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S.D. Bader, Dongqi Li, and Z.Q. Qiu‡

Materials Science Division

Argonne National Laboratory, Argonne, IL 60439

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6th Joint Magnetism and Magnetic Materials - Intermag Conference, Albuquerque, NM, June 20-23, 1994

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*Work supported by the U.S. Department of Energy , Basic Energy Sciences-Materials Sciences, under contract #W-31-109-ENG-38.

‡Permanent address: Physics Department, University of California, Berkeley, CA.

MAGNETIC AND STRUCTURAL INSTABILITIES OF ULTRATHIN Fe(100) WEDGES* (INVITED)

S. D. Bader, Dongqi Li, and Z. Q. Qiu†

Materials Science Division, Argonne National Laboratory, Argonne, IL 60439.

An overview is provided of recent efforts to explore magnetic and related structural issues for ultrathin Fe films grown epitaxially as wedge structures onto Ag(100) and Cu(100). Experiments were carried out utilizing the surface magneto-optic Kerr effect (SMOKE). Ordinary bcc Fe is lattice-matched to the primitive unit cell of the Ag(100) surface. Fe wedges on Ag(100) can be fabricated whose thick end has in-plane magnetic easy axes due to the shape anisotropy, and whose thin end has perpendicular easy axes due to the surface magnetic anisotropy. A spin-reorientation transition can thus be studied in the center of the wedge where the competing anisotropies cancel. The goal is to test the Mermin-Wagner theorem which states that long-range order is lost at finite temperatures in an isotropic two-dimensional Heisenberg system. Fe wedges on Cu(100) can be studied in like manner, but the lattice matching permits fcc and tetragonally-distorted fcc phases to provide structural complexity in addition to the interplay of competing magnetic anisotropies. The results of these studies are new phase identifications that help both to put previous work into perspective and to define issues to pursue in the future.

* Work supported by US DOE BES-MS under contract W-31-109-ENG-38.

† Permanent address: Physics Dept.; University of California, Berkeley.

I. Introduction:

It is of interest to explore magnetic instabilities associated with competing anisotropies in ultrathin epitaxial films. A simple approximation is to equate the two dominant contributions to the anisotropy energy density:

$$\frac{2K_s}{t} = 2\pi M^2 \ .$$

This provides the condition for which the 'shape' anisotropy ($-2\pi M^2$) balances out the surface anisotropy ($2K_s/t$), where K_s is the surface anisotropy constant, t is the thickness of the magnetic film, and M is the magnetization. The underlying assumptions are that any other anisotropy terms can be ignored, *and* that the magnetization orientation within the film is uniform (*i.e.* all spins point in the same direction). The critical thickness for spin reorientation is :

$$t_R = \frac{K_s}{\pi M^2} \ .$$

For $t<t_R$ the easy axis of magnetization is perpendicular to the film plane (*i.e.* vertical), while for $t>t_R$ the easy axis is in-plane. The question is what happens in the vicinity of $t=t_R$?

Recent theoretical treatments and ideas can be summarized. Firstly, in an isotropic two-dimensional (2D) Heisenberg system there is no long-range order at finite temperature. This is known as the Mermin-Wagner theorem.[1] Hence, spin melting might be expected to occur in the vicinity of t_R. Pescia and Pocrovsky[2] used a renormalization group approach to consider thermal fluctuations, and concluded that the spin reorientation occurs as a phase transition at a temperature T_R which can be less than the Curie temperature T_C. They used the idea of Jensen and Bennemann[3] of a transition driven by the entropy of disorder on going from a uniaxial to a planar spin configuration as temperature increases. Morr, Jensen

and Bennemann[4] recently used a Greens function approach within the random phase approximation to phenomenologically describe the loss of long-range order in the vicinity of the spin-reorientation phase transition. However, it is known that even arbitrarily small anisotropies can restore long-range order.[5] Therefore, it also may be necessary to explore the additional anisotropies, such as arise from the bulk or from finite-size effects, or from higher-order terms in the expansions of the anisotropies than the dominant terms that vary as the square of the magnetization. The higher-order terms can manifest themselves in the vicinity of t_R where the dominant anisotropy terms vanish. Experimentally, ultrathin magnetic films are always grown on a substrate for which lattice constants and thermal expansion coefficients tend to be mismatched to some degree. Thus, epitaxial strain also can influence the magnetic surface or interface anisotropy. This has been treated recently for a test case, for example, by Victora and MacLaren[6] who used electronic structure calculations.

