Temperature-dependent ferromagnetic-resonance study in ultrahigh vacuum: Magnetic anisotropies of thin iron films

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20- to 350-Å Fe thin films have been prepared on W(110) and studied by ferromagnetic resonance in ultrahigh vacuum. The magnetic anisotropy of Fe films and its temperature dependence were determined independently from the angular and the frequency dependences of the resonance field in the temperature range between 300 and 600 K. A switching of the easy axis of the magnetization from [$\bar{1}$10] to [001] was confirmed at a film thickness of about 110 Å. This is disucssed in terms of surface and volume contributions to the anisotropy energy.

I. INTRODUCTION

In recent years magnetic thin films have gained increasing interest for fundamental research and technological applications. Much effort has been devoted to understand the magnetic phase transition,[1–4] the magnetic moment,[5–7] and the magnetic anisotropy.[8–11] Thin Fe films have been studied extensively in the past.[12] One of the most interesting questions in such studies is the magnetic anisotropy and their temperature dependence, which determines the orientation of the magnetization. Recently there have been some reports in the literature concerning the magnetic anisotropy and the orientation of the magnetization of Fe(110) films. Prinz, Rado, and Krebs[13] have used ferromagnetic resonance (FMR) to determine the magnetic anisotropy of Fe(110) on GaAs at 300 K and have observed the switching of the magnetization of the Fe films from the [$\bar{1}$10] to the [001] direction with increasing film thickness of 50–150 Å. Gradmann, Korecki, and Waller[14] and Elmers and Gradmann[15] have performed a detailed investigation of the magnetic anisotropy of Fe(110) on W(110) using conversion electron Mössbauer spectroscopy and a magnetometer and found the same type of switching of the magnetization at 300 K as in Fe(110)/GaAs(110). The switching occurs between 95 and 70 Å depending on the preparation temperature. This observation was confirmed by Kurzawa *et al.*[16] with spin- and angle-resolved photoemission and Baumgart, Hillebrands, and Güntherodt[17] with a Brillouin light-scattering study. However, these determinations and observations were limited to room temperature. Since magnetic anisotropies are mostly dependent on the reduced temperature (in most cases much more than the magnetization itself) only temperature-dependent measurements yield complete information. Those measurements have been performed in the temperature range between 4 and 300 K but not in ultrahigh vacuum[18] (UHV). To determine the temperature dependence of the magnetic anisotropy between 300 and 600 K and to check the observed effect in Fe(110)/W(110) films by a different method, we performed further experiments.

In this paper we present an experimental study of the magnetic anisotropy of Fe/W(110) in UHV and its temperature dependence using ferromagnetic resonance (FMR). As discussed by Gradmann *et al.*,[19] Fe(110)/W(110) is an ideal system for the investigation of magnetic properties. It is thermodynamically stable. This means that for thermal treatments (up to 600 K) segregation and intermixing problems are negligible. Recently thickness dependent anisotropies were observed in Fe(110) films on Mo(110) (Ref. 20) questioning if previous work is correct.[15] The present work does not focus on the preparation of highest quality epitaxy as described in Refs. 19, 21, and 22. As the literature cited above, e.g., Refs. 13, 15, 19, and 20, shows, there exist common features for films prepared in a 10^{-6} mbar vacuum or in UHV. FMR is a well established method for a quantitative determination of magnetic anisotropies[13,23] but only a few efforts were made to carry out FMR *in situ*.[24,25] This guarantees that the magnetic properties of the film remain unchanged and are not affected by oxidation or cover layers. Using the measured temperature, angular, and frequency dependences of the resonance field H one can deduce the magnetic anisotropy energy precisely.

II. THEORETICAL MODEL

A brief summary of the theory of the FMR is given here. We consider the magnetic anisotropy of Fe films on W(110) in the framework of uniaxial and cubic anisotropy. The coordinate system used in our calculation is shown in Fig. 1.

