the Ni/Cu(100) system and that a 3-ML Fe film on Cu(100) shows a perpendicular easy axis as well. It indicates that the Fe/Ni interface has a considerable influence on the magnetic anisotropy and favors an in-plane alignment. We will discuss this issue again in Sec. IV.

Another conclusion can be derived from the size of the magnetization for 14-ML Ni/3-ML Fe and 11-ML Ni/3-ML Fe bilayers. The magnetization values are higher than for the uncovered 3-ML Fe film on Cu(100), but the difference can be attributed to the contribution of the Ni film to the overall magnetization. The observed magnetization can be described as the sum of the contribution from the Ni film⁴¹ and the contribution from the 3-ML Fe film, which is of the order of 580 μ rad. This implies also that the magnetic moment of

the underlying Fe film cannot be dramatically altered by the presence of the Ni capping layers. Hence, we can conclude that the high-spin state of fcc iron is stable upon overcoating by Ni.

For the cases in which the Ni film thickness is 4.7 and 8 ML, respectively, the measured magnetization is much smaller. The reduction of total magnetic moment due to thinner Ni layers cannot produce such a dramatic decrease in the measured magnetization, because Ni has both a much smaller magnetic moment and a rather weak magneto-optic interaction compared to Fe. The small measured magnetization in these two samples unambiguously indicates that the easy axis is not aligned in the perpendicular direction. These two samples also show puzzling magnetization curves. Most of the M - T curves are characterized by an inclined line superimposed on an inverted hysteresis loop (Fig. 2). An inverted loop means that the coercivity and remanent magnetization are negative [see the loop of 4.7-ML Ni/3-ML Fe/Cu(100) in Fig. 2; the arrows denote the sweeping direction of the applied field]. Identical loops have been measured in Cu/Co and Cu/Ni superlattices.⁴² Negative remanent magnetization and coercivity have also been observed in Fe thin films^{43,44} and exchange-coupled systems.^{45,24} To our knowledge, negative remanence and coercivity only appear when the hysteresis loops are recorded in a direction almost perpendicular to the magnetic easy axis. A recent study⁴³ revealed that in a system with several competing anisotropies, negative remanence and coercivity can be observed if the magnetic field is not applied exactly perpendicular to the easy axis but rotated away from this direction by a small amount of less than 5° .

We can hence conclude for the Ni/Fe bilayer with a 3-ML Fe underlayer that the Fe/Ni interface favors an in-plane alignment, which leads to a higher Ni film thickness for the spin reorientation. The overlayer of Ni, however, does not destroy the ferromagnetic high spin state of the Fe underlayer.

We now turn our attention to the Fe thickness regime where a ferromagnetic live layer exists on Cu(100). We now want to see how this film is influenced by a Ni overlayer. In the following we fix the Fe underlayer thickness at 9 ML but vary the thickness of the Ni top layer. Seven samples have been studied with Ni film thicknesses of 17.5, 16, 14, 12.5, 11.8, 10.4, and 8 ML, respectively. A collection of typical hysteresis loops is presented in Fig. 4. Data on the magnetization of these samples are plotted in Fig. 5 as a function of temperature. The coercive field of several samples is shown in Fig. 6. Depending on the thickness of the Ni layers, three different types of behavior are observed. For small Ni thicknesses, a positive anisotropy field H_{an} is observed.⁴⁰ This holds for 8-ML Ni/9-ML Fe/Cu(100) and 10.4-ML Ni/9-ML Fe/Cu(100). No hysteresis effect was measured and the measured magnetization is rather small. Therefore both samples are characterized by an in-plane anisotropy. However, this in-plane anisotropy decreases with increasing film thickness. Hence for the 10.4-ML Ni film there is already evidence for a rotation of the anisotropy. At 97 K, a very small but discernible hysteresis loop is detected, which implies that the easy axis has rotated out of the film plane. For the samples

