Magnetic susceptibility measurements near the multicritical point of the spin-reorientation transition in ultrathin fcc Fe(111)/2 ML Ni/W(110) films

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Iron films grown on a two-monolayer Ni/W(110) substrate undergo a temperature-driven spin reorientation which can be followed experimentally from a low-temperature, perpendicularly magnetized monodomain state all the way to the high-temperature paramagnetic state without a significant change of the atomic structure. Measurements of the magnetic susceptibility, using the magneto-optic Kerr effect, give the entire temperature vs thickness phase diagram for the spin reorientation, including measurements close to the multicritical point where the reorientation and Curie temperatures are equal. The technique is particularly sensitive to the formation of the perpendicularly magnetized domains associated with the spin reorientation and reveals that films with a thickness in the neighborhood of the multicritical behavior also display a maximum in the temperature at which domain formation begins. Thinner films undergo the transition from ferromagnetic-to-paramagnetic behavior directly from the domain state, but no signature of the critical temperature itself is seen in the magnetic susceptibility. [S0163-1829(97)03937-4]

I. INTRODUCTION

The spin-reorientation transition is a fascinating magnetic ultrathin-film phenomenon which depends on the interplay of perpendicular magnetic anisotropy, spin fluctuations, and domain formation. Following an early prediction of Néel,[1] experiments have shown that the reduced symmetry of an ultrathin film can lead to a magnetocrystalline anisotropy which favors a perpendicular orientation of the magnetic moment and is even able to overcome the shape anisotropy in very thin films. Numerous studies[2] have further shown that as the film thickness is increased at constant temperature, the relative magnitudes of the magnetocrystalline and shape anisotropies are reversed, leading to a reversal of the sign of the perpendicular anisotropy, so that the magnetic moment reorients to lie in plane. While the change in the shape anisotropy with film thickness is relatively simple to calculate, the magnetocrystalline anisotropy is usually treated as a phenomenological functional which depends implicitly on the film structure, strain, temperature, and other variables. Thus one may speak loosely of spin-reorientation transitions which are driven by, for example, structural transitions,[3] strain in the film,[4] or temperature.[5–9] The first two categories are examples of the complicated interplay between film structure and magnetism which is of great current interest. The last category is in a sense more fundamental, since the spin reorientation results from thermodynamic considerations within a fixed structure and is inherently related to the role of fluctuations in a two-dimensional magnetic system. As a result, a great deal of effort has been expended on the theoretical description of the temperature-driven spin-reorientation transition.[10–13] It has been possible to study in detail only a relatively few experimental systems showing this behavior.

Pappas *et al.*[5] provided experimental evidence of temperature-driven spin reorientation, using fcc Fe/Cu(001) films grown at low temperature. They found that the temperature at which the reorientation occurred decreased with thickness and that a temperature range $\Delta T \sim 20$ K of negligibly small remanence accompanied the reorientation. The reduced remanence was subsequently shown by spin-polarized electron microscopy to be due to an instability against the formation of small domains[6,10] of perpendicular magnetization below the spin-reorientation temperature T_R and not due to a loss of magnetization.[12] Studies of fcc Co/Au(111) also demonstrated the importance of domain formation in spin reorientation,[14] but these studies were made as a function of film thickness because of the thermal instability of the films. A temperature-driven reorientation is observed for bcc Fe/Ag(001).[7] For this film system, Qiu *et al.*[8] were able to plot a partial temperature vs thickness phase diagram, showing the magnetic order-disorder (i.e., ferromagnetic to paramagnetic) phase boundary at low thicknesses and the spin-reorientation phase boundary at greater thicknesses. The temperature vs thickness phase diagram has also been investigated theoretically. A continuum, mean-field theory of the perpendicularly magnetized domains found three distinct domain structures in the approach to T_R from below.[10] Renormalization group calculations,[11] which neglected domain effects, predicted an intermediate thickness range containing both a maximum in T_R and a multicritical point where the order-disorder and spin-reorientation transition boundaries meet. Unfortunately, there are no measurements in these thickness and temperature ranges.