To calculate the resonance frequency one follows the approach of Suhl[26] The free-energy density of the system is, considering the uniaxial and cubic anisotropy energies,

$$E=-HM(\sin\vartheta_H\cos\varphi\sin\vartheta+\cos\vartheta_H\cos\vartheta)+2\pi M^2\cos^2\vartheta+K_n\cos^2\vartheta+K_u\sin^2\vartheta\sin^2\varphi$$
$$+K_1[\tfrac{1}{4}\sin^4\vartheta\sin^4\varphi+\tfrac{1}{4}\cos^4\vartheta+\sin^4\vartheta\sin^2\varphi\cos^2\varphi+\sin^2\vartheta\cos^2\vartheta\cos^2\varphi-\tfrac{1}{2}\sin^2\vartheta\cos^2\vartheta\sin^2\varphi]\ , \quad (1)$$

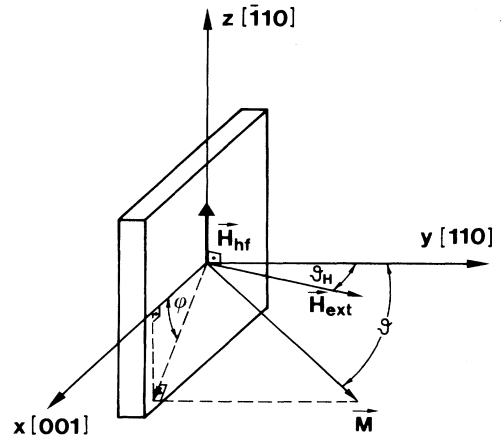

FIG. 1. Coordinate system used to describe the ferromagnetic resonance experiment. The microwave field is parallel to the z axis. The external magnetic field can be rotated in the x-y plane. The y axis is the surface normal.

where the first term is the Zeeman energy, the second the demagnetization energy (M: saturation magnetization). The terms containing the constants K_n and K_u represent the out-of-plane and in-plane uniaxial anisotropy energies, respectively. The last term (second line) is the cubic anisotropy energy K_1. The equilibrium condition for the magnetization and the resonance condition for the FMR can be found using the following equations:[26,27]

$$\frac{\partial E}{\partial \vartheta}=\frac{\partial E}{\partial \varphi}\equiv 0 , \tag{2}$$

$$\left[\frac{\omega}{\gamma}\right]^2=\frac{1}{M^2\sin^2\vartheta}\left[\frac{\partial^2 E}{\partial \vartheta^2}\frac{\partial^2 E}{\partial \varphi^2}-\left[\frac{\partial^2 E}{\partial \vartheta \partial \varphi}\right]^2\right] . \tag{3}$$

Now we consider the more general case of experimental configurations as shown in Fig. 1. It is reasonable to assume that the magnetization lies in the x-y plane, when the resonance absorption occurs. As will be seen later, this assumption will turn out to be correct. For films ≥ 110 Å, the easy direction is the [001] direction and the magnetization lies in the x-y plane. For films ≤ 110 Å, the easy direction is the [1̄10] direction. The resonance field at 9 GHz is large enough to overcome the in-plane anisotropy field and to turn the magnetization into the x-y plane. On this condition the equilibrium angle φ_{eq} is equal to zero and the equilibrium condition is

$$\frac{\partial E}{\partial \vartheta}=-HM\sin(\vartheta_H-\vartheta_{eq})-(4\pi M^2+2K_n)\sin\vartheta_{eq}\cos\vartheta_{eq}$$
$$+K_1(\cos^3\vartheta_{eq}\sin\vartheta_{eq}-2\sin^3\vartheta_{eq}\cos\vartheta_{eq})\equiv 0 , \tag{4}$$

The resonance condition can be derived from Eqs. (1) and (3) yielding

$$\left[\frac{\omega}{\gamma}\right]^2=\left[-H\cos(\vartheta_H-\vartheta_{eq})+4\pi M_{eff}(2\sin^2\vartheta_{eq}-1)+\frac{K_1}{M}(12\sin^4\vartheta_{eq}-15\sin^2\vartheta_{eq}+3)\right]$$
$$\times\left[H\frac{\sin\vartheta_H}{\sin\vartheta_{eq}}-+\frac{2K_{eff}}{M}+\frac{K_1}{M}(\sin^2\vartheta_{eq}-1)\right] , \tag{5}$$

with

$$4\pi M_{eff}\equiv 4\pi M+\frac{2(K_n+K_1)}{M} ; \quad K_{eff}\equiv K_u+K_1 .$$

In order to determine the magnetic anisotropies, one can perform measurements of the angular dependence of the resonance field and fit it with Eqs. (4) and (5).