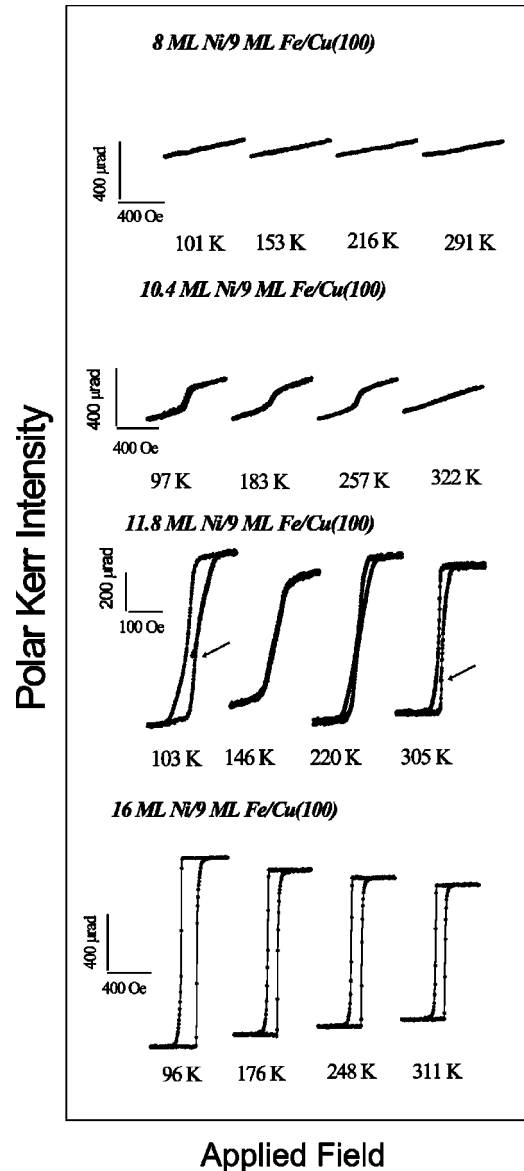


FIG. 4. A collection of hysteresis loops for a series of samples with different Ni film thicknesses on 9-ML Fe/Cu(100). The arrows indicate the starting points for the first loop.

with thick Ni overlayers (14, 16, and 17.5 ML), rectangular-like hysteresis loops are recorded, indicating a negative anisotropy field. The temperature dependence of the measured magnetization can be described by a linear increase with decreasing temperature. Only in a limited temperature regime is a deviation from this linear behavior observed. The deviation is reduced with increasing Ni thickness. The linear temperature dependence is usually a distinctive phenomenon of two-dimensional systems.⁴⁶⁻⁴⁸ A theoretical study²⁹ has predicted a linear temperature dependence of the magnetic moment of the interface Fe layer in the 11-ML Fe/Cu(100) system. We suggest that it is the Fe/Ni or Fe/Cu interface layer that contributes in our case to the linear temperature dependence. The Ni film could not cause this effect since the dimensionality crossover from three-dimensional (3D) Heisenberg to 2D XY behavior occurs at 7 ML for Ni films,¹⁸ which

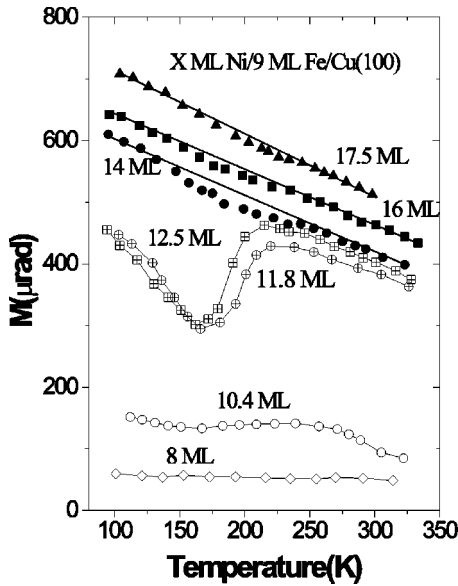


FIG. 5. Temperature dependence of the remanent magnetization for a series of samples with different Ni film thickness on 9-ML Fe on Cu(100).

is much lower than the thickness of our samples. For the two samples with a thickness of 11.8 and 12.5 ML, the magnetization curves present a deep dip around 175 K (Fig. 5). With temperature decreasing from room temperature, the magnetization at first increases slowly, but then starts to fall dramatically around 210 K. Between 160 and 170 K, a deep minimum appears until the magnetization starts to increase again with decreasing temperature. Traces of these dips are also visible for bilayers with larger Ni film thickness. This implies that the mechanism responsible for the decrease of magnetization is also present in the thicker Ni films but is compensated more efficiently by competing contributions to the total energy.

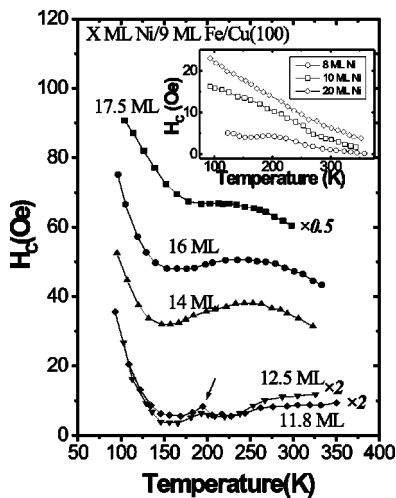


FIG. 6. Temperature dependence of the coercive field for a series of samples with different Ni film thickness on 9-ML Fe on Cu(100). For a comparison, the coercive behavior of Ni films on Cu(100) with different thickness is shown in the inset.