Alternatively, it also becomes important to explore non-uniform magnetization configurations. For example, Yafet and Gyorgy[7] considered the conditions necessary to stabilize ferromagnetic stripe domains in an atomic monolayer that possesses strong surface magnetic anisotropy. They found that while the short-range part of the dipolar interaction gives rise to the familiar $-2\pi M^2$ shape anisotropy term, the long-range part, which can be represented as a domain wall-wall interaction, can result in a lower energetic state than the uniformly magnetized case. The wall-wall interaction, however, has to be evaluated explicitly for the domain configuration under consideration. The domain size will reach an experimentally observable magnitude in the vicinity of the spin-reorientation transition. Thieville and Fert[8] treated twisted spin configurations using a micromagnetic approach. Alternatively, Erickson and Mills[9] reached similar conclusions for canted spin structures by considering spinwave excitations in 2D films. They found that the excitation energy becomes imaginary in a gap region near the spin-reorientation transition, implying that there exists a static spin-density wave in real space in this region (*i. e.* the stripe domain

structure). A striped domain structure in a 2D film can be viewed as a 1D ordered system. It is well known that 1D ordered states are unstable against thermal fluctuations. Indeed, Kashuba and Pokrovsky[10] examined 2D striped domains by means of renormalization group methods and found that while thermal fluctuations destroy the regular *spatial* distribution of the stripe domains predicted by Yafet and Gyorgy[7] for the T=0 case, the *orientation* of the domain walls remains stable in the presence of thermal fluctuations. Therefore, they reached the conclusion that the 2D stripe domains can be mapped onto the smectic liquid-crystal case.

It is of interest to discuss in more detail the issue of a film of fixed thickness that undergoes a temperature-dependent spin-reorientation transition from perpendicular at low T ($T<T_R$) to in-plane at high T ($T>T_R$). The question arises as to the existence of a temperature gap ΔT for which either long-range order disappears or for which a uniform magnetization configuration is unstable with respect to canting, twisting or domain formation. Erickson and Mills roughly estimate a value of $\Delta T/T_R \sim 0.5\%$ for an ultrathin Fe film. A gap due to domain formation can be appreciably larger in magnitude than this estimate. This is because the gap would signal the T range over which the scale of the domain structure shrinks to dimensions that are less than the sample size or probing length.

Much has been said about 2D spin-reorientation phenomena so far, but perhaps the most fascinating aspect to appreciate is that it is predicated on the existence of perpendicular surface anisotropy that is strong enough to compete with the shape anistropy of the film. For Fe the shape anistropy gives rise to an ~20kG demagnetizing field. Thus, the surface anisotropy must be quite substantial in magnitude. Néel[11] was the first to recognize that unusually strong surface anisotropies relative to bulk anisotropies can exist. The bulk magnetocrystalline anisotropy was first attributed to the spin-orbit interaction by Van Vleck.[12] Néel recognized that strong spin-orbit interactions could arise from the broken symmetry at the surface. Much more recently Gay and Richter[13] performed the first computational study to confirm that strong surface anisotropies can be anticipated in

particular systems. Wang, Wu and Freeman[14] have contributed the most recent state-of-the-art advances to this demanding approach.

Gradmann[15] was the first to experimentally identify perpendicular easy axes attributed to strong surface anisotropy. He recently reviewed his work that dates back to the 1960's on a variety of films, in an outstanding book chapter. Jonker *et al.*[16] reawakened interest in the surface anisotropy issue in 1986. They attributed the non-observation of spin-polarization in photoemission experiments on Fe/Ag(100) films as being due to the existence of vertical easy axes, and to the technical constraint that the spin-detectors employed in their experiments were only sensitive to in-plane magnetization components. Recent work on surface magnetic anisotropy in ultrathin films have been reviewed in a variety of publications.[17]

Another experimental manifestation of strong surface anisotropies can be observed in enhanced coercivity (H_c) values displayed in the ultrathin regime. The earliest studies of this type are due to Hirsch[18] who observed a factor of ~5 enhancement in H_c for ultrathin Fe interleaved with Cu. Bader[19] documented a number of systems for which H_c peaks in the monolayer region. Engel *et al.*[20] have observed H_c peaks in monolayer-range films that occur as a function of the thickness of a nonmagnetic overcoating layer. This can be due to electronic effects or morphological (*i.e.* wetting) changes in the magnetic layer. Gradmann has documented similar examples of coercivity and anisotropy changes due to nonmagnetic overcoats.[15]

The first experimental identifications of temperature-dependent 2D spin-reorientation transitions in transition-metal films are due to Pappas, Kämper, and Hopster.[21] They studied both Fe/Ag(100) and Fe/Cu(100). A ΔT range of ~20-30K was identified that had vanishingly small remanence. Allenspach and Bischof[22] subsequently imaged the magnetic domains of Fe/Cu(100) and observed striped domains with smectic liquid-crystal-like orientational order, as described by Kashuba and Pokrovsky.[10] However, the question is still not answered as to whether the spontaneous magnetization,

as opposed to the average magnetization, is at all reduced or not in the immediate vicinity of the spin-reorientation transition.

In the present work we describe recent SMOKE contributions to this fundamental problem in 2D magnetism. We first examine Fe/Ag(100) for which magnetic instabilities can be studied without concern for structural instabilities, since Fe is well lattice matched to the Ag(100) square net. Then we progress to the more complex case of Fe/Cu(100) which can exhibit similar spin-reorientation behavior to Fe/Ag(100), but also possesses structural instabilities.