With the external magnetic field applied in the film plane ($\vartheta_H=90°$), the resonance condition has the familiar form

$$(\omega/\gamma)^2=[H+4\pi M_{eff}]\left[H+\frac{2K_{eff}}{M}\right] . \tag{6}$$

As will be shown in Sec. III, Eqs. (4), (5), and (6) are used to determine the magnetic anisotropy $4\pi M_{eff}$ and K_{eff} of Fe(110) films on W(110). It is noteworthy to say that in FMR these quantities are measured in absolute and not in arbitrary units. Therefore its values can be compared to other parameters (i.e., saturation magnetization), see Sec. IV.

III. EXPERIMENT

The experiments were performed in an UHV chamber described previously.[28] The residual pressure of the chamber is $<2\times10^{-11}$ mbar. The W(110) substrate was cut from a single crystalline rod of tungsten and polished to within 0.5 ° of the [110] orientation by using standard metallographic techniques. The substrate was cleaned *in situ* by argon-ion sputtering and high-temperature annealing. Surface cleanliness was verified by Auger electron spectroscopy (AES). No contaminations were found within the detection limit of the Auger system ($<1\%$ of a carbon monolayer). Fe films were evaporated from a $5N$ Fe rod using the electron-beam evaporation method. The iron was mounted in an Al_2O_3 ceramic. The oxygen contamination was at about 5% as determined by AES. Epitaxy was performed at a substrate temperature of 300 K and for the 200 Å film at 600 K. The film thickness was calibrated using the Auger signal-time plot and a quartz microbalance. Low-energy electron diffraction (LEED) did not show a difference from the W(110) pattern up to 2 monolayers in accordance with Ref. 19 predicting a pseudomorphic growth for the first two layers at a substrate temperature of 300 K. Then the LEED spots began to broaden resulting in a diffuse pattern for all energies at about 10 monolayers. We therefore assume that the films consisted of small crystallites mainly oriented in the (110) face, which is the closest packed face

of a bcc lattice. This assumption is supported by the magnetic properties of our films.

Measurements of the angular, frequency, and temperature dependences of the resonance field were carried out *in situ* on freshly evaporated films. The temperature of the samples was monitored with a thermocouple and measured with a precision of 1 K. Typical temperature dependences of FMR spectra of a 54 Å Fe film at 9 and 4 GHz are shown in Fig. 2. There is an aligned and a nonaligned mode as had also been reported by Prinz, Rado, and Krebs.[13] This is a confirmation that the spontaneous magnetization lies in the film plane but perpendicular to the applied field along the W[001] direction. The linewidth are 63 and 45 G, respectively. Similar results were obtained by Heinrich *et al.*,[8] whereas Prinz, Rado, and Krebs[13] have published slightly smaller FMR absorption lines. The larger linewidths are caused by substrate mismatch and internal strain. The linewidths are obviously not affected by the oxygen contamination of the surface. The resonance position shifts with temperature. This study (angular, frequency, and temperature variation) shows the full power of the ferromagnetic resonance for an investigation of ultrathin films.

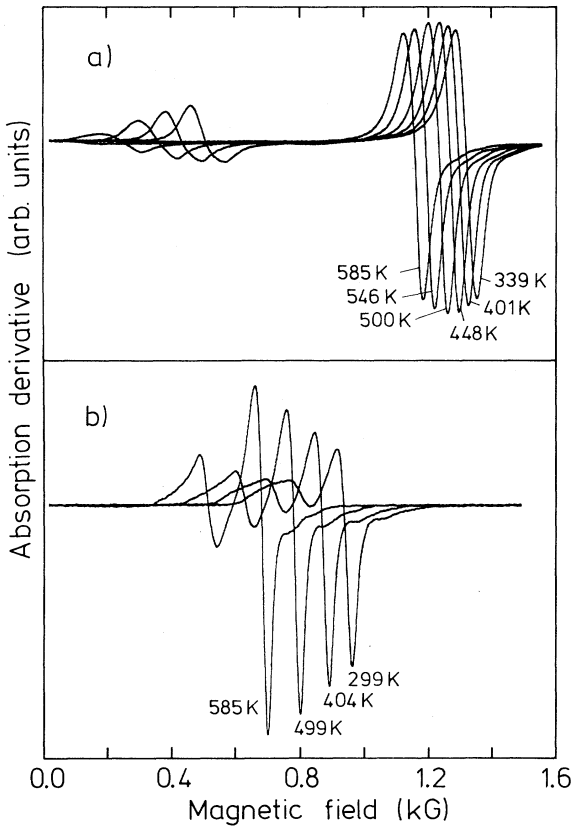

FIG. 2. Typical experimental FMR spectra of 54 Å Fe film at 9 GHz (a) and 4 GHz (b). The external field is applied in the film plane and parallel to the [001].