The present paper reports a study of the magnetic properties of fcc Fe(111) films grown on a two-monolayer (2-ML) Ni(111)/W(110) substrate. This system proves to be an example where a single film may be followed through both the spin-reorientation transition and the subsequent order-disorder transition as the temperature is raised. A combination of structural stability and weak intrinsic magnetocrystalline anisotropy moves the critical Fe film thickness for spin

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reorientation to much lower values than in other systems, so that the Curie temperature remains below the limit of film stability and allows the complete temperature vs thickness diagram to be investigated, including the multicritical region. In this study, the magnetic characterization is accomplished by measuring the magnetic susceptibility of the film. Since this quantity is sensitive to magnetic orientation, domain formation and motion, and critical phenomena, it is a powerful probe of the reorientation transition. The paper is divided into three further sections. In Sec. II, the film structure and method for measuring the magnetic susceptibility are reviewed. Section III contains the experimental results and discussion, and Sec. IV presents conclusions.

II. EXPERIMENTAL METHOD

Use of a Ni(111)/W(110) substrate for the growth of Fe films was undertaken as part of a search for a method to grow fcc Fe films which displayed a simpler growth process than that observed using a Cu(001) substrate.[15] Use of the Ni buffer film results in good wetting and reduces the mixing of Fe into the substrate.[16] Use of the (111) orientation matches the surface plane to the crystallographic transition front for the martensitic transition in bulk Fe, so that the inevitable fcc to bcc transition in the Fe films occurs through a continuous evolution of the surface cell geometry and stacking position, rather than through the complicated faceting and roughening process seen for Fe/Cu(001).[17] Angle-resolved Auger electron spectroscopy and scanning tunneling microscopy[18] confirm that an annealed, 2-ML Ni buffer permits the growth of high-quality fcc Fe, but that a 1-ML Ni buffer gives growth similar to the bcc Fe observed for growth directly on W(110). The first 3 ML of Fe grow in a nearly layer-by-layer fashion on the 2-ML Ni/W(110) substrate.[16] The propagation of strain from the bcc W(110) substrate results in a slightly strained fcc (111) lattice which has twofold in-plane rotational symmetry, similar to the Ni buffer.[19] At an Fe coverage of 4 ML, a surface cell intermediate to fcc and bcc first appears. Other details of film growth and structure have been reported elsewhere.[16]

Previous magnetic studies of Ni/W(110) films using a magnetic resonance technique[20] indicate that a 2-ML film has an in-plane moment with an order-disorder temperature T_C approximately at room temperature. The 2-ML Ni films used in this study showed no order-disorder transition in the susceptibility at temperatures above 200 K. Because the finite-thickness scaling of the Curie temperature is very steep in this thickness range, a small difference in thickness calibration may account for the discrepancy. A preliminary survey[18,21] of the magnetic properties of the Fe/2 ML Ni/W(110) films showed that they are perpendicularly magnetized at and below a coverage of 1 ML. The present measurements of the external magnetic susceptibility $\chi_{\mathrm{ext}} = \partial M/\partial H_{\mathrm{ext}}$ were made using the Kerr effect[22] to detect the variation ∂M upon application of a small external field modulation $\partial H_{\mathrm{ext}}$. An existing dc Kerr apparatus[23] was modified for the ac Kerr effect measurements by incorporating lock-in amplification techniques with special attention to optimization of the signal-to-noise ratio.[24] Stray magnetic fields were eliminated to order 10 mG by three pairs of large, mutually perpendicular coils. *In situ* magnet coils generated a 210-Hz ac field with H_{ext} oriented along one of three mutually perpendicular directions labeled with respect to the W(110) substrate, giving $\chi_{\perp}$, χ_{001}, and $\chi_{\bar{1}10}$. Real and imaginary parts were measured sequentially. Applied field amplitudes were usually 15 and 6 Oe for perpendicular and in-plane measurements, respectively. The optically calibrated measurements gave rms Kerr rotations of order of 1 μrad. Approximate SI magnetic calibrations were made by assuming the magnetization of bulk Fe and comparing the calibrated Kerr rotation to the rotation per monolayer in saturated hysteresis loops.

