

## Magnetic and structural properties of thin Fe films grown on Ni/Si

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Fe films ranging in thickness from 6 to 90 Å have been prepared on Ni layers with constant thickness of 140 Å grown on Si(100) in ultrahigh vacuum. Magnetic properties have been studied by spin-polarized neutron reflection and superconducting quantum interference device magnetometry and structural properties have been investigated by small-angle x-ray reflectometry and reflection high-energy electron diffraction. As the main result of this work it is shown that the magnetic states of the Fe films strongly depend on their thicknesses. For Fe layers  $\leq 32$  Å, the average magnetic Fe moments come out to be very low around  $0.2\mu_B$ . For Fe layers  $\geq 60$  Å, the Fe moments increase by a factor of about 10 close to the value known for bulk iron. Most probably these changes can be attributed to a structural phase transition of the Fe films from fcc-like to bcc with increasing Fe layer thickness. Fe films below 32 Å are either antiferromagnetic or exhibit almost vanishing Fe moments. The discussion includes a comparison of our results for Fe/Ni bilayers with results published for Fe/Ni multilayers and for Fe films on Cu.

### I. INTRODUCTION

The structure and magnetic properties of magnetic thin films and multilayers have attracted much attention in recent years.<sup>1-4</sup> Extensive theoretical and experimental studies have been focused on the structural and magnetic behavior of face-centered-cubic (fcc) and body-centered-cubic (bcc) iron films, in particular for Fe/Cu systems.<sup>2,5-9</sup> Below about ten monolayers, Fe films grown on Cu show fcc-like phases with various magnetic states including a low moment antiferromagnetic state or a high moment ferromagnetic state. Above about ten monolayers, Fe films on Cu are usually bcc structured and ferromagnetic. The magnetic properties of Fe/Cu films sensitively depend on lattice parameters, symmetry, and film growth conditions. In spite of the many investigations, the correlation between the complex magnetic and structural phases is still a matter of debate.

Partly similar interesting properties might be expected for Fe layers grown on Ni and for Fe/Ni multilayers. Recent studies of structure and magnetic properties of  $Fe_n/Ni_n$  multilayers yielded partly conflicting results.<sup>10-14</sup> It has been reported<sup>10-13</sup> that for layer thicknesses  $d > 40$  Å ( $d_{Fe} = d_{Ni}$ ) Fe layers exhibit bcc and Ni layers fcc structures, whereas for  $d < 20$  Å both have fcc-like structures. In several works<sup>11-14</sup> a ferromagnetic high moment state has been found for fcc Fe films in  $Fe_n/Ni_n$  multilayers, while in Ref. 10 a reduced averaged Fe moment has been reported for such systems.

This conflicting situation has been one of the reasons for our study of magnetic properties of Fe/Ni bilayers grown on Si substrates. Thin Fe layers grown on Ni offer the chance to stabilize fcc Fe which is predicted to support a complex magnetic behavior ranging from nonmagnetic, antiferromagnetic, or ferromagnetic spin structures.<sup>2</sup> The nearest-neighbor exchange interaction and also the magnitude of the Fe moments in fcc Fe are assumed to be very structure sensitive.<sup>2,15</sup> Moreover, at certain film thicknesses fcc Fe can undergo a phase transition to bcc Fe with corresponding changes of magnetic properties. Motivated by these problems we have studied properties of thin Fe layers on Ni layers with con-

stant thickness of 140 Å as a function of Fe layer thickness in the range 6–90 Å. Magnetic properties have been studied by spin-polarized neutron reflectometry (SPNR) and superconducting quantum interference device (SQUID) magnetometry. In difference to a conventional magnetometer, SPNR can probe the depth dependence of the magnetization or averaged magnetic moment.<sup>16-19</sup> This permits a layer by layer determination of magnetic moments of thin films having two or more different magnetic layers, e.g., Fe/Ni bilayers. The observed magnetic behavior of the Fe films strongly depends on the thickness of the Fe layers and will be compared to results known for Fe/Ni multilayers and for Fe/Cu systems. In difference to a Cu substrate, the Ni substrate has a smaller lattice parameter and is ferromagnetic, the latter can induce magnetic polarization and coupling effects between the Ni and Fe layers.