A close inspection of the evolution of the hysteresis loops with temperature for the 11.8-ML Ni/9-ML Fe bilayer is helpful for understanding this phenomenon. At room temperature, a narrow and corner-rounded hysteresis loop is measured. The sample shows almost 100% remanent magnetization, but the starting magnetization value, which was measured at the beginning when the applied field was swept from 0, is much lower than M_R (see arrow in Fig. 4). These results suggest that the sample shows a small anisotropy field in the direction of the applied field, i.e., perpendicular to the film plane, but the single domain state is not the stable state, so the single domain in the field relaxes into multidomains when the field is removed. This relaxation is much slower than the duration of one sweep of the applied field. This kind of relaxation procedure is easy to observe in magnetic films with a small anisotropy field.⁴⁹ With decreasing temperature, the remanent magnetization also decreases gradually and disappears at 220 K. Correspondingly, the loops become more and more inclined and gradually evolve into a sheared “hourglass” shape. However, the high field magnetizing curve after reversal remains a horizontal line until 220 K are reached. This shape evolution bears surprising resemblance to that reported in Ref. 49 but there the direction of change is reversed with respect to temperature. This implies that it is not the temperature itself but rather the different temperature dependence of the competing anisotropies that dominates the evolution. The shape change of the hysteresis loop mainly results from two factors: the anisotropy field and the nucleation field of the reversal domain. When the temperature is reduced below 220 K, the shape of hysteresis evolves differently. The area of the hysteresis loop shrinks rapidly and degenerates into two almost overlapping inclined lines; the high-field part of the magnetizing curve now also becomes inclined. Hence the measured magnetization is no longer identical to the saturation magnetization. Reflected in the hysteresis loops is the steep drop in magnetization around 200 K. When the measured magnetization has reached the minimum value, the hysteresis effect is very small but still discernible. Therefore the magnetizing curve has the form of a straight line with two different slopes: a large slope for the center (low-field) segment and a small one for the high-field part. This magnetizing curve can be understood if the main mechanism involved in the magnetization reversal process is the reversible motion of the domain wall in low fields and the reversible rotation of magnetic moments in high field. The conclusion is that upon cooling below 210 K, the easy axis of magnetization rotates away from the perpendicular direction continuously. The angle with which the easy axis deviates from the film normal increases continuously when the temperature is reduced from 210 K to 160 K. With further reduction of temperature, the easy axis returns towards the normal direction, as is reflected in the increase of the measured magnetization and the hysteresis loop as well as the reduction in the slope of the high-field magnetizing curve.

The coercive field seems more subject to the interface coupling (Fig. 6). Even though both single Ni/Cu(100) films (see the inset in Fig. 6) and 9-ML Fe/Cu(100) (see Fig. 1) show a monotonically temperature-dependent coercivity, the

coercive field of all the Ni/Fe bilayers, if they show measurable coercivity, presents a more complicated behavior. With decreasing temperature, for the samples with a thick Ni top layer (17.5, 16, and 14 ML), the coercive field first increases, then gradually starts to decrease and at last rises sharply at low temperatures. Hence a minimum and a maximum are observed in the “wave-shaped” H_C - T curves. The undulation amplitude becomes smaller when the Ni layer thickness increases. Even more complex is the behavior of bilayers with 11.2- and 12.5-ML Ni. Here a much wider dip is observed, with possibly two minima. Yet again, at low temperatures a rapid increase in coercivity is observed.

In contrast to the Ni/3-ML Fe/Cu(100) films, two new features have been observed in Ni/9-ML Fe/Cu(100) bilayers. A magnetization drop was observed to start around 210 K for the samples that show a small perpendicular anisotropy field at room temperature. In addition the coercivity demonstrates a nonmonotonic temperature dependence. We will return to these observations in Sec. IV.

B. Fe/Ni bilayers on Cu(100)

The deposition sequence of the bilayer could have a pronounced impact on the magnetic phases and anisotropies in the bilayers. Therefore we have also investigated the magnetic behavior of Fe/Ni bilayers. The slight difference in lattice constants of Cu (3.62 Å), Ni (3.52 Å), and γ -Fe (3.59 Å)⁹ will introduce strain into the epitaxially grown bilayers. The strain will relax if the film thickness is beyond the pseudomorphic growth region.⁵⁰ The magnetic anisotropy is closely related to the film strain. In addition, the magnetic properties of the fcc Fe layer are very sensitive to the lattice spacing.^{51–53} Theoretical calculations show that the nonmagnetic and antiferromagnetic solutions are almost degenerate for lattice constants around 3.5 Å. Hence the change of the deposition sequence could profoundly modify the magnetic properties of the bilayers. To confirm this, we have investigated two typical samples: 9-ML Fe/10-ML Ni/Cu(100) and 9-ML Fe/15-ML Ni/Cu(100).