For low coercive fields, the real (in-phase) part of the susceptibility measures the saturation magnetization, while the imaginary (quadrature) part measures the remanent magnetization or, equivalently, the presence of dissipation through hysteresis.[22] Both components fall off when the coercive field grows much larger than the applied field. At the Curie temperature of an order-disorder transition, the internal susceptibility $\chi_{\mathrm{int}} = \partial M/\partial H_{\mathrm{int}}$ diverges. In thin films, the internal field is itself strongly influenced by the magnetization through the demagnetization field, $H_{\mathrm{int}} = H_{\mathrm{ext}} - NM$, where N is the demagnetization factor.[25] As a result,

$$\chi_{\mathrm{ext}} = \chi_{\mathrm{int}}/(1 + N\chi_{\mathrm{int}}). \quad (1)$$

For an infinite, flat film, $N_{\parallel} = 0$ for $H_{\mathrm{ext}} \parallel M$ in plane, and at an order-disorder transition $\mathrm{Re}\{\chi_{\mathrm{ext}}\}$ diverges to form a narrow peak of order $10^3 - 10^4$ SI units, located close to the critical temperature T_C. $\mathrm{Im}\{\chi_{\mathrm{ext}}\}$ has a peak just below T_C, where the coercive field is less than the applied field and goes to zero when remanence disappears at T_C. The in-plane susceptibility may be measured using the longitudinal geometry for the Kerr effect. For $H_{\mathrm{ext}} \parallel M$ normal to the surface, the polar Kerr effect is used. Since $N_{\perp} = 1$, the divergence of χ_{int} at an order-disorder transition causes $\chi_{\mathrm{ext}} \rightarrow 1/N_{\perp} \approx 1$, so that the measurement is insensitive to the transition. In contrast, far below an order-disorder transition the susceptibility is derived from changes in the domain-averaged magnetization due to domain wall motion which is driven by H_{ext}. The demagnetizing factor in Eq. (1) does not play a role in this process. For perpendicular magnetization, the polar susceptibility due to domain effects does not saturate and, therefore, dominates the total signal. For in-plane magnetization the in-plane susceptibility due to domain effects is dwarfed in the presence of a diverging critical susceptibility. The two measurement geometries are therefore most sensitive to different magnetic phenomena. Measurements of χ are particularly well suited to the study of the spin-reorientation transition, since they are sensitive to domain formation and domain wall motion in perpendicularly magnetized films.

III. RESULTS AND DISCUSSION

Figure 1 presents the real (a) and imaginary (b) parts of the magnetic susceptibilities for all three field orientations, for a 2.25-ML Fe film on 2 ML of Ni/W(110). The measurements are reversible as long as the temperature does not exceed 400 K. The polar susceptibilities have been multiplied by a factor of 70 so that they can be viewed on the same plot as the in-plane measurements. At low temperatures, it is clear from $\mathrm{Im}\{\chi\}$ that the film displays remanence in the polar susceptibility, but not the in-plane susceptibility, and is

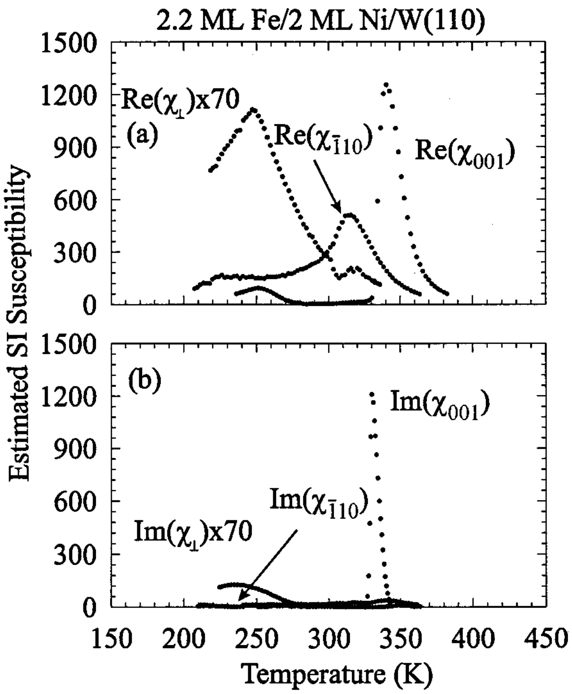

FIG. 1. Magnetic susceptibility of a 2.2-ML Fe film grown on a 2-ML Ni/W(110) substrate. χ is measured for applied fields oriented along three mutually perpendicular directions referred to the surface of the W substrate. The estimated scale assumes the bulk magnetization of iron.

