

Curie temperature and morphology in ultrathin Co/W(110) films

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The magnetic properties of ultrathin Co films grown on W(110) were studied as a function of Co thickness (1–5 monolayers), substrate temperature during the growth ($170\text{ K} \leq T_s \leq 420\text{ K}$), and annealing temperature (T_{an} , 800 K) for films grown at room temperature. Magnetic results are obtained by means of *in situ* magneto-optical Kerr effect in static fields and small oscillating fields. The Curie temperature T_C of 1.4–2 monolayers varies linearly from 160 to 450 K. No change of this behavior is found for depositions at $170\text{ K} < T_s < 370\text{ K}$. Furthermore, we measure a large ac susceptibility $\chi_{\text{max}} \approx 3000$. This indicates the good magnetic homogeneity of the film. Layers annealed to $T_{\text{an}} > 440\text{ K}$ show a much lower T_C and a reduced χ_{max} . Deposition at $T_s > 420\text{ K}$ yields the same behavior. This is correlated to changes in the morphology of the Co layers as observed by Auger spectroscopy.

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I. INTRODUCTION

The para- to ferromagnetic phase transition in ultrathin films has attracted considerable interest in the past decade.¹ For example, the Curie temperature T_C in the monolayer range is drastically lower than in the bulk due to the finite-size effect.^{2,3} In the case of Ni (Refs. 4–6) and Gd,⁷ the dependence of T_C on the film thickness can be measured over a large thickness regime. For Fe or Co, on the other hand—with their much higher bulk Curie temperature (1043 and 1388 K)—it is impossible to determine T_C for more than three to four monolayers (ML) without strong structural modifications. Upon heating the overlayer either diffuses into the substrate [above $\sim 450\text{ K}$ for Co/Cu(001) (Ref. 3)] or changes from a flat film to a three-dimensional island film [for example, Co and Fe on Mo(110) (Ref. 8) or Fe (Ref. 9) and Gd on W(110) (Ref. 10)]. In the case of island formation, one expects that T_C increases since the average height of the islands must be larger than the thickness of the continuous layer.¹¹ However, if the initial layer thickness is very small, one might expect that disconnected islands (clusters) form which are laterally so small that the whole “film” behaves as an assembly of superparamagnetic particles.^{12,13} For example, evidence for superparamagnetic behavior has been reported for $D < 4$ ML Co on Ru(0001).¹⁴ Such a film is expected to show a decreased ordering temperature which results from the blocking of thermal fluctuations of individual ferromagnetic clusters. With a change of island size and separation a transition from superparamagnetism to ferromagnetism may be observable. For example, for Co/Cu(001) a jump of T_C by more than 150 K at about 1.8 ML

was measured and attributed to a change of morphology.¹⁵ In the case of Fe/W(110),¹⁶ it was found that at about 1.4 ML long range order may even be suppressed. More recent results, however, show that ferromagnetism exists in this thickness regime and that an extremely high coercive field (0.3 T) appears.¹⁷ Similar behavior could be expected for Co/W(110) and will be investigated here.

The large surface energy (2.9 J/m^2) of W compared to the one of Co (2.0 J/m^2) (Ref. 18) leads to a layer-by-layer growth at room temperature.^{19–21} Upon heating to 600 K the continuous film breaks up into islands, while the first Co monolayer is believed to be thermodynamically stable with no interdiffusion with the substrate.¹⁹ Hence the possibly different types of long range order, that is the behavior of T_C , can be studied for different thicknesses as a function of temperature without alloy formation. We will present results for Co deposited at different substrate temperatures and for films which were annealed up to 800 K after deposition at 300 K. After a brief review of the structure of the Co films^{20–22} we will focus on the magnetic properties of very thin films (below 5 ML). The magnetic characterization is performed *in situ* using the magneto-optic Kerr effect (MOKE). ac-susceptibility measurements in a small oscillating magnetic field (ac-MOKE) and hysteresis loops (dc-MOKE) recorded as a function of temperature allow one to precisely determine T_C . The ac-MOKE signal is calibrated in SI units, which yields quantitative information on the magnetic homogeneity of the film.²³ We will correlate our magnetic results with structural changes in our films as observed by Auger spectroscopy.