### II. SAMPLES AND EXPERIMENTS

A series of Fe/Ni bilayers have been grown on oxidized Si(100) substrates by electron-beam evaporation in an ultrahigh vacuum system with a base pressure of  $1 \times 10^{-9}$  mbar. The substrate temperature has been maintained at about 300 K. The deposition rate has been 0.4 Å/s for Ni and 0.2 Å/s for Fe. Film thicknesses have been determined by a calibrated quartz microbalance. The thickness of the Ni layers has been kept constant at 140 Å and the thickness of the Fe layers was varied from 6 to 90 Å. To prevent oxidation of the Fe layers for *ex situ* measurements, the bilayers have been covered with an Au layer.

Structural investigations of the layer systems have been performed *in situ* during the growth by reflection high-energy electron diffraction (RHEED) and *ex situ* by small-angle x-ray reflectivity and transmission-electron microscopy (TEM). Measurements by TEM and RHEED proved the polycrystalline structure of the samples. During the growth we have observed by RHEED for the Ni films ring patterns with a structure similar to the pattern known for polycrystal-

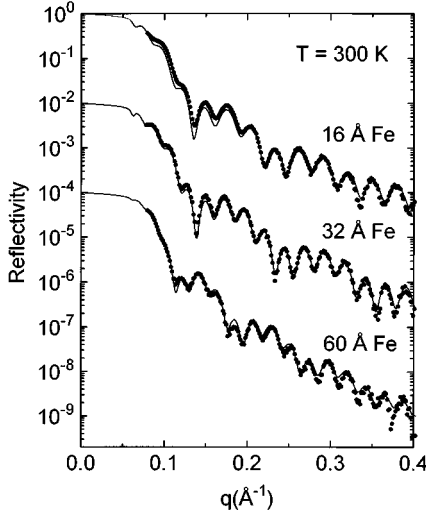


FIG. 1. X-ray reflectivities of three different Fe layers grown on Ni/Si (100) and covered with Au. The solid lines represent the best fits. The resulting layer thicknesses are collected in Table I. For clarity the plots of the data for the Fe(32 Å) and Fe(60 Å) are shifted on the vertical axis.

line fcc Ni in electron-diffraction experiments using the TEM technique. For Fe films thicker than 60 Å on Ni, RHEED yielded a ring pattern typical for bcc iron. For Fe films thinner than 32 Å a clear change of the ring patterns compared to the thicker Fe films has been observed but we have not been able to derive a conclusive identification of the structure (presumably fcc-like as discussed below) of these Fe films.

X-ray reflectivity measurements were performed to control the film thicknesses and to measure roughness and interfacial properties. Figure 1 shows the reflectivities of three samples, which were measured with  $\text{CuK}\alpha$  x-ray radiation. The solid lines in Fig. 1 represent the fits using genetic algorithm and least-squares fitting methods, which are described in detail in Ref. 20. The fits to the experimental data confirm the layers to have a well layered structure with a small roughness at the interfaces (2–6 Å). The resulting thicknesses are collected in Table I and compared to the nominal layer thicknesses determined by the quartz microbalance. As can be seen in Table I, the agreement is quite satisfying. To characterize samples in the text and in figures we use the nominal layer thicknesses  $d_n$ .

We now turn to the description of the measurements of magnetic properties. By SQUID magnetometry hysteresis loops were measured for all nine samples in the temperature range of 5 to 300 K in an external field  $H$  applied parallel and perpendicular to the film plane. Furthermore, the temperature dependence of the susceptibility has been investigated for selected samples. A short report of parts of the SQUID measurements has been given in Ref. 21.

Spin-polarized neutron reflectivity has been proven to be a very useful method for determining magnetic moments and magnetization profiles;<sup>16–18</sup> its sensitivity is sufficient to determine the absolute moment of Fe films in the monolayer range.<sup>19</sup> The SPNR measurements were performed at the reflectometer POSY at the Intense Pulsed Neutron Source at

TABLE I. Summary of the layer thicknesses of the samples as determined by the calibrated quartz microbalance,  $d_n$  (referred to as nominal thicknesses in the text), and by x-ray reflectivity measurements,  $d_r$ .