At room temperature, the 9-ML Fe/10-ML Ni/Cu(100) sample shows no hysteresis loops but considerable measured magnetization (Fig. 7). This behavior indicates that the sample has a small positive anisotropy field. The magnetization prefers to lie in the film plane but is prone to follow the perpendicular applied field. No hysteresis effect is detected down to 222 K. At 204 K, however, a hysteresis loop appears. With decreasing temperature, the width of the hysteresis loop, i.e., the coercive field, increases rapidly. Figure 8 shows how the measured magnetization and the remanent magnetization change with temperature. The temperature dependence of H_C is plotted in the inset of this figure.

Below 210 K, the difference between the magnetization measured at 380 Oe and the remanent magnetization is reduced rapidly. With a further decrease of temperature M_R decreases again, presumably since now the in-plane anisotropy starts to prevail. It should be noted that both 10-ML Ni on Cu(100) and 9-ML Fe on Cu(100) show a perpendicular anisotropy. The fact that 9-ML Fe on 10-ML Ni on Cu(100) only show this anisotropy over a very limited temperature

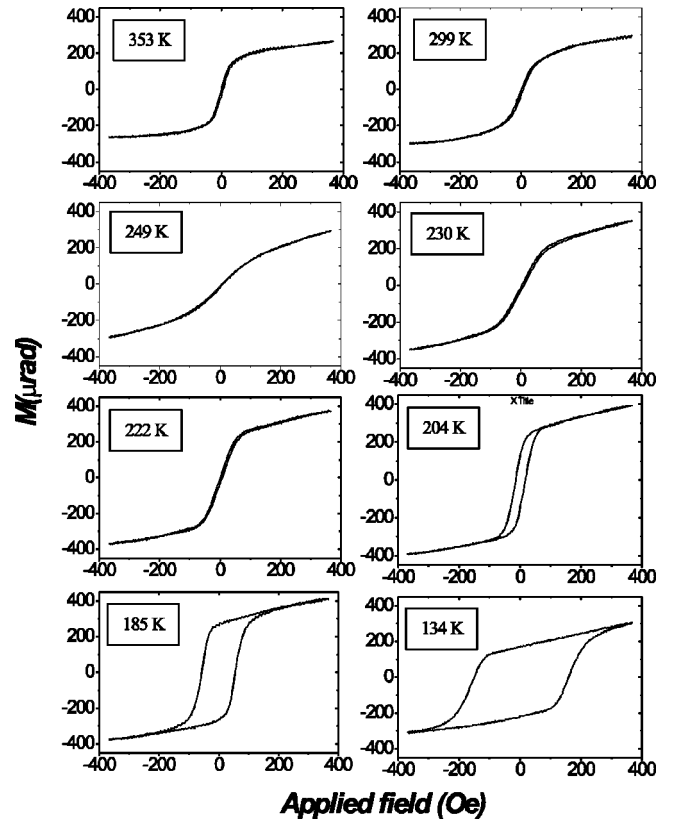


FIG. 7. Magnetization curves at different temperatures for 9-ML Fe/10-ML Ni/Cu(100).

range implies again that the deposition of Fe on Ni enhances the in-plane anisotropy. The strong decrease of the magnetization measured at 380 Oe could be due to a ferromagnetic-paramagnetic phase transition around 250 K in parts of the film. Indeed, the Curie temperature for the surface live layer of 9-ML Fe on Cu(100) is about 270 K. Hence we suggest

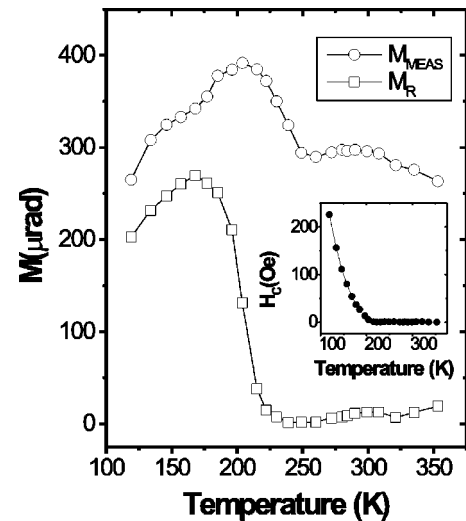


FIG. 8. Magnetization M_{MEAS} measured at 380 Oe (open circle) and remanent magnetization M_R (open square) for 9-ML Fe/10-ML Ni/Cu(100). The sample is not yet saturated at 380 Oe. The coercivity is plotted in the inset.