therefore perpendicularly magnetized. Although the magnitude of $\mathrm{Re}\{\chi_{\perp}\}$ is relatively small, it is nonetheless much greater than unity and, taken in conjunction with remanence, must represent the presence of perpendicularly magnetized domains. This is consistent with the broadness of the peak in $\mathrm{Re}\{\chi_{\perp}\}$. To interpret this peak as an order-disorder transition is unphysical, as it requires $N_{\perp} \leqslant 0.05$ in Eq. (1). A perpendicular remanent magnetization is detected in $\mathrm{Im}\{\chi_{\perp}\}$ up to 280 K, after which there is a gap from 280 to 322 K in which no remanent magnetization is detected. At higher temperatures, remanence reappears in plane, as indicated by a peak in $\mathrm{Im}\{\chi_{001}\}$, demonstrating that the spins have reoriented along W[001] or, equivalently, the $[01\overline{1}]$ direction of the strained fcc Fe. Comparison to the low-energy electron-diffraction (LEED) pattern[16] shows that the magnetization lies along the direction of in-plane compressive strain in the films and perpendicular to the direction of in-plane tensile strain. These strains are apparently sufficient to create a uniaxial in-plane anisotropy. The form of χ_{001} conforms to that expected near an in-plane order-disorder transition. $\mathrm{Im}\{\chi_{001}\}$ goes to zero very near to the temperature of the peak in $\mathrm{Re}\{\chi_{001}\}$, giving $T_C = 342 \pm 2$ K. The considerable width [full width at half maximum (FWHM)=25 K] of the latter peak is attributed to the large ac field of 6.0 Oe used for this particular measurement.[26] In the region of zero remanence, there is also a purely real response in $\chi_{\overline{1}10}$, which is discussed later.

In overview, it is clear that a spin-reorientation transition occurs at an Fe film thickness of only ≈ 2 ML, whereas at least 4–6 ML are required for Fe/Ag(001) (Refs. 7 and 8) and Fe/Cu(001).[5–7] This indicates that the perpendicular magnetic anisotropy of this system is rather weak and suggests that there is some cancellation of the contributions from the three surfaces involved (UHV/Fe, Fe/Ni, and Ni/W). This seems reasonable, as Ni/W(110) films have negative (i.e., favoring in-plane) surface anisotropy and the positive surface anisotropy of Fe/Cu(111) is roughly half as large as that of Fe/Cu(001) or Fe/Ag(001).[27] Because the Fe film is thinner when reorientation occurs, the Curie temperature is relatively low and allows the spin-reorientation and order-disorder transitions to be observed sequentially on the same film without altering the metastable structure. The detailed behavior of the susceptibilities can be interpreted using the domain model of Abanov *et al.*[10] They calculated a reduced (i.e., unitless) magnetization m, which results from the difference in the spin-up and spin-down domain widths upon the application of a reduced external magnetic field strength h. In this mean-field domain model, the external polar susceptibility in the limit of small h is

$$\chi_{\perp} = \partial m/\partial h = [(\pi/4)\Omega n^*(T)]^{-1}, \quad T < T_R, \tag{2}$$

