Diss. ETH Nr. 12092

STRUCTURAL AND MAGNETIC INVESTIGATIONS OF ULTRATHIN MICROSTRUCTURES

A dissertation submitted to the
SWISS FEDERAL INSTITUTE OF TECHNOLOGY ZÜRICH

for the degree of
Doctor of Natural Sciences

presented by

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1997
"Alles Leben ist Leiden"

Buddha
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Abstract

It is the aim of this Dissertation to establish a new field of Physics, namely the magnetism of ultra-thin microstructures. While the magnetism of atomically thin films on top of a non-magnetic substrate is well established, we do not know how the Physics looks like when we confine such films laterally to geometries on the micron scale and below.

The problem to be solved is how to produce and characterise well defined and clean magnetic microstructures with an atomic scale thickness. Solving this rather subtle material science problem was the true scope of this dissertation.

For this aim we have developed a self-made Scanning Tunnelling Microscope, which is the essential tool in both characterising the growth and the morphology of ultra-thin films and microstructures. As an example of the crucial role of this instrument, we have now a clear picture of the morphology of various ultra-thin films including Co/Cu(001) and Fe/W(110). We are now able to state that layer-by-layer growth in these films means formation of flat, two-dimensional islands growing two-dimensionally together when more and more atoms arrive. On the scale of such islands (about 100 Å) these films are, at least in the initial stage of growth, perfect.

Yet, an atomic resolution experiment reveals atomic sized defects (about 10% of an atomic layer), which have never been seen before and which could be important in determining the magnetic functioning of these films. These defects are so new and unexpected that we are still in the process of characterising them.

The knowledge of metal epitaxy acquired during this study serves as the basis for the process of microstructuring. We have succeeded in developing a novel mask technique which allows to induce microsized holes in a thin metal foil with virtually all possible patterns we wish. These foils are placed between the Molecular Beam Epitaxy source in Ultra High Vacuum and the substrate. As a consequence, mesoscopic ultra-thin islands with geometry at will can be produced in-situ, where magnetism has a chance to survive, see Fig. 1.

As a first result, we were able to switch independently islands as small as about 2 micrometers and as close as 5 micrometers. Moreover, we were able to study the behaviour of shape anisotropy versus crystalline anisotropy in two-dimensional systems. The result is that a long stripe of Fe on W(110) is always magnetized along the axis specified by the magnetocrystalline anisotropy. In contrast, the shape anisotropy wins for a similar
stripe of Co/Cu(001), where magnetocrystalline anisotropy is weaker. The possibility of controlling the various anisotropies opens a wide future for manipulating magnetic properties and observing some unusual phenomena which should occur when the strength of these interactions is comparable (see e.g. the reorientation transition in extended ultrathin magnetic films). While the feasibility of microstructuring is, to our opinion, the main result of this Dissertation, these experimental observations already reveal the new Physics behind ultra-thin microstructures.

Fig.1: 160 x 70 (μm)$^2$ scanning Kerr image of Fe islands with a diameter of about 4 μm distributed 'at random'.
Zusammenfassung


Um dieses Ziel zu erreichen, wurde von uns ein Rastertunnelmikroskop entwickelt, mit welchem wir das Wachstum und die Morphologie der ultradünnen Filme und der Mikrostrukturen charakterisiert haben. Die Wichtigkeit eines solchen Instruments zeigt sich darin, dass wir nun genaue Kenntnisse über das Wachstum verschiedener dünner Filme besitzen, wie die hier dokumentierten Systeme Co auf Cu(001) und Fe auf W(110). Wir wissen nun, dass lagenweises Wachstum für diese Filme bedeutet, dass flache, zweidimensionale Inseln durch Anlagern von weiteren Atomen am Rand zweidimensional zusammenwachsen. Innerhalb der Grössenordnung solcher Inseln (ungefähr 100 Å) sind diese Filme zumindest zu Beginn des Wachstums perfekt.


Die während dieser Studie erlangten Kenntnisse über Metall-Epitaxie gaben uns das Basiswissen für die nun zu erforschenden Mikrostrukturen. Eine neuartige Technik zur Herstellung von Masken, welche wir hier erfolgreich entwickelt haben, erlaubt es uns, dünne Metallfolien mit Löchern von der Größe eines Mikrometers herzustellen, wobei die Anordnung dieser Löcher in der Folie beliebig sein kann. Solche Masken werden in die Ultrahochvakuumkammer eingeschleust und zwischen die Aufdampfquelle und die Probe gesetzt. Damit können wir nun beliebige mesoskopische ultradüne Inseln in situ herstellen, in einem Umfeld, wo der Magnetismus solcher Inseln eine Überlebenschance hat (Fig.1).
Summary

The main aim of this dissertation is to investigate the epitaxial growth and the magnetic properties of ultra-thin microstructures. This will be reported for the two magnetic model system Co on Cu(001) and Fe on W(110).