Fcc Fe(100) is lattice matched to Cu(100). In the ultrathin limit a tetragonally distorted fcc structure is stable. This fct structure relaxes to an undistorted fcc structure upon increasing the Fe thickness.[23] The fct phase is itself unusual for an element. Only elemental Sn has a naturally occurring fct phase. In the case of Fe/Cu(100) the fct structure is stabilized epitaxially. (This is one of the attributes of modern thin-film-magnetism research - the ability to artificially stabilize new phases.) Eventually, thicker Fe films grown on Cu(100) transform to the equilibrium bcc structure. Bcc(110) epitaxial relationships to fcc(100) have been described elsewhere.[24] Metastable phases, therefore, abound in Fe/Cu(100) as a function of growth temperature and Fe thickness. The fct spin-reorientation transition, for example, is only found for low-T growth ($T \leq 200K$). At higher temperatures the fct structure relaxes to fcc and the magnetic properties change dramatically, as studied recently by Thomasen *et al.*[25] and Li *et al.*[28] Thus, Fe/Cu(100) provides a system rich in magnetic and structural instabilities. The interplay of these instabilities has intrigued, challenged and stymied a whole generation of modern researchers. An enormous collective effort has been devoted to this system. It is the prototype chosen by many who seek a rite of passage into the realm of surface magnetism.

II. Experimental Details:

Background information on the sample preparation, characterization and magneto-optic-measurement techniques appear elsewhere.[26,27,28] For the sake of completeness we briefly mention that the Fe samples were prepared by ultrahigh vacuum (UHV) evaporation, also known as molecular beam epitaxy (MBE). The Fe wedges are formed by linear translation of the substrate behind a mask during growth. Evaporation rates are in the range of 1 monolayer (ML) every ~2-3 min. The wedges have a slope of ~0.2ML/mm for Fe/Ag and ~1.5ML/mm for Fe/Cu. The slopes are substantially larger for the Fe/Cu wedges in order to scan the broader region of interest that includes a range of the fct, fcc and bcc phases. The crystals were ~1cm in length, and the laser beam for the SMOKE studies was focused to ~0.2mm. Thus ~50 discrete positions could be sampled along a wedge. The substrates were prepared utilizing standard surface-science techniques involving sputter-anneal cycles. Low- and high-energy electron diffraction (LEED and RHEED) were used to characterize the substrates and films. RHEED oscillations during Fe growth are published in Refs. 27 and 28.

Magneto-optic Kerr-effect measurements were made at the He-Ne laser line using *p*-polarized light. The films were magnetized using a split-coil superconducting solenoid that provides a homogeneous field with low trapped flux at zero current (<10 Oe). Hysteresis loops were generated with no attempt to convert to ellipticity units. Ellipticities are reported elsewhere for related film structures.[29] The important point for the present studies is that the signal is proportional to the magnetization. The remanent magnetization M_R was used to track the phase transitions in order to avoid possible field-induced transitions. The hysteresis loops are used to identify the easy axis of magnetization. Square polar loops identify vertical easy axes, while square longitudinal loops identify in-plane easy axes. To obtain polar or longitudinal signals the sample was positioned appropriately within the solenoid to face one of two orthogonal laser stations used to generate the data. Thus, it was necessary to rotate the sample by 90° to obtain data for both

configurations. This introduced an absolute uncertainty in the positioning of the beam along the wedge, but it presented no obvious problem for our purposes. The hysteresis loops permitted the H_c values to be obtained. They were used to identify the structural transitions for Fe/Cu and to augment the spin-reorientation studies for both systems.

III. Results:

A. Fe/Ag(100):

The spin-reorientation transition for Fe/Ag(100) is shown in Figs. 1(a) and 2. Figure 1(a) displays the transition as a function of temperature for a fixed Fe thickness. This is the manner in which the transition was studied originally by Pappas, *et al.*[21] who used polarized electron scattering to identify a region ΔT with vanishing magnetization. The high signal-to-noise ratio and data density in Fig. 1(a) enables one to discern an asymmetric ramp structure within the pseudogap encompassed by ΔT. Thus, if the remanence vanishes, it does so over a very limited T-range (a few Kelvin at most), as estimated by Erickson and Mills. Note that M_R in Figs. 1(a) and 2 are normalized to the film thickness, and the longitudinal and polar M_R values at saturation are normalized to each other for clarity. (The polar signal is ~25 times as intense as the longitudinal signal, as expected.) The question that Fig. 1(a) raises is what is the root cause of the remanence decrease within ΔT? Is long-range ferromagnetic order lost anywhere within this region, as might be anticipated from the Mermin-Wagner theorem? How do domains or other non-uniform spin configurations manifest themselves within ΔT? If long-range order does not vanish, do enhanced thermal fluctuations in 2D suppress the magnetization even partially due to the system becoming more isotropic in the vicinity of T_R? These questions should motivate future progress in the field.

Figure 2 provides similar data to Fig. 1(a) but as a function of Fe thickness along a wedge. The region Δd and the point d_R are defined operationally in an analogous manner to ΔT and T_R in Fig. 1(a). The asymmetric ramp structure within Δd is very similar in

appearance to that observed in Fig. 1(a) within ΔT. The same questions apply as outlined above. It is interesting to note that Kashuba and Pokrovsky[10] have found that their calculations linearize to first-order expansion in both T and d in the vicinity of T_R and d_R, as observed experimentally in Figs. 1(a) and 2.