where Ω is the dimensionless scale of the dipole interaction energy and $n^*(T)$ is the linear density of domains. The authors show that near the spin-reorientation temperature, as the perpendicular anisotropy approaches zero, $n^*(T)$ grows exponentially, giving a temperature range ΔT of complicated domain patterns which exhibit greatly reduced remanence. Figure 1 gives $\Delta T \approx 40$ K, in rough agreement with measurements using small fields on other Fe film systems.[5,7,8] Equation (2) shows that $\mathrm{Re}\{\chi_{\perp}\}$ decreases as the domain density increases in the approach to T_R, in agreement with Fig. 1. At lower temperatures, the model predicts a single-domain state. At sufficiently low temperature, the experimental $\mathrm{Re}\{\chi_{\perp}\}$ is governed by the coercive field and increases with temperature. A peak in $\mathrm{Re}\{\chi_{\perp}\}$ is formed at the intersection of these two limiting behaviors and should be a reliable, although approximate, indicator of the formation of the domain phase from the single-domain state.

The purely real response in $\chi_{\overline{1}10}$ near the spin reorientation is likely related to imperfections in the 2-ML Ni buffer. LEED (Ref. 16) results indicate that Fe grown on a 1-ML Ni buffer is poorly ordered, and scanning tunneling microscopy[28] images show that the growth morphology is similar to that seen for bcc growth directly on W(110). Figure 2(a) shows $\mathrm{Re}\{\chi\}$ measured for a 1.5-ML Fe film grown on a 1-ML Ni buffer. $\mathrm{Re}\{\chi_{\overline{1}10}\}$ indicates an in-plane magnetization along $\mathrm{W}[\overline{1}10]$, as is seen for Fe grown directly on W(110).[29] The width of the peak at the order-disorder transition at 345 K is consistent with Eq. (1) with $N_{\parallel} \approx 0$ and the smaller ac field modulation of 2.0 Oe used in this experiment. Data for Fe grown on 1.5 ML Ni (not shown) reveal simultaneous remanence along the surface normal and $\mathrm{W}[\overline{1}10]$, consistent with distinct magnetic behavior on the regions of 1 and 2 ML Ni. Although use of an ideal 2-ML Ni buffer should result in only fcc Fe, scanning tunneling microscopy images[18] of a 2-ML Ni buffer show small regions of size <10 nm of 1- and 3-ML Ni thickness, comprising about 5% of the film. We speculate that Fe grown on 1 ML Ni in these regions may continue to give a weak, nonremanent response in $\mathrm{Re}\{\chi_{\overline{1}10}\}$ when surrounded by the predominant fcc Fe portion of the film. As can be seen in Fig. 1, this response displays a broad peak just below T_R. Apparently,

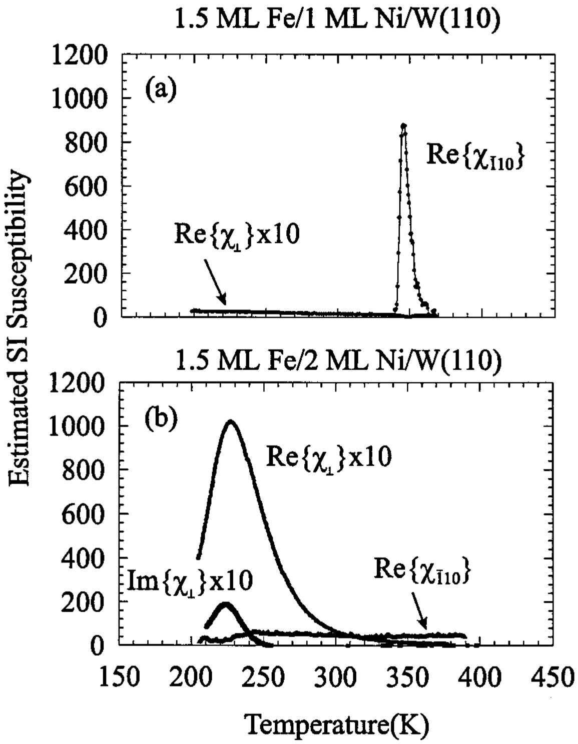

FIG. 2. (a) Magnetic susceptibility of a 1.5-ML Fe/1-ML Ni/W(110) film indicates in-plane magnetization along W[$1\bar{1}0$]. (b) Magnetic susceptibility of a 1.5-ML Fe/2-ML Ni/W(110) film indicates normal magnetization at low temperatures and no spin reorientation. An order-disorder transition from the perpendicularly magnetized state would produce a maximum Re$\{\chi_\perp\}$ of 1 and would not be detected.