IV. RESULTS AND DISCUSSION

A. Angular and frequency dependence of the resonance field

To determine the magnetic anisotropy of Fe films, a series of angular dependence experiments were carried out at 9 GHz and at different temperatures. In such measurements the resonance field is determined as a function of the angle ϑ_H between the external magnetic field and the film plane (Fig. 1). An angular dependence of FMR spectra at 500 K is shown in Fig. 3. It can be seen that the resonance field is strongly dependent on the angle ϑ_H between the external field and the film plane. With decreasing the angle ϑ_H the resonance field shifts to higher fields. Typical results for the angular dependence experiments are shown in Fig. 4. The magnetic anisotropies were evaluated by fitting the angular dependence of the resonance field with Eqs. (4) and (5). The obtained results are displayed in Table I and the best fits are shown as solid lines in Fig. 4. As can be seen from Table I, the in-plane anisotropy is strongly dependent on the film thickness, which will be further discussed in Sec. IV C. The determined values of $4\pi M_{\text{eff}}$ are within 10% in agreement with the saturation magnetization $4\pi M$ of the bulk Fe at the same temperature. Similar results were also obtained by Elmers and Gradmann,[15] and Baumgart, Hillebrands, and Güntherodt.[17] This suggests that in comparison with $4\pi M$ the out-of-plane anisotropy field $2K_u/M$ of Fe films on W(110) is small. $4\pi M_{\text{eff}}$ can be replaced by $4\pi M$. This is different from the results of Krebs *et al.*,[11] where Al cover layers or oxidation of Fe(110)/GaAs(110) films led to a strong reduction of $4\pi M_{\text{eff}}$ with decreasing film thickness.

To check the determined anisotropies from the angular dependence experiments, the frequency dependence of the resonance field was also used with the external magnetic field parallel to the film plane and at different temperatures. Typical results for 54 Å Fe films at 300 and 500 K are shown in Fig. 5. From a least-squares fit with Eq. (6) and the data, we determined $4\pi M_{\text{eff}}$ and K_{eff}. In

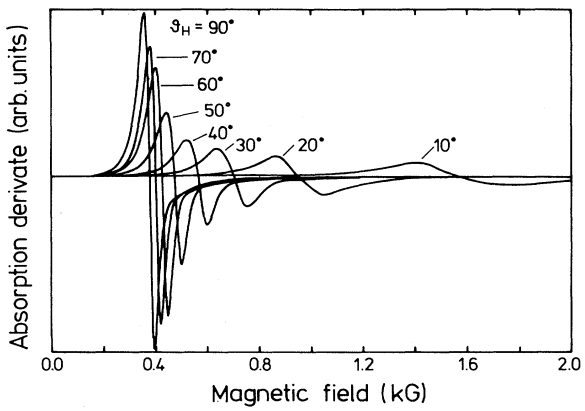

FIG. 3. FMR spectra for a 130 Å Fe film as a function of the external magnetic field orientation (ϑ_H) with respect to the film plane. The spectra were taken at 9 GHz and at 500 K.

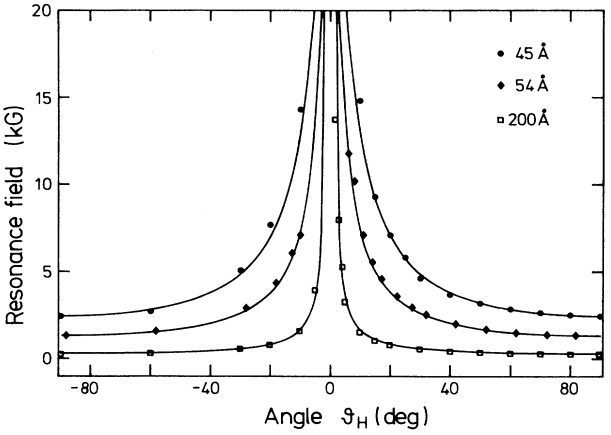

FIG. 4. Angular dependence of the resonance field. Solid curves are calculated according to Eqs. (4) and (5) and with parameters shown in Table I.