A new ultra high vacuum instrument allowing in situ Kerr microscopy and Scanning Tunnelling Microscopy (STM) is described in section 1. The Kerr microscope has a spatial resolution of about 1 μm.

In section 2, the growth of Co on a stepped and on a flat Cu(001) surface is investigated with STM, Auger Electron Spectroscopy (AES), and Low Energy Electron Diffraction (LEED). Island shape and size differs drastically for Co evaporated onto a flat or a stepped Cu(001) surface. Deviations from ideal layer by layer growth are small. Evaporating at 540 K results in an island free surface, annealing to 495 K induces depressions into the Co islands but does not change their shape and arrangement. Cu grown on top of Co/Cu(001) decorates the Co islands. A retarding field Auger system and an Auger spectrometer with cylindrical mirror analyser are calibrated simultaneously with the help of the STM.

The growth of Fe on W(110) is described in section 3. Due to the large lattice mismatch between Fe and W only in the first 1.5 atomic layers (AL) grow in a layer by layer mode. A surface reconstruction with the lattice vectors \( \mathbf{a}_1 = (40,0) \) Å and \( \mathbf{a}_2 = (20,40) \) Å is observed with the beginning of the third layer.

Missing Co atoms are found to be the most frequent defects in Molecular Beam Epitaxy Co films grown on Cu(001), as revealed by room temperature STM. The density of these atomic-sized holes grows with increasing film thickness. Holes are found to move to neighbouring sites, their movement being most likely thermally driven. This is shown in section 4.

The magnetic properties of Fe and Co microstructures are investigated with Scanning Kerr Microscopy (SKEM) and described in section 5. We show that a 5 μm wide and 4 AL thick Fe stripe on W(110) with an aspect ratio of 600 : 1 does not have an easy axis along the stripe. This is in contrast to a Co stripe on Cu(001) which has a magnetic easy axis along the stripe. We observe that for Fe microstructures the coercive field is larger than for Fe films. This is not true for Co microstructures. Furthermore we compare the strong influence of the sweeping rate on the coercive field of a Fe island and a Fe film. The minute reflectivity difference between substrate and atomically thin magnetic structure can be measured with the SKEM.
Section 1.

UHV-System

For the measurements on magnetic microstructures we constructed a new Ultra High Vacuum (UHV) system consisting of three chambers. Samples, STM-tips and crucibles for the evaporation source are introduced through a load lock into the preparation chamber. Surface cleaning, film growth, and surface characterization with Low-Energy Electron Diffraction (LEED) and Auger spectroscopy as well as STM-tip preparation is done in this chamber. Auger spectra can be collected by using a retarding field analyser (RFA) or a cylindrical mirror analyser (CMA). For the investigation of the magnetic onset, Kerr effect measurements can be done during film growth. After preparation the samples are transferred to the Kerr chamber for spatially resolved Kerr effect measurements or to the STM chamber for the structural characterization. All three chambers are independently pumped and have a base pressure better than $5 \times 10^{-11}$ mbar. The sample transfer is built for the handling of tetragonal bodies (transporters) [1.1] with two holes for picking them up with the fork of a magnetically driven wobble stick [1.2] and two additional holes perpendicular to the others for holding the transporters on the transfer, the manipulator or the measuring positions. Transporters can carry a sample, a tip holder with a STM tip [1.3] or crucibles for the evaporation source.

1.1. Evaporation source

Metals are evaporated from a Knudsen cell type evaporation source with in situ exchangeable crucibles. The crucibles are made from tungsten and can be transferred through the UHV system and load lock like sample holders. New crucibles are annealed before charging. This reduces the time needed for outgassing the evaporator and improves the purity of the evaporated material. An additional evaporation stage (Fig.1) is designed for the growth of laterally structured thin films. This is done by placing a diaphragm between evaporation source and sample [1.4]. The diaphragm is mounted onto a transporter and can be sputtered and annealed. During evaporation the transporter with the diaphragm is placed on an inertial slider which allows an accurate positioning in front of the sample surface. Evaporation source and diaphragm are 300 mm apart. This together with the ability to place the diaphragm
extremely close to the sample surface reduces boundary effects which occur due to the non point like character of the material source. Diaphragms for the evaporation of thin stripes are made from GaAs (Fig.2 left hand side). A piece of a GaAs wafer is cleaved and the two resulting pieces are mounted in a distance of 5 µm or more onto a special transporter. Metals are evaporated through the transporter and the slit between the GaAs cleavage faces onto the sample.