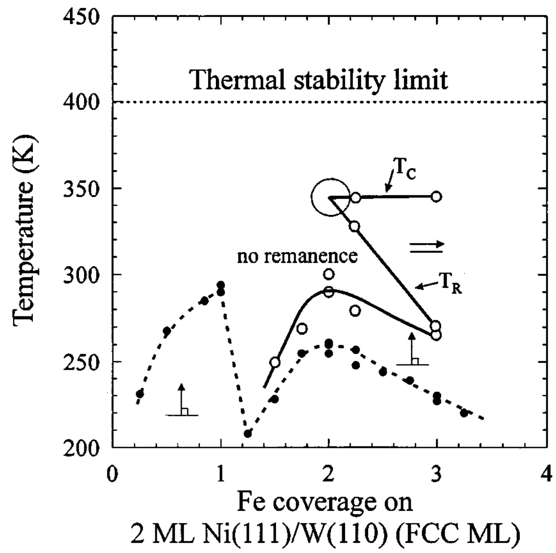

FIG. 3. Schematic phase diagram of the spin-reorientation transition in Fe/2 ML Ni/W(110). The solid dots connected by a dashed line are the peak positions of Re$\{\chi_\perp\}$ and are a rough marker of the formation of the perpendicularly magnetized domain phase. The open circles connected by a solid line are locations where the imaginary part of either the perpendicular or in-plane susceptibility becomes zero. The boundaries of the in-plane phase are labeled as the reorientation temperature T_R and Curie temperature T_C. Regions of the diagram are marked as having perpendicular, in-plane, or no remanence. A circle indicates the multicritical region where the perpendicular, in-plane, and paramagnetic phases meet.

the properties of these small regions of the film become influential close to T_R where the direction of magnetization is not otherwise well defined.

Figure 2(b) presents magnetic susceptibilities measurements for 1.5 ML of Fe on a 2-ML Ni buffer. Comparison with Fig. 1 shows that the low-temperature peak in $\chi_\perp$ associated with the domains of perpendicular magnetization is present, but that there is no signature of the in-plane reorientation or the order-disorder transition in χ_{001}. Since domain formation has begun, but spin reorientation has not yet occurred, the order-disorder transition in this film must proceed directly from the perpendicularly magnetized domain phase. The Curie temperature cannot then be detected by measurements of $\chi_\perp$, as was discussed earlier. Even if a well-defined Curie temperature exists, the demagnetization factor $N_\perp=1$ in Eq. (1) produces only a very broad, small peak in the *external* susceptibility and the measurements are insensitive to the transition.[30] The fact that remanence is detected in Im$\{\chi_\perp\}$ makes it clear that the Fe is not antiferromagnetic, as has been found for Fe/Cu(001) films grown at room temperature,[3] since the fcc (111) surface of antiferromagnetic Fe exposes equal numbers of sites of each spin orientation.

The absence of the spin-reorientation transition at low coverages is supported by the systematic dependence of the magnetic susceptibility on the Fe thickness, as summarized in Fig. 3. Measurements for all three applied field orientations were performed at thicknesses of 1.5, 2.0, 2.25, 3.0, and 4.0 ML, and individual susceptibility components were measured at other thicknesses. Solid dots represent the peak positions of Re$\{\chi_\perp\}$, and the dashed lines connecting them are guides to the eye. Open circles represent the points where remanence vanishes or appears and are joined by a solid line. The regions on the diagram are labeled according to whether perpendicular, in-plane, or no remanence was observed in Im$\{\chi\}$. For Fe thicknesses $2<x\leqslant 3$ ML, $T_C>T_R$, and the order-disorder transition proceeds from the reorientated phase. Thus the solid line indicating the appearance of in-plane remanence in Fig. 3 in this region may be labeled as giving T_R, and the solid line indicating the disappearance of in-plane remanence may be labeled as giving T_C. Three markers of the spin-reorientation process—the temperature where in-plane remanence appears, the upper limit of perpendicular remanence, and the peak in Re$\{\chi_\perp\}$—all *decrease* with increasing thickness in this regime. For $1<x\leqslant 2$, the order-disorder transition occurs from the perpendicular domain phase and, since it cannot be observed in $\chi_\perp$, the line giving T_C cannot be shown. However, in this thickness range, the two markers of the spin-reorientation process *increase* with increasing thickness. Since the spin-reorientation transition is not observed for $x=2$ and first seen at $x=2.25$, the multicritical point of the system must be at values intermediate to these and at approximately 335 K, as indicated by a circle in Fig. 3. The data in Fig. 1 are therefore an observation of the magnetic susceptibility in the neighborhood of the multicritical point where the spin-reorientation transition and the order-disorder transition lines meet. It is interesting to note that this occurs at the thickness where the markers of the spin-reorientation transition have maxima, in