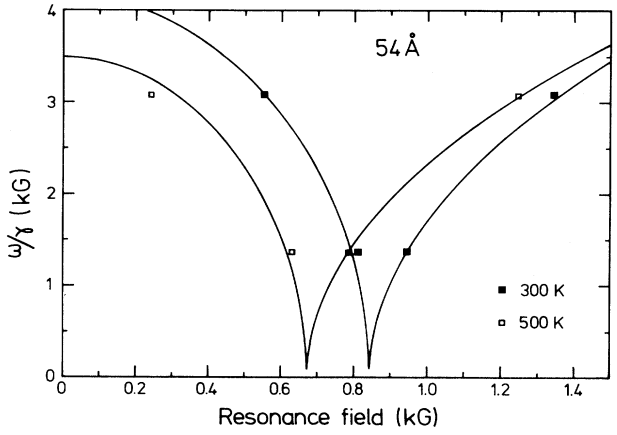

FIG. 5. FMR frequency dependence on the external magnetic field applied in the [001] axis in the (110) plane at a 54 Å Fe film grown on W(110).

agreement with the above results (Table I), we obtained $K_{\text{eff}} = -0.78 \times 10^6$ erg/cm^3 and $4\pi M_{\text{eff}} = 20.3$ kG at 300 K. Of most importance here, the magnetic anisotropies determined from two independent analyses, namely from the angular and the frequency dependences of the resonance field, agree very well. This is an indication that FMR is a useful and reliable method in determining the magnetic anisotropy of thin films.

B. Temperature dependence of the magnetic anisotropy

The temperature dependence of the resonance field H was measured between 300 and 600 K. Typical results for several Fe films are shown in Fig. 6. Using Eq. (6) and these experimental data, we can calculate the in-plane magnetic anisotropy, if $4\pi M_{\text{eff}}$ is known. Since in comparison with $4\pi M$, the out-of-plane anisotropy field is small, as shown and discussed above, we take $4\pi M$ from the literature[29] instead of $4\pi M_{\text{eff}}$ in Eq. (6) in our calculation.

The determined temperature dependence of the in-plane anisotropy is shown in Fig. 7. As we have already noted from Fig. 6, the anisotropies are seen to be divided into two distinct regions: a positive region, where the film thickness is thicker than 130 Å, and a negative region, where the film thickness is thinner than 100 Å. The crossover between these regions occurs at about 110 Å at 300 K, which is the critical thickness for the switching of the easy direction of the magnetization. The positive and negative anisotropies indicate that [001] and [$\bar{1}$10] are the easy direction, respectively. The switching thickness observed in our experiment at 300 K is larger than those obtained by Elmers and Gradmann[15] and by Braumgart, Hillebrands, and Güntherodt.[17] They have found 50–70 Å and 84 Å, respectively. This observation can be interpreted as arising from the larger in-plane surface anisotropy of our Fe films, which will be discussed in the following section. For the thinner films, where strong in-

TABLE I. Values of the effective in-plane anisotropy K_{eff} and the effective magnetization $4\pi M_{\text{eff}}$ for three Fe films obtained from a fit to Eqs. (4) and (5). This may be compared to the saturation magnetization $4\pi M = 21.54$ kG (Ref. 29).

Thickness	$K_{\text{eff}}/(10^6$ erg/cm$^3)$	$4\pi M_{\text{eff}}/kG$
45 Å	−1.89(11)	20.8(5)
54 Å	−0.77(13)	20.6(5)
200 Å	0.15(14)	20.1(5)