II. EXPERIMENT

Co films were deposited by electron beam evaporation in a vacuum better than 2×10^{-10} mbar on a W(110) single crystal as described earlier.²⁴ Low energy electron diffraction (LEED) and Auger electron spectroscopy (AES) were performed to check the structure and cleanliness of the film. The thickness of the film is calibrated by means of the Co(775 eV)/W(163/169 eV) Auger amplitude ratio: the thickness error is about 10%. The substrate temperature T_s is varied between 170 and 420 K. The MOKE measurements are performed in the longitudinal geometry: the external field is applied in the film plane along the easy axis of the Co magnetization.²² The Kerr effect is detected by a photoelastic modulation technique described elsewhere.²⁴⁻²⁶ With the same setup, we record hysteresis loops in static magnetic fields up to 100 Oe and magnetic susceptibilities in a small oscillating field ($f=70$ Hz; amplitude=1.4 Oe). It is worthwhile to note that the amplitude of this field is important.²⁷ The susceptibility maximum and the accuracy in the determination of T_C increases for smaller modulation amplitudes.^{23,28} We observed the same behavior for our cobalt films. We chose an amplitude of 1.4 Oe, which gives a good signal to noise ratio and a small error bar for T_C (~ 1 K).

III. GROWTH

The growth of Co on bcc W(110) has been studied by several groups.^{21,22,29} The growth is pseudomorphic up to ~ 0.7 ML. Further deposition leads to a distorted hcp (0001) film in the Nishiyama-Wasserman orientation $[1120]$ Co \parallel $[001]$ W up to 10 ML. Above this thickness, the film progressively relaxes into the bulk hcp Co(0001) structure. It has also been shown that the Co film grows layer by layer at 100 K,²¹ 130 K,¹⁹ and 300 K.²⁰⁻²² Johnson *et al.*¹⁹ observed that a 3 ML thick layer-by-layer Co film deposited at 130 K forms three-dimensional clusters above 500 K. However, it is possible that T_l (the temperature above which the layer-by-layer film breaks up into three-dimensional islands) varies with the thickness of the film as has been shown for Co, Fe, or Ni on Mo (110) (Ref. 8), and Gd on W(110).¹⁰ Our Auger data versus evaporation time confirm the layer-by-layer growth mode ($T_s=340$ K). The thermal stability for five different Co thicknesses, 1, 1.6, 2.5, 3.5, and 4 ML ($T_s=340$ K), as a function of thermal treatment is reflected in the Auger amplitude ratios Co(53 eV)/W(163/169 eV) shown in Fig. 1. The Auger ratios of 2.5, 3.5, and 4 ML remain constant up to an annealing temperature of about 500 K and then strongly decrease with increasing temperature. This change corresponds to a breakup of the flat continuous film into three-dimensional (3D) islands. There is also a slight, gradual decrease of the 1.6 ML Auger ratios near 500 K which indicates that some smaller clusters thicker than a double layer form. The absence of a clear jump as for the thicker films (2.5, 3.5, and 4 ML) can be explained, if only the second partially filled layer participates in the island formation. The ratio of 1 ML remains constant which indicates that it is thermally stable. It is interesting to remember that Co films deposited at 130 K (Ref. 19) show an increase of the Co (775 eV)/W(163/169 eV) ratio up to ~ 500 K, which

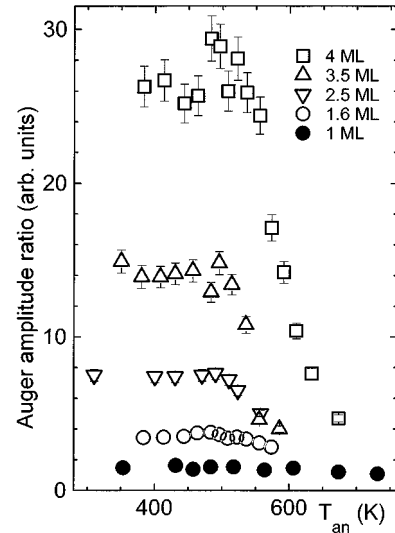


FIG. 1. Co(53 eV)/W(163/169 eV) Auger amplitude ratio versus annealing temperature for 0.9, 1.6, 2.5, 3.5, and 4 ML Co on W(110). Each value has been measured at the same temperature (~ 350 K). The error bars for the 0.9 and 1.6 ML data are smaller than the symbols.

is interpreted as a smoothing of the film. Our films deposited at 340 K do not need any thermal treatment to improve their quality. It will be confirmed below by our magnetic measurements.