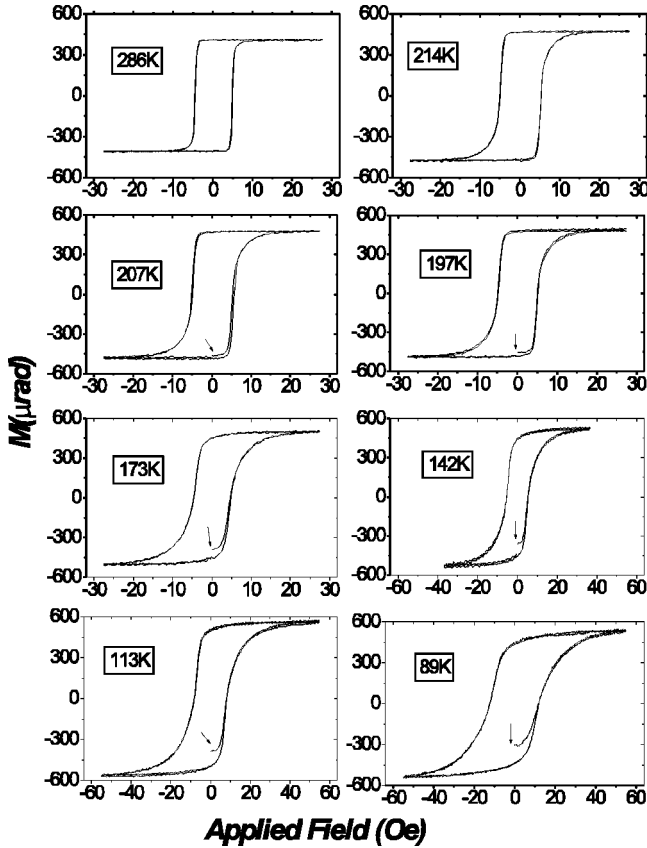


FIG. 9. Hysteresis loops for the first two sweeps at different temperatures for 9-ML Fe/15-ML Ni/Cu(100). The arrows indicate the starting points for the first loop.

that the transition in the measured magnetization around 250 K could be related to the Curie temperature of the surface live layer of Fe on Ni/Cu(100). The situation is nevertheless not that clear cut since we cannot derive much further information from the magnitude of the measured magnetization. 10-ML Ni on Cu(100) has a Kerr signal at remanence of approximately $170 \mu\text{rad}$ at 250 K. 9-ML Fe on Cu(100) could add a Kerr signal of $225 \mu\text{rad}$ at 200 K (Fig. 1). However we also expect a contribution of similar size from the Fe/Ni interface, which should lead to a ferromagnetic live layer at the Fe/Ni interface. Then the measured magnetization above 250 K would be attributed to the Ni film and the magnetic live Fe layer at the Fe/Ni interface.

A different behavior is found for the 9-ML Fe/15-ML Ni/Cu(100) sample. The hysteresis loops of two sweeping cycles at various temperature can be found in Fig. 9. At room temperature, a rectangularlike loop is observed. The remanent magnetization is identical with the magnetization measured at 380 Oe, which should correspond to the saturation magnetization M_S . Hence the sample possesses a perpendicular anisotropy field and a stable single-domain state. Below 220 K two changes are detected. The hysteresis loops become increasingly round. This leads to a reduction in remanent magnetization with decreasing temperature (see Fig. 10). In addition, the starting points of the hysteresis loops (denoted by arrows in Fig. 9) are no longer located on the M - H curves recorded during the second sweeping cycle. The

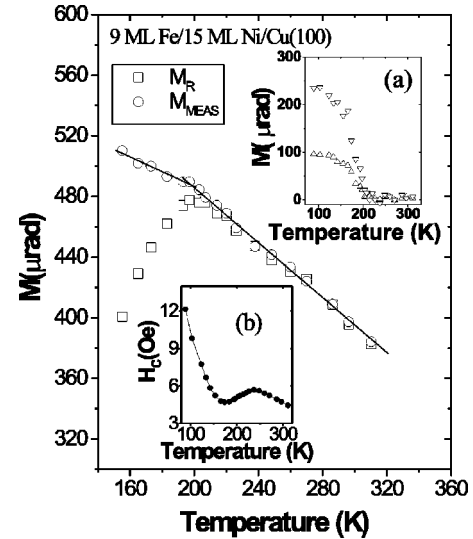


FIG. 10. Magnetization M_{MEAS} measured at maximum field in Fig. 9 (O) and remanent magnetization (open square) for 9-ML Fe/15-ML Ni/Cu(100). In inset (a), the difference between M_{MEAS} and M_R (∇) as well as between M_{MEAS} and M_{START} (Δ) is displayed. Inset (b) shows the temperature dependence of the coercivity.

deviation of the starting magnetization M_{START} from M_R is first discernible at 207 K and increases with decreasing temperature. To understand this phenomenon it is necessary to describe the measuring procedure in more detail. The measurement was carried out with increasing temperature. Data were recorded for two sweeping cycles. The sweeping rate of the applied field is 0.015 s/Oe. The time interval between two subsequent measurements is about 5 min.