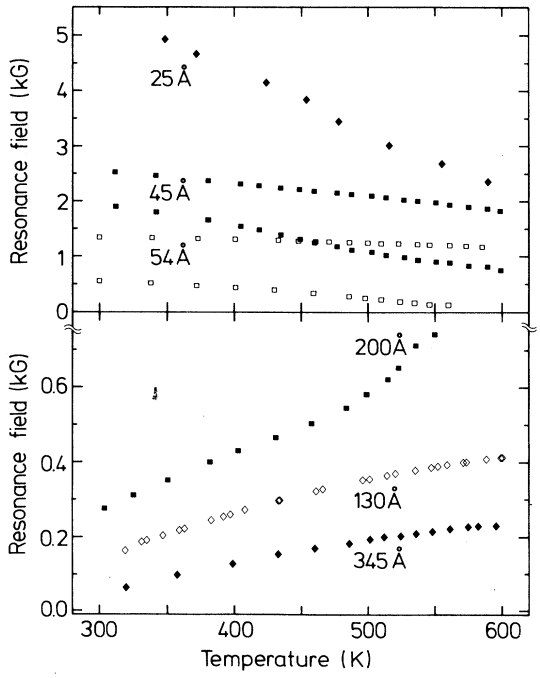

FIG. 6. Temperature dependence of the resonance field for six films (Ref. 35). The external magnetic field is applied parallel to the [001] axis in the film plane. Note that for 45 and 54 Å two resonance lines are detected as described in the previous section.

plane anisotropies due to surface anisotropy contributions are present, a nearly linear decrease of the anisotropy is observed. The thicker films show a behavior that is quite similar to the bulk (cubic anisotropy K_1).

The sample (200 Å), which was grown at the substrate temperature of 600 K, shows a quite different behavior from those grown at 300 K seen in Fig. 7. The magnetic anisotropy of this film is much smaller than that of 130 and 345 Å films. We explain this behavior as due to the influence of the substrate temperature during the evaporation on the magnetic anisotropy. It is also interesting to note in Fig. 7 that the magnetic anisotropy of this film changes from positive to negative values with increasing temperature. This indicates that the easy direction of the magnetization switches from [001] to [$\bar{1}$10] direction with changing temperature. The reason for this behavior is a different temperature dependence of K_1 and K_u.[30]

C. Surface and volume anisotropy

So far, we have presented the temperature and thickness dependences of the in-plane magnetic anisotropy. It is also interesting to further analyze this anisotropy in the surface and volume contributions. According to a phenomenological approach described by Gradmann,[31] one can express the effective in-plane anisotropy as

$$K_{\text{eff}} = K_v + \frac{K_s}{d} , \tag{7}$$

where K_v and K_s are the volume and surface in-plane anisotropies, respectively. d is the film thickness. A least-squares fit for Eq. (7) and the experimental data for the in-plane anisotropy, determines K_v and K_s. Plots of K_{eff} versus $1/d$ at two temperatures are shown in Fig. 8. The solid curves are the best fits. It is seen in Fig. 8 that our data are in reasonable agreement with the $1/d$ linear relation (7). The obtained results for K_s and K_v at different temperatures and the available data for Fe films on W(110) from Refs. 15 and 17 are listed in Table II. To our knowledge it is the first time that the temperature dependence of the surface anisotropy of Fe(110) films is determined in this temperature range.

As seen in Table II, the agreement between the volume contribution and the cubic anisotropy is quite good, which is a confirmation of the consistency of our data. The surface anisotropy deduced in our experiments is slightly larger than the values obtained by Elmers and Gradmann[15] and by Baumgart, Hillebrands, and Gütherodt.[17] This difference may be accounted for by the difference in the structure of the surface. Our Fe films were grown at 300 K and were not in a perfect single crystalline state resulting in a rather rough surface. According to the structure study of Fe films on W(110),[32,33] Fe films grown at 300 K have a surface with some step structures. By contrast, Fe films grown at 500~600 K have a flat surface. The in-plane surface anisotropy of Fe films with a stepped surface should be larger than that with a flat surface. This was also confirmed experimentally by Jian Chen and Erskine[34] and Albrecht and co-workers.[21,22] The quantitative comparison of our results with those of Elmers and Gradmann, and Baumgart, Hillebrands, and Güntherodt requires a knowledge of the complete surface structure, which is not yet available in the present work.