To augment Fig. 1(a) further, we show the corresponding H_c data in Fig. 1(b). The transition at T_R is defined by a peak in H_c which is almost 3-times as large as its value at $(T_R-\Delta T)$. For $T>T_R$ the value of H_c drops precipitously because the vertical axis becomes a hard direction for the magnetization. For $T<(T_R-\Delta T)$, H_c decreases substantially as T increases. Thus, the hysteresis loops are shrinking in area in this interval as T increases. This can be due to enhanced thermal fluctuations as T_R is approached, or to the fluidity of the domain structure. The peak in H_c at T_R, in any case, represents a striking observation and a challenge to interpret.

Figure 3 summarizes results for the spin-reorientation phase-boundary determinations for Fe/Ag(100). The data represent measurements taken along wedges at different temperatures. The pseudogap regions are not indicated in Fig.3, but Curie temperatures T_C are shown for some initial Fe thicknesses. The Curie transitions are found to be 2D-Ising-like, as described in Ref. 27. The Curie transitions can be examined to provide an estimate of the intrinsic width that might apply also to the spin-reorientation transitions. The 3-5% tails reported at T_C imply that the correlation length is limited by finite-size effects to ~100Å, which corresponds to a typical terrace width in a metallic single crystal.[27] Thus, with inclusion of the Curie transitions, Fig. 3 delineates the M=0, $M_{\parallel}$ and $M_{\perp}$ phase regions.

B. Fe/Cu(100):

The region of stability of vertical easy axes for Fe/Cu(100) was first explored systematically by Liu *et al.*[30] They constructed a phase diagram to represent the metastability of the system by plotting growth temperature *vs.* Fe thickness for a large

number of films. Vertical easy axes were observed in a region bounded by a thickness of ~6ML Fe and growth temperatures that do not extent very much above room temperature. The onset of ferromagnetic signals for the thinnest films occurred between ~1-3ML. Figure 4 shows a somewhat different mapping than that of Liu *et al.*[31] for one of our wedged samples grown at low temperature. Figure 4 is analogous to the plot of the Fe/Ag(100) data in Fig. 3. The left boundary in Fig. 4 represents the onset of detection of ferromagnetic signals with vertical easy axis (*i.e.* T_C), while the right boundary represents the termination of the vertical ferromagnetic phase (*i.e.* T_R). The left boundary is believed to be influenced by the non-ideal morphology of the films (*i.e.* intermixing) as well as by decreased T_C values for decreasing film thickness.

Subsequent to the work of Liu *et al.*[31] interest focused on the region beyond the right boundary of Fig. 4. It became clear that two phases exist in this region depending on growth temperature. For low temperature (~100K) growth the spin-reorientation transition ensues, as for Fe/Ag(100). This occurs even if the samples are annealed to the vicinity of room temperature after growth, as in the work of Ref. 21. For example, the spin-reorientation is shown in Fig. 5 for a wedge grown at 190K, annealed at room temperature, and measured at 110K. However, different results are obtained for room-temperature growth. Xhonneux and Courtens[31] found evidence for a non-magnetic phase beyond the 6-7ML Fe phase boundary. Thomasen *et al.*[25] further clarified the new phase identification as having a surface ferromagnetic 'live' layer with vertical easy axis. Li *et al.*[28] more recently confirmed the work of Thomasen *et al.*[25] and, furthermore, found evidence that the ferromagnetic surface is the termination of an antiferromagnetic (AF) phase. Antiferromagnetism does not manifest itself directly in SMOKE measurements, so further explanation is necessary. While Ref. 25 observed a constant magnetization in the region of interest, Li *et al.*[28] found that upon cooling the constant signal developed peaks of monolayer-type amplitude. For an AF structure built up of alternating ferromagnetic (100)

sheets, a peak would arise due to an uncompensated ferromagnetic sheet whenever there is an odd number of total layers in the structure.

Structural studies indicate that the AF phase corresponds to the relatively undistorted fcc structure. The $M_{\perp}$ ferromagnetic phase that precedes it in Fe thickness corresponds to the unrelaxed fct structure. The spin-reorientation transition takes place within the fct structure, and the transition to fcc is suppressed at low-temperature growth. The fct structure is probably strain stabilized under these conditions due to the morphology of the interface and/or to magnetostriction effects. In this latter regard, Hembree *et al.*[32] recently documented for Fe/Cu(100) striking field-induced metastable states attributed to magneto-elastc effects. It is also of interest to speculate on the relationship of the AF fcc phase identified by Li *et al.*[28] to that observed in γ-Fe coherent precipitates in Cu host alloys. For the bulk alloys the Néel temperature T_N is low as is the magnetic moment ($<1\mu_B$).[33] For the films the magnetic ordering temperature is much higher (200-250K) suggestive that the moments may also be larger. However, it remains to be determined to what extent the surface ferromagnetic layer boosts the properties (T_N and μ) of the underlying AF film.

Finally, with increasing Fe thickness the bcc phase is reached at ~10-12ML Fe. The bcc films are ferromagnetic with in-plane easy axis dictated by the shape anisotropy. The coercivity exhibits striking peaks at the phase transitions, as shown in Fig. 6 for a wedge than spans the fct-fcc transition as well as the bcc transition. The coercivity provides a convenient way to track phase boundaries between fct-fcc, fcc-bcc and fct-bcc, as well as for the spin-reorientation transition. The most recent magnetic phase diagram for the growth of Fe/Cu(100) is presented in Ref. 28.