agreement with the prediction of Politi *et al.*[11] This behavior is at least consistent with the partial phase diagram of Fe/Ag(001) reported by Qui *et al.*,[8] where only the regions removed from the multicritical point were investigated.

At 1 ML Fe, there is a sudden change in the temperature at which the peak in Re$\{\chi_\perp\}$ occurs, suggesting that films less than 1 ML thick form a qualitatively distinct region. For submonolayer Fe films, Re$\{\chi_\perp\}$ (not shown) again has a broad peak consistent with domain wall motion. Since the films have perpendicular remanence down to a thickness at least as small as 0.25 ML Fe, the Ni moments in the 2-ML buffer must be actively participating in the formation of the perpendicular magnetization (particularly when the Fe islands are below the percolation limit). It is remarkable, but not unique,[31] that such a small coverage can greatly alter the magnetic properties of a film. Taken together, these observations suggest that properties of submonolayer Fe films are determined by the formation of the Fe/Ni interface. Monolayer Fe islands on a Ni film are a reasonable approximation to ultrathin fcc (111) films of alloys made of equivalent amounts of Fe and Ni, which have been shown to have a perpendicular magnetization.[32] The fact that the peak position of Re$\{\chi_\perp\}$ changes very close to 1 ML Fe tends to confirm that the films grow with good layer completion and suggests that the magnetic behavior of the thicker films is dominated by the Fe overlayer, and not by the interface. It is not clear whether or not the order-disorder transition temperature T_C also shows an abrupt change at 1 ML Fe. Further experiments are required to examine this region of the phase diagram.

IV. CONCLUSIONS

In summary, measurements of the ac magnetic susceptibility of fcc Fe/2 ML Ni/W(110) films have allowed a systematic investigation of the spin-reorientation process and produced a complete, schematic temperature vs thickness phase diagram. For thicker films, there are experimental markers for the perpendicular phase, the formation of the perpendicular domain phase, a reorientation to the in-plane phase, and the order-disorder transition from the in-plane phase to the paramagnetic phase. For thinner films, the order-disorder transition proceeds directly from the perpendicular domain phase and there is no clear marker of the Curie temperature in the magnetic susceptibility. Measurements indicate that the multicritical point where the in-plane, perpendicular, and paramagnetic phases meet is at a thickness between 2 and 2.25 Fe ML and a temperature of about 335 K. The film thicknesses in the multicritical region where $T_R \approx T_C$ display a temperature maximum for the formation of the domain phase. There is evidence that submonolayer Fe films form a distinct region where the magnetic properties are governed by the formation of the Fe/Ni interface. This film system is well suited to further studies of the details of spin reorientation, including a search for experimental evidence of domain melting phases close to reorientation, the nature of and universal behavior in the multicritical region, and the description of the transition from the perpendicular domain phase to the paramagnetic phase. Furthermore, the magnetic properties of films $\geqslant$4 Fe ML, where a rather continuous fcc to bcc transition occurs, have yet to be investigated. Finally, the present studies show that measurements of the magnetic susceptibility are a useful, and more widely accessible, complement to spin-polarized microscopy for observing spin reorientation.

ACKNOWLEDGMENTS

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