| Au layer |       | Fe layer |       | Ni layer |       |
|----------|-------|----------|-------|----------|-------|
| $d_n$    | $d_r$ | $d_n$    | $d_r$ | $d_n$    | $d_r$ |
|          | (Å)   |          | (Å)   |          | (Å)   |
| 60       | 59    | 6        | 7     | 140      | 139   |
| 60       | 60    | 10       | 13    | 140      | 139   |
| 60       | 57    | 16       | 17    | 140      | 135   |
| 60       | 54    | 24       | 24    | 140      | 161   |
| 60       | 55    | 32       | 26    | 140      | 171   |
| 70       | 77    | 40       | 49    | 140      | 154   |
| 70       | 74    | 60       | 53    | 140      | 143   |
| 80       | 82    | 80       | 84    | 140      | 159   |
| 60       | 62    | 90       | 87    | 140      | 147   |

the Argonne National Laboratory<sup>22</sup> and at the reflectometer V6 at the research reactor BER II at the Hahn-Meitner-Institut, Berlin.<sup>23</sup> The former works at a constant angle,  $\theta$ , between the incident beam and the sample surface. The reflected neutrons are measured at  $2\theta$  in a range of neutron wavelengths. The neutron wavelength,  $\lambda$ , is determined by the time of flight between source and detector. The reflectometer in Berlin works with a monochromatic neutron beam at a fixed wavelength of 4.60 Å and the reflected neutrons are detected as a function of  $2\theta$ . At both reflectometers the reflectivity, defined by normalizing the intensity of the reflected neutrons to the neutron flux, has been determined as a function of the momentum transfer of the neutrons,  $q = 4\pi\lambda^{-1}\sin\theta$ . All SPNR measurements were performed at 300 K in an external magnetic field parallel to the surface of the samples. The incident neutrons were spin polarized parallel or antiparallel to the magnetic field.

For magnetic materials the reflectivity depends on the spin orientation of the neutrons. The spin-dependent optical potential seen by the neutrons in a medium can be written as:<sup>24</sup>

$$V^\pm = (2\pi\hbar^2/m)(bN \pm CB), \quad (1)$$

where  $b$  is the average nuclear scattering amplitude of the medium and  $N$  the average atomic density.  $C$  is a constant and  $B$  the magnetic induction in the medium, which results from ordered atomic magnetic moments. The signs in Eq. (1) refer to the spin orientation of the neutrons parallel (+) or antiparallel (–) relative to the external magnetic field. In scattering experiments the component of the neutron momentum perpendicular to the surface,  $k_0 = 2\pi\lambda^{-1}\sin\theta$ , is modified by the optical potential Eq. (1), so that<sup>24,25</sup>

$$k_z^\pm = [k_0^2 - 4\pi(bN \pm CB)]^{1/2}. \quad (2)$$

This modified neutron momentum enters into the neutron wave functions. The resulting reflectivity  $R^\pm(q)$  can be calculated by solving the Schrödinger equation.<sup>26</sup> By fitting the measured reflectivity, one can determine magnetic moments, magnetization profiles, film thicknesses and average atomic densities.

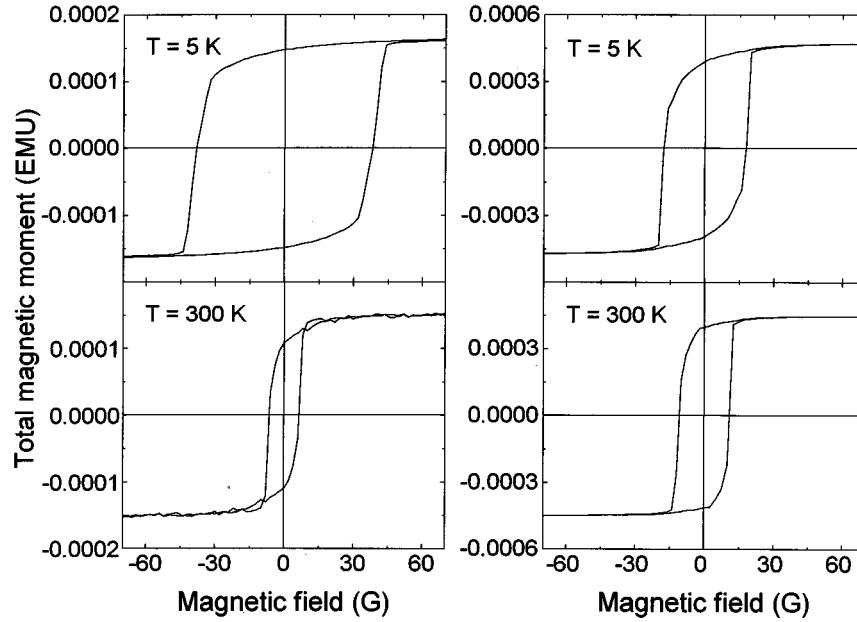


FIG. 2. Magnetic hysteresis loops at different temperatures for Fe/Ni bilayers measured by SQUID magnetometry in an external field parallel to the film plane. The results refer to the layer systems given in Table I with Fe layer thicknesses of 24 Å (left) and 60 Å (right side).