IV. Summary:

Recent SMOKE studies for Fe/Ag(100) and Fe/Cu(100) were highlighted. The documentation of the spin-reorientation transition for Fe/Ag(100) provides detailed

corollary measurements to those of Refs. 21 and 22. A suppressed but nonvanishing remanent magnetization was found within most of the pseudogap region in temperature and Fe thickness that characterizes the phase transition. The coercivity also exhibits a pronounced peak. The role of thermal fluctuations in a nearly isotropic 2D Heisenberg system is quite dramatic and can suppress long-range order and/or give rise to domain structures with unusual characteristics.

For fct Fe/Cu(100) the spin-reorientation transition occurs for low-temperature (≤200K) growth. However, for room-temperature growth, the ferromagnetic fct phase with vertical easy axis transforms to an AF fcc phase with a surface ferromagnetic 'live' layer. Fe films of ~10-12ML transform to the equilibrium bcc ferromagnetic structure with in-plane easy axis of magnetization. The Fe/Cu(100) system offers complexity associated with the interplay of magnetic and structural transitions. However, coercivity peaks can be used to track a variety of its phase transitions. The underlying mechanisms for achieving phase stability and that control coercive behavior remain a challenge to pursue in the future.

Acknowledgment:

This work was supported by the U.S. DOE Basic Energy Sciences-Materials Sciences under contract W-31-109-ENG-38.

Figure Captions:

Fig. 1 The perpendicular and parallel components of the remanent magnetization for a 6.0ML Fe film grown on Ag(100) *vs* temperature are shown in (a) and the corresponding coercivities from polar SMOKE measurements are shown in (b).

Fig. 2 Perpendicular and parallel components of the remanent magnetization at room temperature along an Fe/Ag(100) wedge.

Fig. 3 Magnetic phase diagram of Fe/Ag(100).

Fig. 4 Boundaries of the uniform ferromagnetic phase with vertical easy axis for an fct Fe/Cu(100) wedge as a function of measurement temperature and Fe thickness. The left boundary delineates the onset of detection of a ferromagnetic signal, while the right boundary terminates in a transition to the spin-in-plane fct phase.

Fig. 5 Spin-reorientation transition for Fe/Cu(100).

Fig. 6 Coercivities along a wedge of Fe/Cu(100) illustrating peaks at the phase transitions. The left transition is to the fcc phase, while the right transition is to the bcc structure.