As is obvious from Fig. 9, the starting magnetization is smaller than the magnetization in the second loop once the sample temperature is less than 214 K. Figure 10 shows in the top inset the differences between M_{MEAS} and M_R and between M_{START} and M_{MEAS} as a function of temperature. These plots strongly suggest that the magnetic anisotropy changes around 200 K. A nonmonotonic temperature dependence has been observed for the coercive field, similar to what we see in Fig. 5. In Fig. 10 the remanent magnetization and the magnetization measured at 380 Oe are displayed as well. Within the whole temperature range studied no magnetization jump is observed. This implies that the Fe layer of this sample shows no magnetic live layer or it has a live layer but with a Curie temperature considerably above room temperature. A simple analysis can exclude the latter assumption. For 9-ML Fe/10-ML Ni/Cu(100) a measured magnetization of $290 \mu\text{rad}$ at 310 K is observed. For 9-ML Fe/15-ML Ni/Cu(100) a value of $380 \mu\text{rad}$ is found. The difference is attributed to the additional 5 ML of Ni.^{41,54} Presumably the 9-ML Fe film on 10-ML Ni/Cu(100) has a live layer, but with an ordering temperature considerably below room temperature. Therefore it does not contribute to the magnetization. On the other hand, a magnetization of about $200 \mu\text{rad}$ was measured for 9-ML Fe on Cu(100) below 270 K, which is attributed exclusively to the magnetic live layer. Therefore if 9-ML Fe/15-ML Ni/Cu(100) had a live layer

with a Curie temperature above room temperature, it should show a much higher magnetization at room temperature.

IV. DISCUSSIONS

A. Spin reorientation transition

The present work shows unequivocally that in Fe/Ni bilayers the spin reorientation transition shifts to larger Ni film thicknesses compared with Ni/Cu(100). This conclusion is supported by the data presented in Figs. 4, 7, and 9. While 8-ML Ni on Cu(100) shows a rectangular hysteresis loop at room temperature neither 8-ML Ni/3-ML Fe/Cu(100) (Fig. 3) nor 8-ML Ni/9-ML Fe/Cu(100) (Fig. 5) shows a perpendicular anisotropy field. 9-ML Fe/10-ML Ni/Cu(100) also possesses a weak in-plane anisotropy field at room temperature. This demonstrates that the spin reorientation transition is shifted to larger Ni film thicknesses. Two effects caused by the Fe/Ni bilayer could explain the shift.

The demagnetization field of the bilayer is not the simple sum of the individual contribution of Ni and Fe films with the same thickness on Cu(100). The demagnetization field of the bilayer is usually larger than the sum of the individual contributions, since the magnetic moments of the Fe (Ni) layer also contribute to the local dipolar field exerted on the Ni (Fe) layer. Considering that Fe has a much larger magnetic moment ($2.2\mu_B$) than Ni ($0.62\mu_B$) implies a considerable gain in shape anisotropy due to the bilayer. In addition, when Ni (Fe) is deposited on a Fe(Ni)/Cu(100) film, a Fe/Ni interface replaces a Fe/vacuum surface and a Ni/Cu interface. To our knowledge, no quantitative data on the Fe/Ni interface anisotropy are available. The interface anisotropy possibly contributes to the in-plane anisotropy.¹⁹ This should lead to a thicker Ni layer necessary to achieve the spin reorientation transition in bilayers.

B. Magnetic live layers

It is well established that a magnetic live surface is located at the film surface in 5–11-ML-thick Fe/Cu(100) films.^{7,28} The magnetic live layer shows a Curie temperature around 270 K. The remaining fraction of the film couples antiferromagnetically at low temperature. The surface magnetism is related to the enlarged atomic volume, which favors a ferromagnetic ground state. The question arises whether the magnetic live layer only exists in the Fe/Cu(100) system or if it can also be stabilized in other systems. Measurements at room temperature did not find any evidence for a surface live layer in Fe/Co/Cu(100) and Fe/Ni/Cu(100) systems.^{21,19,24} Instead in both cases magnetic Fe live layers at the interface were observed. For the different Fe/Ni bilayers studied here there is also ample evidence of magnetic Fe layers. This is mainly supported by the size of the Kerr rotation measured and the temperature dependence of the magnetization signal. Consider for example the 9-ML Fe overcoated by various Ni films. The resulting magnetization (Fig. 5) cannot be explained by the Ni film only. This becomes evident if films with different Ni thickness are compared. Subtracting the Ni magnetization, which we have determined in a previous study to 18–24 μrad per Ni ML, which in-