### III. RESULTS AND DISCUSSION

Typical examples of the hysteresis loops are displayed in Fig. 2. The magnetization of the bilayers was found to be saturated at about 100 G at 5 K and to be oriented in the film plane for both the Fe (if magnetic) and the Ni layers. The hysteresis loop measurements allow the extraction of the total magnetic moments for all Fe/Ni bilayers. Since the Ni layer thickness has been kept constant, the observed changes of the total averaged magnetic moment can be correlated to the change of the Fe layer thickness (see Fig. 2 for the two selected examples). For Fe films of 32 Å or less, there is almost no contribution of the Fe atoms to the total magnetization observed. The magnetic moment of the Ni layers is found to be  $0.5\mu_B$ , which is close to that of the bulk.

Figure 3 shows the spin-dependent reflectivities of three samples, which were measured at 300 K in an external field of 800 G, well within the region where the magnetization is saturated. The large differences between the (+) and (−) spin reflectivities in Fig. 3 arise from the magnetizations of the Ni and Fe layers. The reflectivities are found to vary strongly with the thickness of the Fe layers. The variation of the period of these oscillations provides an additional possibility to determine the thicknesses of the films. The details of the structure are strongly modified by magnetic scattering. For  $d_{\text{Fe}} \leq 32$  Å the (+) and (−) reflectivities track each other, while for  $d_{\text{Fe}} \geq 60$  Å they are out of phase.

A quantitative analysis of the experimental data has been performed by the same fitting procedure used in the analysis of the x-ray reflectivities.<sup>20</sup> In the first fitting step, the parameters for the thicknesses and roughnesses of the Au, Fe, and Ni films have been fixed to the values determined from the quartz microbalance and the x-ray reflectivities and further the bulk densities have been taken as input parameters. Then

the only remaining fitting parameters are the average magnetic moments of the Fe and Ni layers. As the next step of the fitting procedure, we have taken the thicknesses and the nuclear scattering amplitude densities of the three layers as free parameters. Compared to the first step, the second step yields almost the same values for the magnetic moments of the Fe and Ni layers and for the total thicknesses

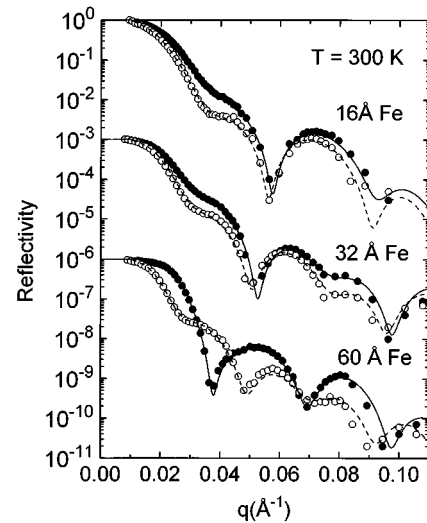


FIG. 3. Spin-polarized neutron reflectivities of three layer systems with varying Fe layer thickness grown on Ni/Si and covered with Au (see Table I for the individual thicknesses). The solid and open circles represent the reflectivities for the neutron spin parallel and antiparallel to the external magnetic field, respectively. The solid lines are the best fits. The plots of the data for the Fe(32 Å) and Fe(60 Å) are shifted on the vertical axis.

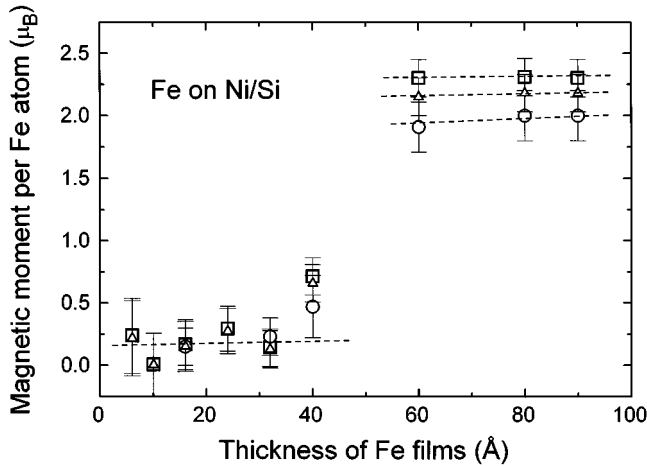


FIG. 4. Average magnetic moment per Fe atom as a function of the Fe layer thickness. The circles refer to data obtained by the SPNR measurements at 300 K. The triangles and squares represent results obtained by the SQUID measurements at 300 and 5 K, respectively. The dashed lines are guides to the eye.