IV. MAGNETISM

The Curie temperature T_C of a ferromagnet is defined as the temperature above which the *spontaneous* magnetization M_S vanishes. However, in many experimental techniques such as SQUID or MOKE, the critical behavior at T_C is investigated by measuring the macroscopic *remanent* magnetization M_R as a function of temperature. At T_C this quantity becomes very small, which makes it difficult to determine T_C accurately. It has also been shown that in the presence of structural inhomogeneities M_R vanishes far above the true T_C .⁵ An easier way to determine T_C is to study the internal susceptibility χ_{int} in small magnetic fields since this quantity diverges at T_C . However, the experimental susceptibility χ_{exp} which is measured in a magnetic field applied in the film plane is limited by the demagnetization factor N_{\parallel} of the sample according to

$$\chi_{\text{exp}} = \chi_{\text{int}} / (1 + N_{\parallel} \chi_{\text{int}}). \quad (1)$$

For ultrathin films N_{\parallel} becomes very small, but not zero, as has been discussed before.²³ The Curie temperature of Co/W(110) depends strongly on the thickness as described below. It turned out that the thickness calibration by Auger spectroscopy (error bar $\sim 10\%$) was too inaccurate for a $T_C(D)$ analysis. Therefore, we have also measured the susceptibility after successive, precisely timed cobalt deposition steps which yields a relative accuracy of better than 0.5%. Only the absolute thickness of the thickest film has been determined by Auger analysis. Figure 2 shows the susceptibility peaks calibrated in SI units for five successive cobalt depositions at 335 K. The relative error of the thickness is

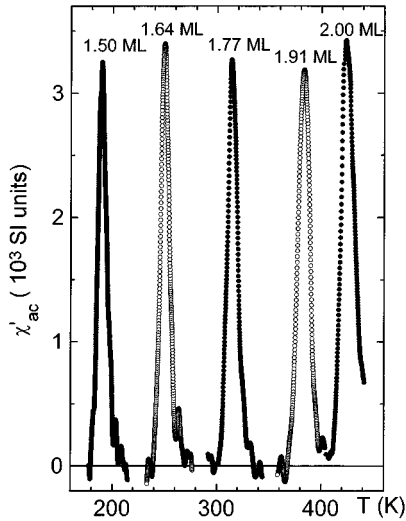


FIG. 2. The real part of the ac susceptibility measured at 70 Hz with a field amplitude of 1.4 Oe for different Co film thicknesses deposited at 340 K. The thickness is given with two digits, because of the strong change of T_C ; clearly this is only a relative accuracy (see text).

smaller than 0.01 ML. Note that $\chi_{\max} \approx 3300$. We assume bulk cobalt magnetization $4\pi M = 1.76$ T for our films. The Curie temperature, which is determined at the maximum of the peak, increases with increasing film thickness. The good quality of our data allows a precise determination of $T_C (< 1$ K). The finite width of the susceptibility peak at half maximum $\Delta\chi_{1/2}$ could be attributed to a thickness variation in the film¹¹ over the spot diameter of the laser (0.5 mm). In our case, we find $\Delta\chi_{1/2} = 10$ K, which can be interpreted as a variation of only 0.02 ML if one uses the T_C dependence from Fig. 3.

A way to estimate the magnetic homogeneity of a sample is to determine its demagnetization factor N_{\parallel} ,²³ which corresponds to the inverse of the experimental susceptibility at T_C [Eq.(1)]. If we assume that our Co film consists of ultrathin disks with thickness h and diameter $2r$, the continuum theory yields a demagnetization factor $N_{\parallel} = \pi/4(h/2r)$.²³ In that case, our N_{\parallel} values, typically 3×10^{-4} , correspond to magnetic disks of nearly 1 μm diameter and about 3 \AA thickness. It indicates that even for a monolayer the magnetic homogeneity is not disturbed by monoatomic steps of the substrate surface. If we suppose a perfect layer-by-layer growth for $D \leq 3$ ML as observed in a recent scanning tunneling microscopy study,²⁹ the demagnetization factor should decrease when going from 1.5 to 2 ML. We find that χ_{\max} does not change which indicates that the amplitude of the oscillating field is the limiting factor as discussed before. The true N_{\parallel} could be smaller than 3×10^{-4} . Figure 3 shows the Curie temperature as a function of the thickness for films deposited at $T_s = 170, 300, 335, 370,$ and 420 K. T_C is determined after precisely timed cobalt deposition steps. Only the data for $T_s = 170$ K are obtained with two different films to avoid a modification of the film by heating.¹⁹ The data with $T_s = 420$ K will be described in the second part of the discussion. The small thickness range (1.4 to 2 ML) for which we show T_C is given by the fact that after heating to above 440 K the Curie temperature was not reproducible.