creases with increasing thickness, leads to a contribution of approximately 225 μrad from the Fe film. This is considerably lower than the magnetization of a homogeneously magnetized Fe film. Please note that the homogeneously magnetized Fe film with a thickness of 3 ML already has a magnetization of more than 600 μrad . On the other hand, the 9-ML Fe film on Cu(100) without a Ni overcoat has a remanent magnetization of approximately 225 μrad , in close agreement with the value attributed to the iron film in the Ni/Fe bilayer on Cu(100). Hence only a small fraction of the Fe film can be ferromagnetic. Since the film still shows a high magnetization above 320 K, i.e., much above the Curie temperature of 270 K for an 9-ML Fe film without Ni overlayer, this implies that the ferromagnetic fraction of the Fe film is located at the Fe/Ni interface. The bilayer hence shows a magnetic live Fe layer at the Fe/Ni interface. The most complex behavior is expected and observed for Fe/Ni bilayers on Cu(100), where the iron film grows on Ni. This deposition sequence should lead to a ferromagnetic live Fe layer at the Fe/Ni interface and possibly also a ferromagnetic surface layer of the iron film. Indeed, evidence for two ferromagnetic live Fe layers with complex coupling phenomena has been observed in a recent study where the films were grown on a thinner Ni underlayer and hence showed an in-plane anisotropy.²² Again, the observed magnetization (Figs. 8 and 10) shows that only a fraction of the Fe film is ferromagnetic. However, the situation is more complex than that for the Ni/Fe bilayer on Cu(100), since we expect now a ferromagnetic contribution from both the Fe/Ni interface and the Fe film surface. The temperature dependence of the measured magnetization for 9-ML Fe/10-ML Ni/Cu(100) shown in Fig. 8 gives evidence that a fraction of the Fe film becomes paramagnetic above 250 K. This temperature is much below the Curie temperature of the Ni film and hence also presumably below the Curie temperature of the Fe magnetic layer at the Fe/Ni interface. Fe films on Cu(100) in this thickness range, however, have a Curie temperature of 270 K. Therefore we attribute the rapid reduction in magnetization observed for the 9-ML Fe/10-ML Ni/Cu(100) sample to the loss of ferromagnetic order of the film surface. No such temperature dependence is observed for the 9-ML Fe/15-ML Ni/Cu(100) sample. This would imply that in this case the Fe film surface has no magnetic live layer. Without further experimental data to support or contradict this assumption we can only speculate about the underlying cause of such a phenomenon.

A possible explanation is that the thickness of the underlying Ni layer has an influence on the lattice parameters of the top Fe layer. Ni can be grown on Cu(100) pseudomorphically up to a critical thickness. Beyond this thickness, dislocations form and the lattice constant starts to relax back to the bulk value. O'Brien *et al.* estimated the critical thickness to 13 ML.¹⁹ Because bulk Ni has a smaller lattice constant (3.52 Å) than Cu (3.62 Å), a smaller atom spacing is expected at the surface of 15-ML Ni/Cu(100) compared with a 10-ML Ni film on Cu(100). Based on the close correlation between structure and magnetism found in the Fe/Cu(100) system where a ferromagnetic fcc Fe film is always associated with an enlarged atomic volume, we speculate now that

the strain relaxation in the Ni layer reduces the atomic volume of the Fe surface layer, resulting in the loss of surface ferromagnetism. However, to confirm this assumption, further quantitative measurements such as a full dynamical LEED analysis would be necessary.

C. Anomalous temperature dependence of magnetization and coercive field

For homogeneous bulk ferromagnetic systems, both magnetization and coercivity decrease with increasing temperature. For the bilayers studied here, we find several cases where deviations from this are observed, e.g., dips are present in the M - T curves for 11–13-ML Ni/9-ML Fe/Cu(100) films. Traces of such dips can also be distinguished for similar bilayers with larger Ni thickness (Fig. 5). All of these films show an S-shaped temperature dependence of coercive field (Fig. 6). In addition, for 9-ML Fe/10-ML Ni/Cu(100) and 9-ML Fe/15-ML Ni/Cu(100), the magnetization was found to decrease at low temperature (Fig. 8 and Fig. 10). In all the cases the deviation are observed to start upon cooling below 200 K. Since it is impossible for material to lose its magnetic order with decreasing temperature, one can speculate whether these anomalous phenomena are related to the reorientation of the film magnetization, which results from the different temperature dependence of competing anisotropies. This mechanism leads to the magnetization flip from the perpendicular to in-plane orientation upon decreasing temperature for the Ni/Cu(100) films.^{38,39} However the flip can only be observed within a very narrow Ni thickness interval around 7.4 ± 0.3 ML. This implies that the in-plane and perpendicular anisotropies are very sensitive to thickness. They are of comparable magnitude only within this narrow thickness range. Since we observe the anomalous temperature dependence in a number of films whose Ni layer thickness differs considerably, the same mechanism in the Ni layer of the bilayers as in the Ni/Cu(100) system cannot be used to explain the observed anomalous behaviors measured here. In addition, neither 3-ML nor 9-ML Fe films on Cu(100) shows such anomalous behavior (Fig. 1). Therefore it can be inferred that the anomalous phenomena arise from the coupling of the Ni and Fe films. There are several mechanisms which could account for the observed temperature dependence.