( $d_{\text{Au}} + d_{\text{Fe}} + d_{\text{Ni}}$ ) of the samples. In the second fitting step, some variations of the relative thicknesses and the nuclear scattering amplitude densities are found. The optimized fits are represented in Fig. 3 as solid lines.

The averaged magnetic Fe moments as a function of Fe thickness are shown in Fig. 4. In all samples studied we have found a constant magnetic moment of the Ni layers of  $0.5\mu_B$ , which is apparently independent of the Fe film thickness. In contrast, the averaged magnetic Fe moments vary dramatically with the Fe layer thickness. The data of the SQUID measurements agree very well with the SPNR results (see Fig. 4). The quantitative agreement of these complementary results confirms the thickness dependence of the averaged magnetic moment of the Fe layers. For Fe layers of 32 Å or less, the averaged magnetic moment of the Fe layers—as deduced consistently from both the SPNR and SQUID measurements—comes out to be  $0.2 \pm 0.2\mu_B$  per Fe atom and can be zero within the experimental error. For the thinnest Fe layer, which is investigated by SQUID magnetometry only, the error increases to about  $0.3\mu_B$ . For Fe layers of 60 Å or more, the Fe moment is found to be  $2.0\mu_B$ , which is close to that of bulk bcc iron. In the region from about 40 Å to maximal 60 Å, we observe a drastic increase of the averaged Fe moment by about a factor of 10. Such a sharp transition from a very low to a high moment state as a function of the Fe layer thickness has not been observed for Fe layers on Ni before.

First we would like to discuss if the observed magnetic Fe behavior can be caused by the formation of Fe-Ni interfacial alloys. The averaged magnetic moments of Fe-Ni alloys increase with Fe concentration and are known<sup>27</sup> to be much larger than the moments observed in the Fe films of 32 Å or less. On this basis one can exclude a significant influence of interfacial Fe-Ni alloys on the very low to high Fe moment transition observed. In agreement with this expectation a trial to analyze the reflectivities under the assumption of the presence of Fe-Ni interfacial alloys resulted in worse fits than those shown in Fig. 3.

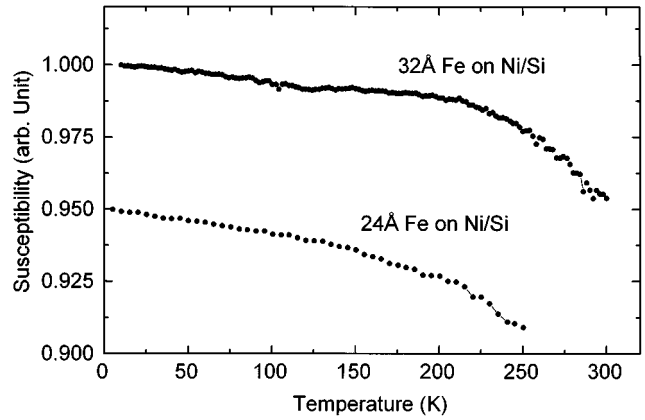


FIG. 5. Susceptibility,  $\chi$ , as a function of temperature for the Fe(32 Å)/Ni(140 Å) and Fe(24 Å)/Ni(140 Å) bilayers, normalized to the values at 5 K. For clarity the plot of the data for the Fe(24 Å)/Ni(140 Å) is shifted on the vertical axis.