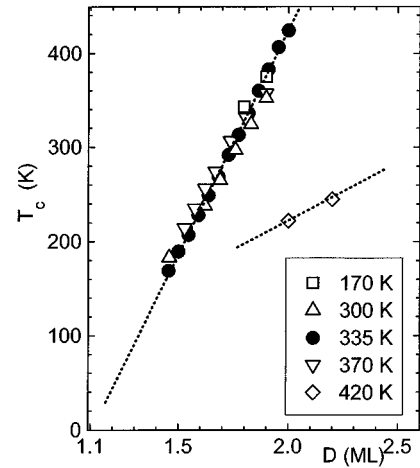


FIG. 3. Curie temperature as a function of Co thickness for different substrate temperatures. The dashed line is a linear fit of the data with $T_s = 335$ K.

The lower limit (1.4 ML) is due to the lowest temperature (150 K) which can be experimentally achieved. This means that long range order may be found at lower temperatures. Let us consider the results for $T_s = 170$ to 370 K for which $T_C(D)$ is the same. First, one should note that our $T_C(1.7 \text{ ML}) \sim 300$ K is in excellent agreement with other results for Co/W(110).³⁰ Secondly, there is no indication of a loss of long range order as in the case of Fe/W(110).¹⁶ Furthermore, we find that T_C is a linear function of D from 1.4 to 2 ML with a slope of ~ 500 K/ML. Interestingly, $T_C(D)$ cannot be fitted with a power law dependence according to the finite size scaling ansatz^{2,6}

$$(T_C(\infty) - T_C(D))/T_C(\infty) = cD^{-1/\nu}, \quad (2)$$

c depends on the material and the coordination number.⁶ The critical exponent ν of the correlation length equals 1 in the 2D case and 0.705 in the 3D case (Heisenberg). For both cases a power law fit cannot be obtained with any c and fixed $T_C(\infty) = 1388$ K. This indicates that Eq. (2) with $\nu = 0.705$ is valid only in the 3D limit⁶ and cannot be applied in the monolayer range. But also the 2D critical exponent does not yield a fit, which may indicate that lateral scaling due to a changing width of monolayer patches is not the reason for the observed $T_C(D)$. Another possible explanation for the observed $T_C(D)$ may be given by the thickness dependence of magnetic anisotropy. It has been calculated³¹ that T_C of a 2D system should increase with magnetic anisotropy K according to

$$T_C = 2T_C(\infty)/\ln(\pi^2 J/K), \quad (3)$$

with J the exchange interaction. Using $J = 26$ meV/atom estimated for a Co(111)/Cu(111) monolayer³² and the out-of-plane magnetic anisotropy $K \approx 35 \mu\text{eV/atom}$ measured for ≈ 0.4 nm Co/W(110),²² one calculates $T_C = 312$ K. This lies well in the temperature range found for 1.4 to 2 ML (Fig. 3). To explain the thickness dependent slope of T_C one can assume that the ratio J/K varies with a certain power N as a function of D , that is $J/K \propto D^N$. However, we find that only an unrealistic exponent $N \approx 10$ yields a reasonable fit to $T_C(D)$. As a result we conclude that each theoretical model

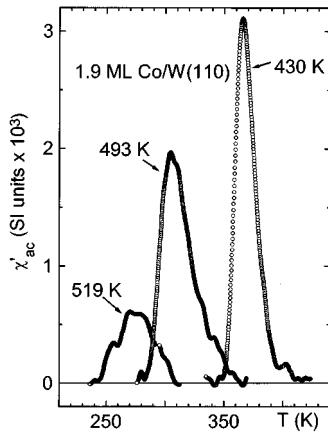


FIG. 4. ac susceptibility of 1.9 ML Co deposited at 340 K after successive annealing at 430, 493, and 519 K.

considered individually only does not explain the experimental behavior. One may speculate, however, that the combination of both, that is finite size effect and change of anisotropy, contribute to the linear behavior.