First of all, Li *et al.* have deduced a Néel temperature of 200 K from MOKE data for Fe film with a thickness of 5–11-ML Fe on Cu(100).³¹ In this study they observed a temperature-dependent oscillation of magnetization with thickness, which was explained by an antiferromagnetic coupling in the interior of the Fe film below 200 K. Such an onset of antiferromagnetic coupling in the interior of the Fe film could also explain why we observe anomalies in the temperature dependence of the magnetization for the Fe/Ni bilayer around 200 K. Unfortunately, up to now, no direct experimental evidence for a Néel temperature of 200 K for Fe film has been found. On the contrary, all experiments to measure the transition temperature of fcc Fe determine a Néel temperature around 70 K.^{30,11,56} Hence, we have to exclude this explanation based upon the experimental data available.

Then one can wonder if interdiffusion at the Fe/Ni or Fe/Cu interface could substantially alter the magnetic coupling. Interdiffusion is a likely candidate for modified magnetic properties as can be inferred from data for various Fe-Ni bulk alloys, which show pronounced changes of magnetic coupling with composition. Even for Fe on Cu(100) interdiffusion has been reported at temperature as low as 300 K.⁵⁵ However, in this study it was found that interdiffusion is very pronounced for ultrathin Fe films (2 ML), while 6 ML of Fe could be heated to 420 K without Cu diffusion to the film surface. Since we do not anneal our samples above 300 K and find peculiarities in the temperature dependence always around 200 K irrespective of the thickness of the Fe film (3 or 9 ML) and for different Ni film thicknesses, we believe that interdiffusion is not very likely to explain the observed phenomena at least for the thicker films, even though we cannot completely exclude it.

In our opinion the most attractive explanation combines the ferromagnetic order at both the Fe interface and the Fe film surface with a temperature dependent oscillatory coupling within the Fe film. In this scenario we would, for example, in the case of 11–13-ML Ni/9-ML Fe/Cu (100), propose that the Fe/Ni interface couples ferromagnetically, but a ferromagnetic contribution at temperatures below 200 K also comes from the Fe/Cu interface. These two ferromagnetic portions of the film are coupled. Since the coupling will oscillate with the thickness of the film between the two ferromagnetic (FM) films, we expect an oscillatory behavior of magnetization. This would nicely explain the data of Li for Fe on Cu(100), but with a somewhat modified interpretation: 200 K would now be the Curie temperature of the Fe/Cu(100) interface. This FM portion couples via an oscillatory exchange coupling with the ferromagnetic Fe film surface, leading to an oscillatory magnetization with Fe film thickness. Such an interpretation would resolve the present controversy between Mössbauer data and MOKE experiments. In this scenario the interior of the film should have a Néel temperature around 70 K, as determined by Mössbauer spectroscopy, a Curie temperature of 200 K for the Fe/Cu interface, and a Curie temperature around 270 K for the Fe film surface, both in line with the MOKE data. This sequence of transition temperature is very plausible and supported by theoretical calculations for Fe/Cu(100),⁵⁷ which show that the magnetic coupling of the Fe atoms is strongest at the film surface and weakest in the film interior. Even though this explanation can hence resolve the above-mentioned controversy, further experimental work is necessary to confirm this scenario for both Fe/Cu(100) and Fe/Ni bilayers on Cu(100).

V. SUMMARY

The magnetic properties of Fe/Ni bilayers on Cu(100) have been studied using MOKE. The Fe/Ni interface has a pronounced influence on the spin reorientation transition. The Ni thickness necessary for the perpendicular spin orientation is consistently increased for all bilayer systems studied here, compared with the Ni/Cu(100) system. This is indicative of an in-plane anisotropy of the Fe-Ni interface. Magnetic live Fe layers are observed at the Fe-Ni interface for

9-ML Fe films, even though the inner part of the Fe film is paramagnetic. Peculiar phenomena are observed in the bilayer concerning the temperature dependence of magnetization and coercivity. The possible mechanism for these temperature-dependent anomalies has been discussed. A new scenario has been proposed to explain the observed anomalies. In this scenario the atoms at the Fe/Cu interface are also ferromagnetically ordered but with a lower Curie temperature of about 200 K, and this part of film couples via an

exchange coupling with the other FM portion, i.e., the surface live layer.

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