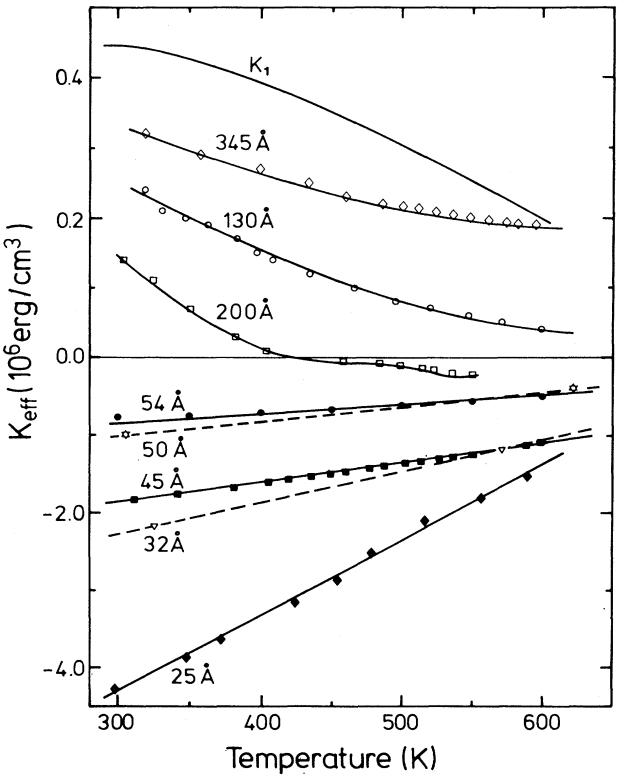

FIG. 7. The effective in-plane anisotropy K_{eff} as a function of temperature for Fe films (Ref. 35). For comparison, the cubic anisotropy K_1 of Fe bulk is also shown. Note the different scale for the positive and negative part.

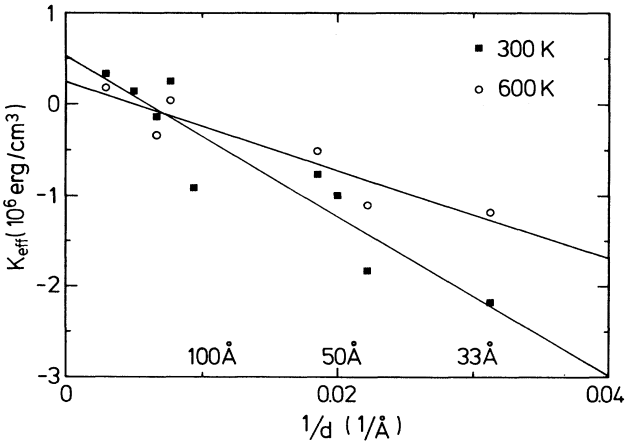

FIG. 8. The effective in-plane anisotropy K_{eff} obtained from the experimental data as a function of the reciprocal film thickness $1/d$ for 300 and 600 K.

TABLE II. Experimental data for the effective in-plane surface anisotropy K_s and the in-plane volume anisotropy K_v at different temperatures. For comparison, the data from Refs. 15 and 17 are also included. K_1 is the cubic magnetic anisotropy of bulk Fe and is taken from Ref. 29.

T (K)	K_s (erg/cm^2)	K_v (10^6 erg/cm^3)	K_1 (10^6 erg/cm^3)	Ref.
300	−0.88(15)	0.53(30)	0.45	This work
400	−0.83(15)	0.43(25)	0.40	This work
500	−0.68(10)	0.30(20)	0.30	This work
600	−0.48(10)	0.25(15)	0.20	This work
300	−0.45(5)	0.65(3)	0.45	15
300	−0.68(8)	1.21(8)	0.45	17

V. SUMMARY

The ferromagnetic resonance in UHV has been employed to investigate the magnetic properties of thin Fe films on W(110). The angular, frequency, and temperature dependences of the resonance field were studied to determine the out-of-plane and in-plane anisotropies in the temperature range between 300 and 600 K. The magnetic anisotropies were found to vary with the temperature, the film thickness and the preparation conditions. The switching of the easy axis of the magnetization from [$\bar{1}$10] to [001] was observed at about 110 Å at 300 K, which confirms the previous observations on Fe films on GaAs (Ref. 13) and W(110) (Refs. 15 and 17). The thickness dependence of the in-plane anisotropy at different temperatures was used to deduce the surface and volume anisotropies and their temperature dependence using the well-known relation of Eq. (7). A decrease of the surface and volume anisotropies was found as the temperature was increased. The volume anisotropy agrees very well with the known cubic anisotropy for bulk Fe. Furthermore, the switching of the easy axis of the magnetization in the film plane was observed for the 200 Å film by changing the temperature. In conclusion, UHV-FMR has been used successfully to learn about the details of the magnetic anisotropies of thin iron films.

ACKNOWLEDGMENTS

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