For completeness we mention, that Fritsche *et al.*²² find $D=0.9$ ML “dead layers,” that is, vanishing magnetic order, from torsion oscillation magnetometry at room temperature. A linear extrapolation of our data would suggest that $D=1.1$ ML do not order ferromagnetically at finite temperature (Fig. 3). However, measurements at lower temperatures are required to verify that possibility. Deposition at $T_s=420$ K yields a much lower T_C (Fig. 3) with a different slope, which suggests that even submonolayers are ferromagnetically ordered if deposited under this condition. We will come back to this below.

As mentioned previously Co films thicker than 1 ML and deposited at 340 K are structurally unstable above ~ 500 K, and irreversible changes of the coercive field, the shape of the hysteresis, and the susceptibility peak are observed above 440 K. We will focus on the changes at the phase transition only. Figure 4 shows the influence of three successive annealing steps on the susceptibility of a 1.9 ML thick Co film. The high and sharp susceptibility peak of the film deposited at 340 K progressively shifts to lower temperatures and becomes smaller and broader with increasing T_{an} . The two last effects are due to an increase of the roughness of the film, that is an increase in $N_{||}$ [Eq. (1)]. Above $T_{an} \approx 520$ K, no peak is detected because either the demagnetization factor is too large, or the Curie temperature is lower than 150 K. In Fig. 5 the characteristics of the ac-susceptibility peak, that is the peak height χ'_{max} and the half width at half maximum $\Delta\chi_{1/2}$, are shown as a function of T_{an} for 1.8, 1.9, and 2.7 ML. A decrease of T_C as a function of annealing is found for all films thinner than 3 ML. Above 440 K there is a correlation between the decrease of χ'_{max} , $\Delta\chi_{1/2}$, and T_C [Figs. 5(a)–5(c)]. χ'_{max} decreases by three orders of magnitude for a change of T_{an} by 100 K only. Also the half width increases dramatically, while T_C decreases by almost 40%. Interestingly, most of these changes occur for annealing temperatures where no change in the Auger ratios is observed. This shows how sensitively the magnetism of ultrathin films reacts to morphological changes, which are hardly detectable by Auger spectroscopy

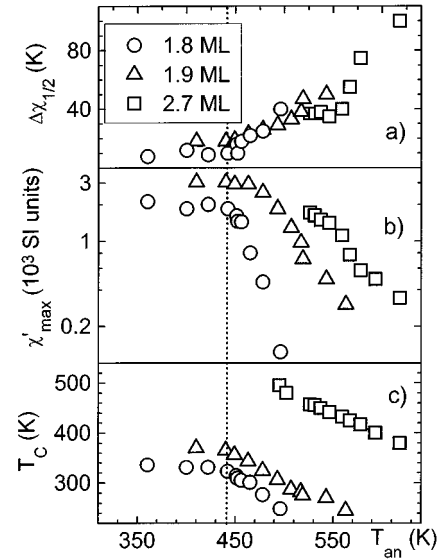


FIG. 5. (a) The half width at half maximum $\Delta\chi_{1/2}$, (b) the maximum of the real part of the ac susceptibility χ'_{max} , and (c) the Curie temperature (T_C), for different Co thicknesses as a function of annealing.

or LEED. One should note that T_C of 2.7 ML Co deposited at 340 K should be much higher than 490 K (according to Fig. 3) which is experimentally found (Fig. 5). There is evidence from hysteresis loops recorded during heating (a sudden decrease of the coercive field above 440 K) that the morphology changes before T_C of a continuous layer is reached.

Interestingly one finds that films which are deposited below 370 K and subsequently annealed up to T_s have the same Curie temperature as the films which are deposited at T_s . Also the height of the χ peak at T_C is similarly small (<300), and $\Delta\chi_{1/2}$ is larger than 70 K.