Other diaphragms for the evaporation of microstructures are made from thin titanium foils having a thickness of 1 µm (Fig.2 right hand side). Microholes with a size between 1 (µm)$^2$ and 20 (µm)$^2$ are produce with the help of a blunt STM-tip.

**Fig.1:** Schematic view of the stage which is used for the evaporation of microstructures.

**Fig.2:** Schematic view of the diaphragm used for the growth of stripes (left hand side) and for microstructures (right hand side).
1.2. Magneto-Optic Kerr-Effect (MOKE)

When linearly polarised light is reflected from a magnetic surface either its intensity or its polarisation changes depending on the exact scattering geometry and on the polarisation of the incoming beam [1.5-1.7].

In the transverse geometry the Kerr effect is proportional to the magnetization $M$ perpendicular to the scattering plane (Fig.3). For $p$ polarized light ($E$ vector in the scattering plane) the reflected intensity $I_r$ is given by $I_r = I_0 + I(M)$. The signal $I(M)/I_0$ is of the order of $10^{-5}$. However the use of highly stabilised laser diodes allows an efficient investigation of atomically thin magnetic layers.

![Fig.3: Schematic view of the MOKE set-up](image)

1.3. Scanning Kerr Microscope (SKEM)

A map of the surface magnetization of a sample is obtained with the Kerr effect (see e.g. Ref. 1.3) using a focused laser beam as light source and scanning the sample. The UHV sample scanning stage is based on inertial sliders similar to those used in STM set ups [1.8]. It allows $x$ and $y$ scanning, rotating the sample, tilting around the $x$ and $y$ axis, as well as the mechanically driven height adjustment of the sample. Fig.4 shows a schematic drawing of the Kerr experiment. The sample is mounted with the surface pointing along the $z$ axis. Independent inertial sliders allow $x$ and $y$ movement as well as the rotation around the $z$ axis. The tilting around the $x$ and $y$ axis is done with a half sphere.
sitting on three Piezo tubes with four segments. These tubes are oriented in a way which allows the tilting of the sample around the x and y axis. The entire set up rests on a precision linear motion feedthrough. A vertically mounted linear ball bearing builds the connection with a DN 150 CF - flange. The light from a stabilized laser diode (λ = 675 nm) [1.9] is directed through a Glan - Thompson linear polarizer (extinction better than 10^-6) and a focusing optics onto the sample surface (see Fig.5). The focusing optics consists out of 5 lenses and is located in the tube of a reentrant UHV view port. Optics [1.10] and view port [1.11] were specially designed in order to reach a diffraction limited laser focus. The key features of this set up are:

- the small distance between last lens and sample surface
- the quality of the lenses and the quartz plate at the end of the reentrant view port (flatness: λ/10, optical parallelism better than 1.4 x 10^-3 degrees, Diffraction index homogeneity: Δn < 2 x 10^-6) which ensures that the wave front quality of the laser beam (better than λ/3) is not affected by the optical set up.

Two identical optical arrangements are used for the focusing of the primary laser beam and the collection of the reflected beam. The coarse adjustment of the focus is done by changing the position of the whole optics along the light path and by varying the distance between lens 1 and 2. While imaging the edge of a magnetic stripe or another well defined feature on the sample surface the sample height as well as the distance between lens 1 and 2 is optimized in order to get a sharp image of the observed feature. The whole optical set up is removed from the chamber while baking the system.

Fig.4: Schematic drawing of the ultra high vacuum scanning stage. The scanning of the sample is achieved by using inertial sliders. Independent sliders are used for the rotation, the x, and the y movement.
1.4. STM-system

1.4.1. Tip preparation

Chemically etched tungsten tips are usually used for the STM imaging. After insertion into the preparation chamber the tips are heated by electron bombardment (600 V, 3 mA - 6 mA) and characterized by field emission. Only tips with a field emission threshold of less than 300 V were used for STM imaging (typically 0.1 nA at 300 V).