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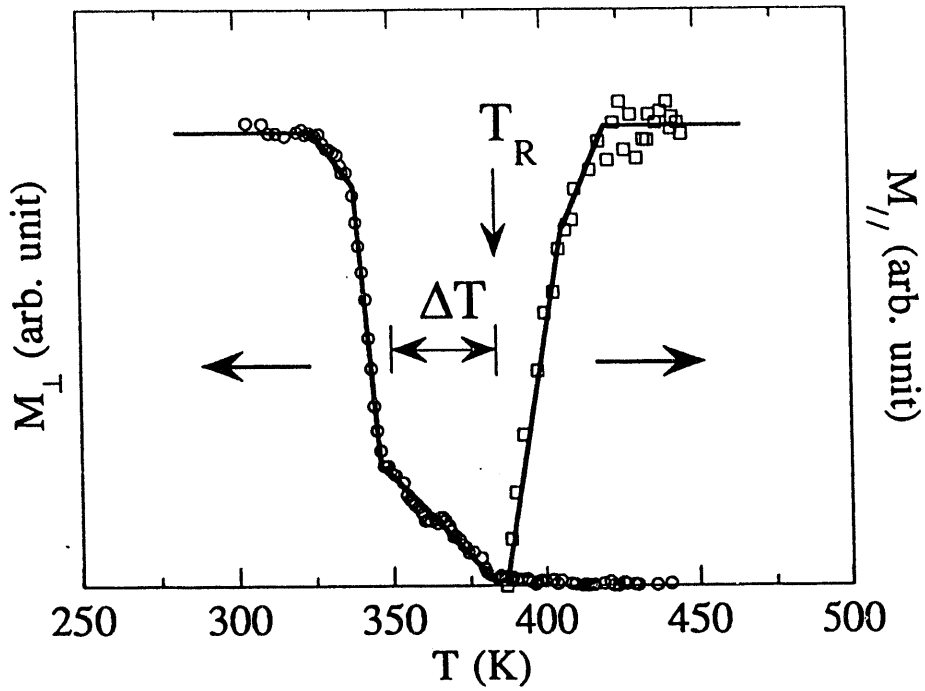
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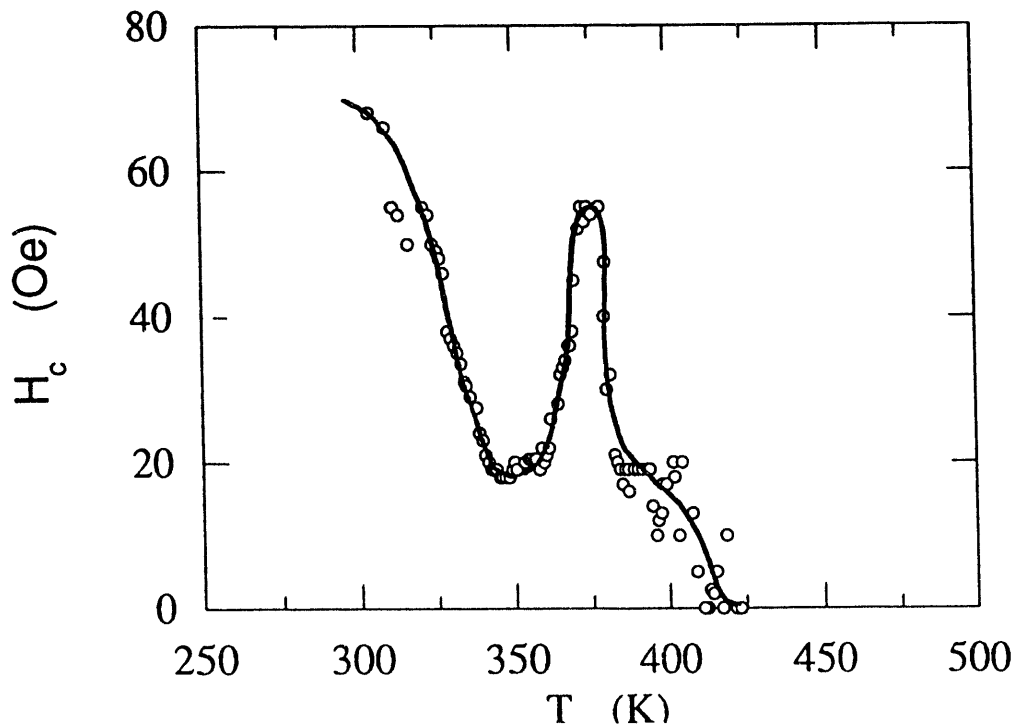
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Fig. 1
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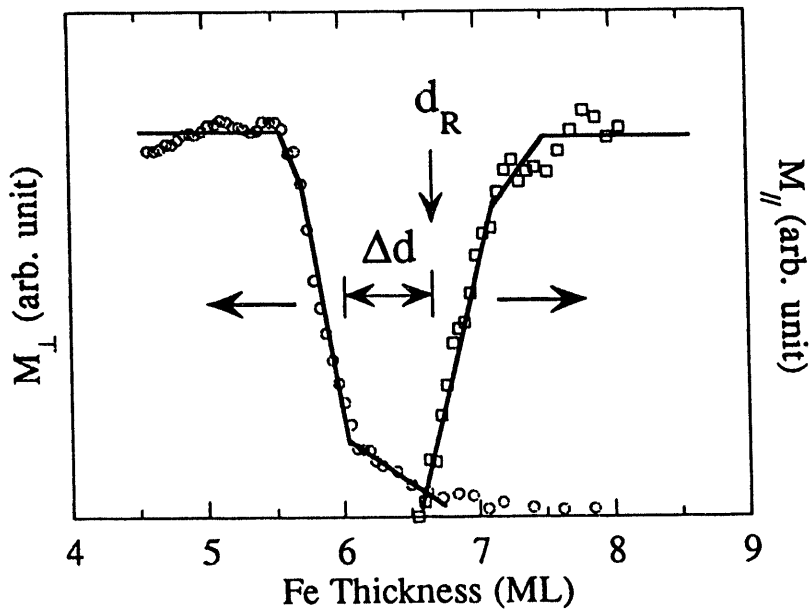

Fig. 2
DD-01

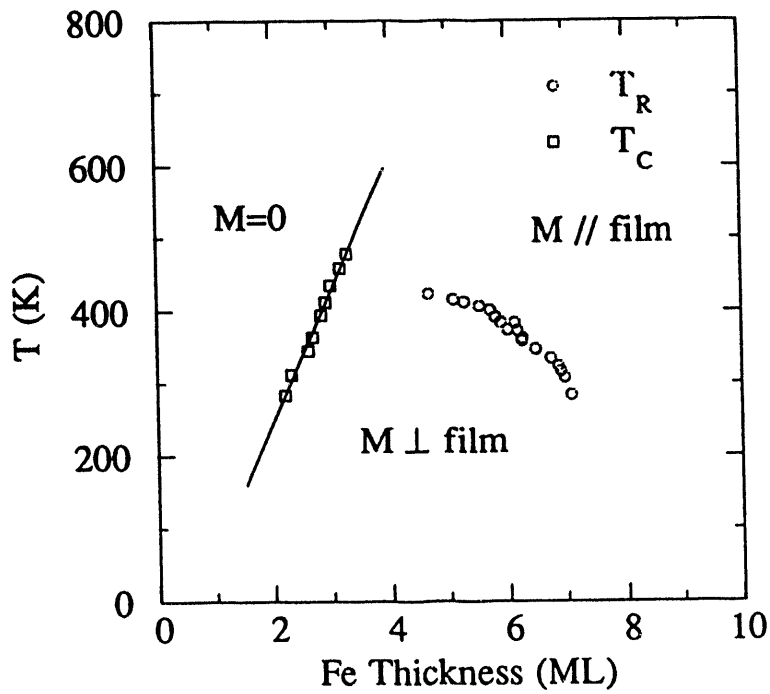
800
600
400
200
0
T (K)
0
2
4
6
8
10
Fe Thickness (ML)
T_R
T_C
M=0
M // film
M ⊥ film