As mentioned earlier a decrease of T_C after heating is unexpected, since one would expect that thicker islands form which have a larger T_C . To explain the different behavior we discuss two possibilities. T_C starts to decrease already at $T_{an} \approx 440$ K, at which temperature no structural modification is evident by Auger spectroscopy. Hence one might argue that between 440 and 500 K strain in the layer is released, and the stress induced anisotropy is decreased. According to Eq. (3) the decrease of the anisotropy will contribute to the initial decrease of T_C . Also a slight modification of the topography, that is the demagnetization factor of the film, may be present. This is seen in the changes of the χ peak [Figs. 5(a) and 5(b)]. At $T \approx 500$ K island formation becomes detectable by Auger analysis, and the susceptibility peak becomes progressively small and broad. The peak of the 1.8 ML film vanishes at 500 K, the one of 1.9 ML at 570 K, and the one of 2.7 ML above 700 K. The gradual decrease of T_C above $T_{an} \approx 500$ K and the broadening of the χ peak may indicate that superparamagnetic islands of decreasing lateral size and increasing separation grow. That is to say a continuous transition from ferromagnetic order with a Curie temperature to superparamagnetic behavior with a blocking temperature is observed. From the available susceptibility data,

however, one cannot unambiguously distinguish between a superparamagnetic blocking and a ferromagnetic ordering temperature. Above 3 ML the Curie temperature increases because enough material has been deposited to form laterally large enough islands, so that the lateral finite size effect plays no role and the height of the islands determines T_C according to Eq. (2). Surprisingly the decrease of the Curie temperature occurs for $D < 3$ ML only. For $D > 3$ ML we do not find a susceptibility peak from 150 K up to 800 K. From Fig. 3 we estimate $T_C \gg 600$ K for an unannealed, flat film which is much higher than the temperature (~ 500 K) for 3D island formation (Fig. 1). As a result, $T_C(D > 3$ ML) cannot be determined without changing the structure of the film irreversibly. The lack of a susceptibility peak, however, does not prove that T_C is higher than 800 K unambiguously, since χ_{\max} may be too small for the experimental sensitivity due to the increase of N_{\parallel} when islands are formed.²³ Therefore we recorded hysteresis loops up to 800 K—our highest achievable sample temperature—which confirms that the layers are ferromagnetically ordered. For $D > 3$ ML we observe that the coercive field of a film deposited at 340 K first decreases slightly as a function of temperature and for $T > 440$ K increases continuously (not shown here). This indicates an increase of T_C or an increase of the uniaxial magnetic anisotropy³² above 440 K. The latter is unlikely, since the breakup of Co film into 3D islands relieves the strain and decreases the uniaxial magnetoelastic strain anisotropy.²² As a consequence, the increase of the coercive field most likely results from a larger T_C . This is the same magnetic behavior as found for 5 to 11 ML Gd films on W(110).¹¹ In this system we found an increase of T_C when the film breaks up into large 3D islands. Hence we conclude that for $D > 3$ ML Co islands form during heating that have lateral dimensions which do not limit the divergence of the correlation length

similarly to the case of Gd/W(110). This interpretation agrees with our Auger results (Fig. 1) which show that large, nearly uncovered W areas should exist between the islands.

V. CONCLUSION

Ultrathin cobalt films deposited on W(110) have been studied *in situ* by ac- and dc-MOKE. The Curie temperature for 1.4 to 2 ML is precisely determined by ac-susceptibility measurements. T_C ($1.4 < D < 2$ ML) varies linearly as a function of film thickness for films deposited at 170 and 370 K. After thermal treatment at $T_{\text{an}} > 440$ K the magnetic properties are not reversible. A transition from a flat film (below 440 K) to a 3D-islands film (above 500 K) is observed. For $D < 3$ ML, T_C continuously *decreases* with increasing $T_{\text{an}} > 440$ K, whereas for $D > 3$ ML T_C *increases*. This different behavior is tentatively ascribed to the formation of islands with different sizes and can be interpreted as a transition from superparamagnetism to ferromagnetism as a function of coverage and thermal treatment. That is to say, below 3 ML the islands are most likely small and disconnected, while above 3 ML the coverage is high enough to form large islands which are ferromagnetically coupled. A recent scanning tunneling microscopy study on thick Co films²⁹ seems to confirm our structural interpretation for $D > 3$ ML. A magnetic “frustration” or an enhanced coercive field as for 1–2 ML Fe on W(110) is not observed.

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