One is led to conclude that in the Fe films of 32 Å or less, the majority of the Fe atoms are either almost nonmagnetic or in an antiferromagnetic state. As a trial to test the presence of antiferromagnetic Fe, we have performed susceptibility measurements by SQUID magnetometry in order to find an indication of a phase transition from antiferromagnetic to paramagnetic in the temperature range of 5 to 300 K. In analogy to antiferromagnetic (fcc-like) Fe films on Cu with Néel temperatures between 60 and 200 K<sup>8,28</sup> it does not seem unreasonable to expect the Néel temperature of a possible antiferromagnetic Fe film on Ni to lie in the range of measurement. Because of possible interdiffusion between the Fe and Ni layers above 300 K, the susceptibility measurements were not extended to higher temperatures. In Fig. 5 the temperature dependence of the susceptibility observed in a field of 100 G is plotted for the Fe(32 Å)/Ni(140 Å) and Fe(24 Å)/Ni(140 Å) films. The monotonic decrease of the susceptibility with increasing temperature is due to the decrease of the magnetization of the Ni layer alone, which follows from a comparison with the susceptibility measured for a Au/Ni(140 Å)/Si(100) layer without an iron film. The data in Fig. 5 show no indication for a peak in the susceptibility which would be typical for a phase transition from antiferromagnetic to paramagnetic. At present it is hard to say if this negative result is conclusive since the sensitivity of this measurement on such a Fe phase transition strongly depends on the (poorly known) enhancement of the Fe spin polarization induced by the ferromagnetic Ni layer. Therefore, we continue to interpret the observed low Fe moments as being due to an antiferromagnetic or an almost nonmagnetic state.

Previous studies of  $\text{Fe}_n/\text{Ni}_n$  multilayers consistently yielded the information that Fe layers with a thickness less than about 20 Å crystallize in fcc-like structures.<sup>10–13</sup> Along with the analogy to the fcc-like growth of very thin Fe layers on Cu, it is plausible to assume that our thin Fe films with very low moments are fcc-like structured, also. As observed by our RHEED experiments the Fe layers thicker than 60 Å change to the bcc structure, consistent with previous observations in  $\text{Fe}_n/\text{Ni}_n$  multilayers. All these features provide strong arguments that the sharp increase of the Fe mo-

ment in the range of 32 to 60 Å can be attributed to a structural phase transition of the Fe layers from fcc-like to bcc structure with increasing  $d_{\text{Fe}}$ . We note that these structural and magnetic phase transitions are also accompanied by a large change in the magnetic anisotropy, which we have measured by SQUID magnetometry.<sup>29</sup>

Of special interest are the very low averaged moments observed in the thinner (32 Å or less) Fe films which we assume to be fcc-like structured. Considerable theoretical effort has been devoted towards an understanding of the complex magnetic states of fcc iron.<sup>2,15</sup> Extremely structure sensitive around a lattice parameter very close to that of Cu, fcc Fe is predicted to be in a nonmagnetic, antiferromagnetic, or ferromagnetic state. Very recently, Kraft *et al.*<sup>5</sup> performed calculations of magnetic properties of fcc Fe films grown on Cu(100) with the result that for 4 up to 11 Fe layers the surface and first subsurface layers couple ferromagnetically, whereas the deeper ones show interlayer antiferromagnetic coupling. Essential parts of these predictions seem to be consistent with experiments.<sup>6-9</sup> For Wigner-Seitz radii slightly larger than Cu, the Fe is predicted to change to the more stable ferromagnetic state,<sup>2,5,15</sup> which is confirmed by the finding of high moment ferromagnetism for fcc Fe films grown on CuAu(111) (Ref. 30) and on CuAu(100) (Ref. 31). Compared to pure Cu, the addition of Au expands the lattice parameter of the substrate and the fcc Fe film. Now it is interesting to note that in the present experiment the magnetic behavior of fcc Fe films is tested under compression. Assuming the lattice parameter of our relatively thick Ni layer to be close to its bulk value of  $a=3.524$  Å, the lattice parameter of the Ni substrate is 2.6% smaller compared to Cu with  $a=3.615$  Å. By all these considerations one is led to expect for thin Fe films grown on Ni a more unstable magnetic behavior towards the antiferromagnetic or even nonmagnetic direction. In fact, our experimental results confirm this expectation. We can conclude that the overwhelming majority of the Fe atoms in the thinner films are in an antiferromagnetic or almost nonmagnetic state; possible contributions of high spin ferromagnetism, e.g., from interfaces, come out to be small. It also follows that the amount of possible bcc Fe precipitates (compare Ref. 31) in our very low moment Fe films is less than about 15%. The upper limit of possible ferromagnetic contributions to the magnetization for our thinnest Fe films  $<20$  Å can be estimated to be smaller than that found for Fe films  $<20$  Å on Cu<sup>6-9,31</sup> and for Fe/Cu multilayers.<sup>32,33</sup>