Fig. 3
DD-01

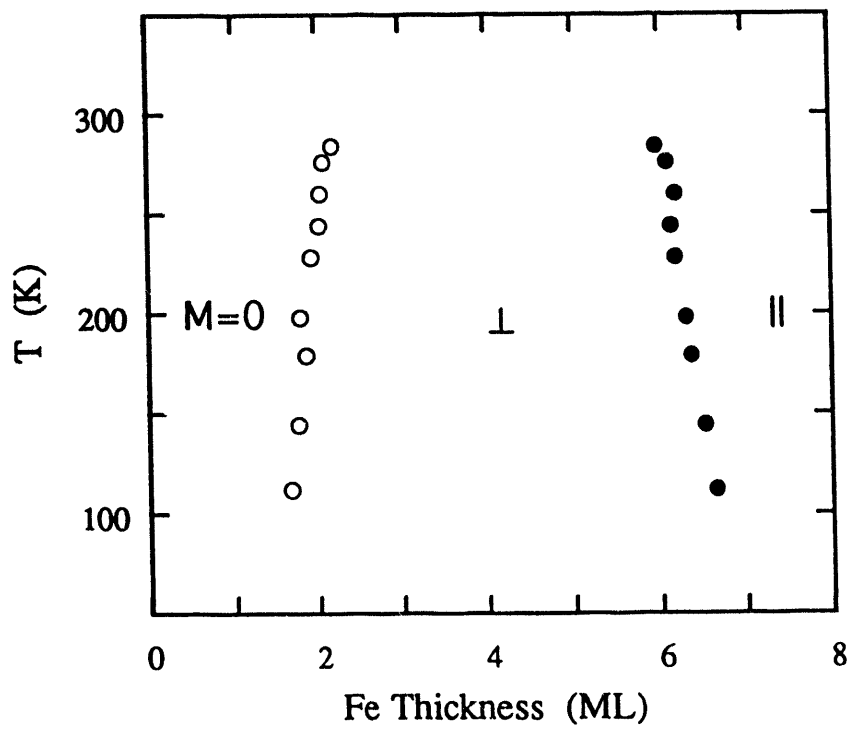

Fig. 4
DD-01

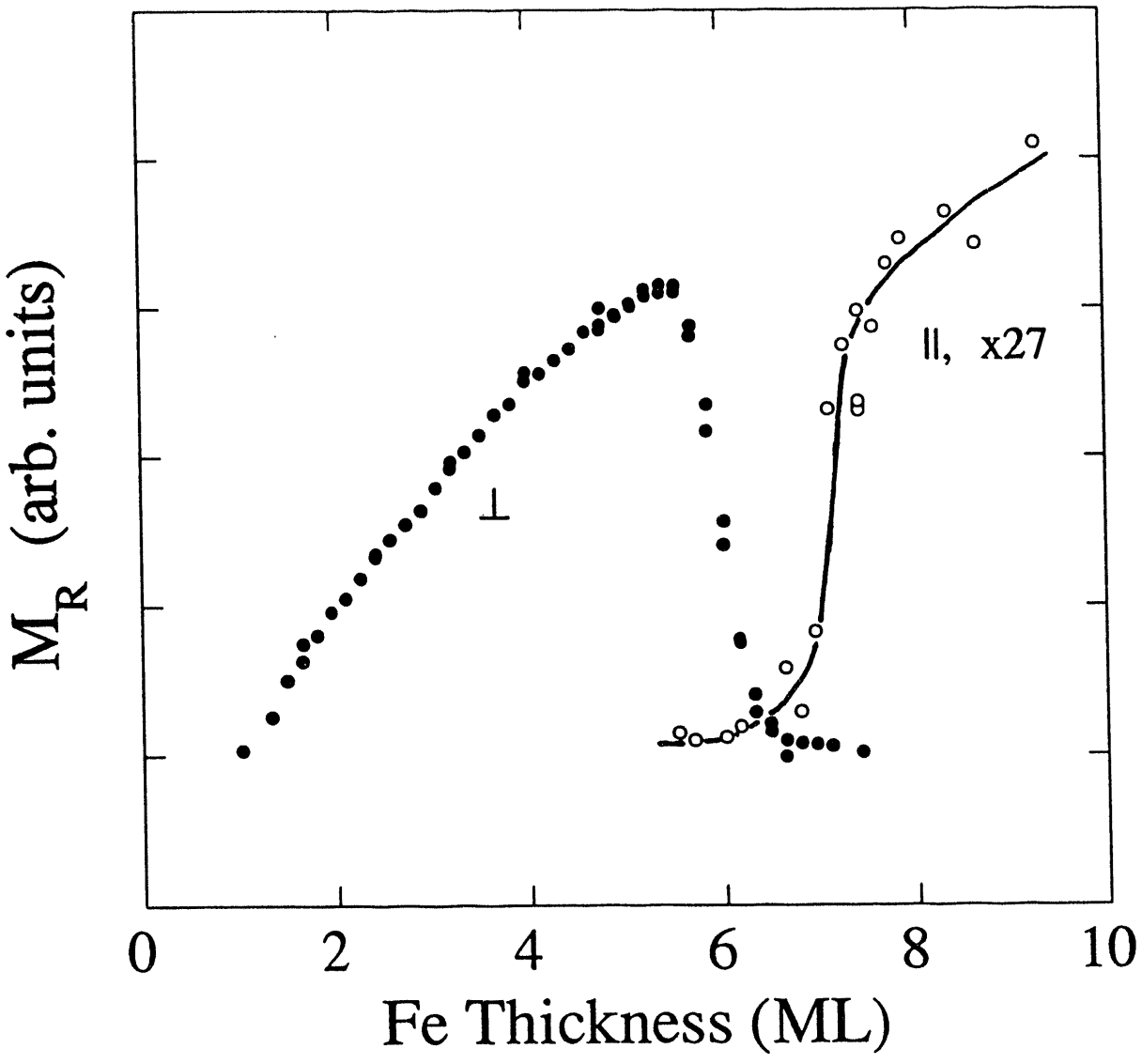

Fig. 5
DD-01

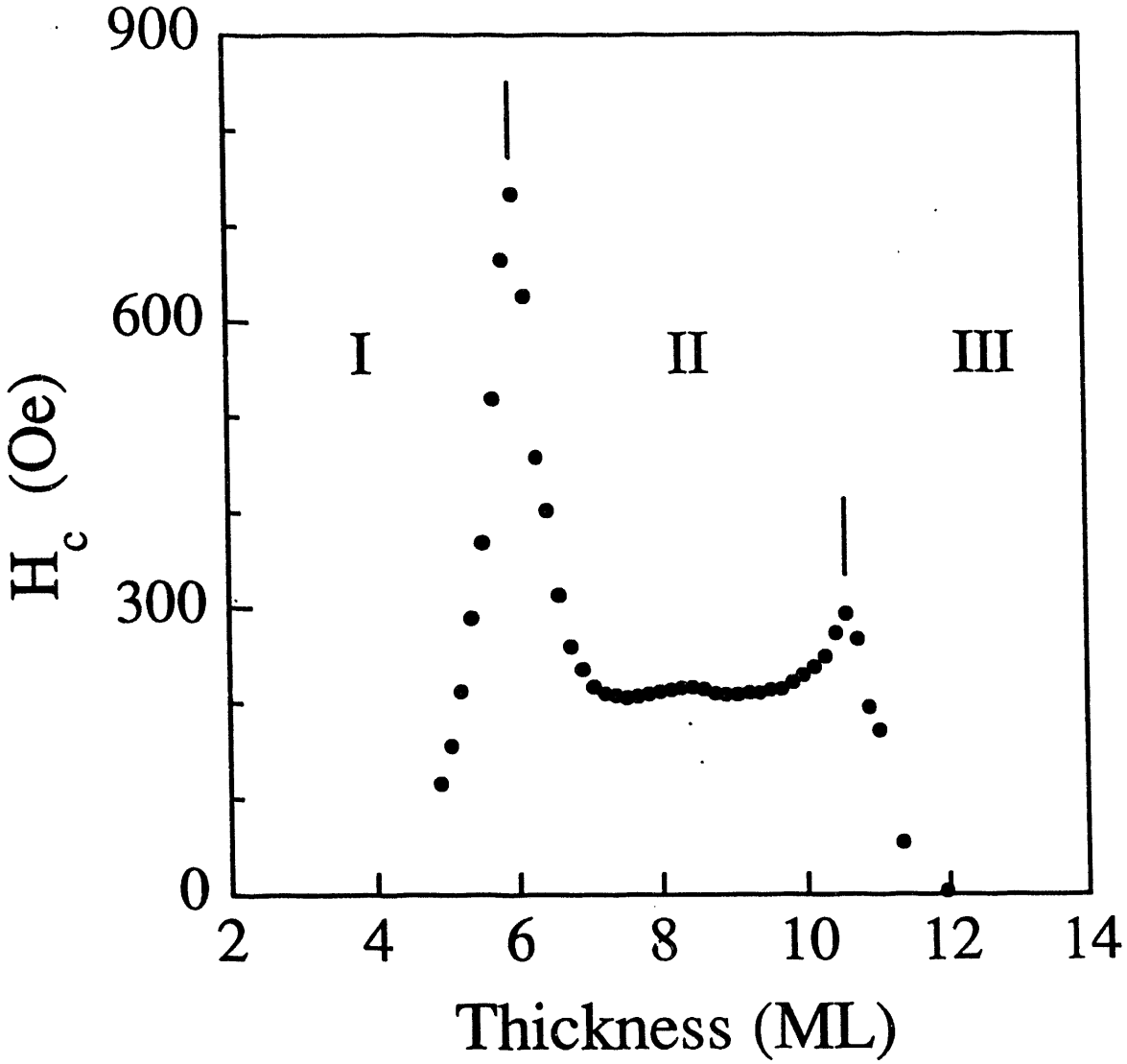

Fig. 6
DD-01

DATE

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7/25/94

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