Conflicting with the expectation and our results described above, in most of the previous investigations essentially high moment ferromagnetism has been reported for the fcc-like thin Fe films in Fe/Ni systems.<sup>11-13</sup> Edelstein *et al.*<sup>12</sup> prepared  $\text{Fe}_n/\text{Ni}_n$  multilayers in the range of thicknesses from 8 to 23 Å ( $d_{\text{Fe}}=d_{\text{Ni}}$ ) by ion-assisted sputtering. By extended x-ray-absorption fine structure (EXAFS) at the Fe K edge and by x-ray diffraction, the structure of these Fe films has been determined to be fcc-like and the averaged Fe moment of the ferromagnetic films has been determined to be  $2\mu_B$ . As an explanation for the observed stabilization of the magnetization, relative to fcc-like Fe films on Cu, Edelstein *et al.* suggest a magnetic coupling between the Fe and Ni layers, which enhances the magnetization of the Fe films. In the light of the present results such an explanation is strongly

disfavored. One might speculate that the different magnetic results might be related to the different methods of film preparation and growth conditions. Ferromagnetic fcc-like Fe films have been found in Fe/Ni multilayers prepared by various sputtering methods<sup>11,12,14</sup> and by thermal evaporation at room temperature in a vacuum of  $10^{-6}$  mbar.<sup>13</sup> Low averaged Fe moments have been found in films prepared under ultrahigh vacuum conditions around  $10^{-9}$  mbar (Ref. 10, this work). In contrast to the present work, Jennett and Dingley<sup>10</sup> observed a rather progressive reduction of the averaged Fe moment with decreasing Fe layer thickness in the range of 15 to 3 Fe layers. Another difference between the bilayers of the present work and the multilayers investigated previously is that in the multilayers one usually expects thickness dependent changes of structural parameters of the fcc phases for both the Fe and the Ni layers (see Refs. 12-14).

#### IV. SUMMARY

We have investigated structural and magnetic properties of thin Fe films in the range of 6 to 90 Å grown on Ni/Si in ultrahigh vacuum. The polycrystallinity of the layer systems have been proved by RHEED and TEM. X-ray reflectivity measurements revealed that the films have well layered structures and small roughnesses at their interfaces. *In situ* RHEED studies and the close analogy to the growth of Fe films known for Fe/Ni multilayers and Fe/Cu systems, provide strong arguments that our Fe films are (predominantly) fcc structured for film thicknesses  $\leq 32$  Å and bcc structured for films  $\geq 60$  Å.

Special effort has been spent for the investigation of magnetic properties of the Fe/Ni bilayers by SPNR and SQUID measurements. The observed magnetic transition from a very low moment to a high moment ferromagnetic Fe state is almost certainly correlated to a structural transition from fcc-like to bcc structured Fe films. The surprisingly small Fe magnetization observed for (fcc-like) Fe films  $\leq 32$  Å cannot be explained by interfacial Fe-Ni alloy formation, but has to be interpreted as being due to an antiferromagnetic or almost nonmagnetic Fe state. The magnetic behavior in these films is consistent with extensive theoretical predictions performed for fcc Fe systems with lattice parameters around or smaller than 3.6 Å.

The results of this work have been compared to results known for the closely related systems Fe/Ni multilayers and Fe films grown on Cu. The high moment ferromagnetism observed in most works for thin fcc Fe films in Fe/Ni multilayers represent, at least partly, a conflicting situation compared to the results of the present work and also to the results known for fcc-like Fe films on Cu. From the results of the present work we can conclude that a possible enhancement of the Fe spin polarization due to the ferromagnetic Ni layer is too small to produce the high moment ferromagnetic state in our Fe films  $\leq 32$  Å.

Besides being ferromagnetic, the smaller lattice parameter of the Ni substrate represent another important difference compared to Fe layers grown on Cu. Whereas some gross features of the results of the present work, e.g., the transition

from low to high Fe magnetization along with the structural fcc-like to bcc transition, seem to be similar to the behavior of Fe films on Cu, it is still highly interesting to investigate the structural and magnetic properties and also the growth of Fe films on Ni in more detail. In particular because of the significantly smaller lattice parameter of the Ni substrate, such investigations can be expected to yield insight into the extremely structure-sensitive and complex magnetic states of fcc-like Fe films.

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