1.4.2. STM

The STM chamber is equipped with two wobble sticks. One has a fork for picking up the transporters from the transfer between the chambers (a magnetically coupled linear transfer). The second wobble stick is equipped with a M1 socket head cap screw driver and a key for picking up STM tip holders and placing them into the end-piece of the tube scanner. An M1 screw rigidly connects tip holder and tube scanner. This screw can be tightened with the wobble stick screw driver. The wobble sticks themselves are a home-
made construction where the bellows of a classical wobble stick [1.12] is replaced by a miniature magnetically coupled linear - rotary motion feedthrough. This allows delicate manipulations without the counter force arising from the pressure difference between chamber inside and outside.

The home built STM has a single - stage coil spring suspension and Eddy current damping. The springs have a eigenfrequency of 1.3 Hz and the damping coefficient $\gamma$ is chosen to be $0.1w_0$ [1.13,1.14]. The tip is oriented along the horizontal axis ($z$ - axis). Coarse approach (along $z$) and coarse positioning along the horizontal axis ($x$ - axis) are done with an inertial slider [1.8]. A second, magnetically loaded inertial slider allows the coarse positioning of the tip along the vertical axis ($y$ - axis). The STM is driven by commercial electronics and software [1.15]. The $x$ and $y$ axis of a half inch tube scanner [1.16] were calibrated by imaging a cleaved GaAs(110) surface with atomic resolution (Fig.6). Single atomic steps on the same surface were used to calibrate the $z$ axis. Samples can be imaging at room temperature. The STM is a rigid construction, images can be collected while the turbomolecular pumps of the preparation chamber and load lock are running.

Fig.6: STM topographic image of a cleaved GaAs(110) surface. The image is used to calibrate the STM tube scanner. The sample voltage is 2 V, the tunnelling current is 0.2 nA.
Section 2.

Growth of Co on a Cu(001) surface

Magnetic properties are very sensitive to structural details. It was found that Co grown on a flat Cu(001) surface is magnetized along the [±110] directions. Grown on a stepped surface, Co exhibits a uniaxial easy axis along the steps [2.1-2.3]. This easy direction of the magnetization is rotated by 90° through the deposition of a minute amount of Cu on the top of Co [2.4]. This demonstrates the dependence of the magnetic properties on the symmetry [2.5] and the morphology [2.6]. In the following the growth of Co on a stepped and a flat Cu(001) surface and the growth of Cu on the top of Co/Cu(001) is described. An Auger system with a retarding field analyser (RFA) and one with a cylindrical mirror analyser (CMA) are calibrated simultaneously with the help of STM.

2.1. Sample preparation

The Cu(001) crystals were mechanically polished down to a grain size of 1 μm. This was followed by electropolishing using a solution with 30% of HNO₃ in CH₃OH. During a few seconds a positive voltage of about 3 V was applied to the crystal. This procedure results in a mirror-like Cu(001) surface. Electropolishing was found to be crucial in order to get a clean, flat, and well-ordered surface. After insertion into the vacuum system the samples were cleaned by Ar ion sputtering using a beam energy of 500 V and a target current of 7 μA. This was followed by annealing the sample up to 770 K during a few hours. Annealing to higher temperatures results in a S-covered Cu surface. Repeating this procedure about five times resulted in a clean surface showing sharp LEED spots. For the last cycle the sample was sputtered during 5 minutes and annealed to 720 K during 15 minutes. Fig. 1 shows typical surface areas of the flat and the stepped Cu(001) crystal. The average distance between steps amounts to 500 Å for the flat and 30 Å for the stepped crystal. The gradient enhanced image on the right hand side of Fig. 1 shows the same area as the picture in the middle. Steps appear as dark lines and the terraces appear gray.

Co and Cu are evaporated from a Knudsen cell type evaporation source. A evaporation rate of 0.2 up to 0.4 AL/min is used to produce the layers described in the following. During evaporation the pressure rises typically up to 5 x 10⁻¹⁰ mbar.
Fig. 1: 1000 Å x 1000 Å STM topograph of the flat Cu(001) surface (left). The [-110] direction is along the horizontal axis of the image. Gray scale range: 2.1 Å. Tunnel current: 0.2 nA. Sample voltage: 0.38 V. Middle: 930 Å x 930 Å STM topograph of the stepped Cu(001) surface. The [-110] direction is along the horizontal axis of the image. Gray scale range: 5.6 Å. Tunnel current: 0.2 nA. Sample voltage: 0.96 V. Gradient enhanced image showing the same area as the picture in the middle (right side).

2.2. Co grown on the stepped and on the flat Cu(001) substrate

 Depositing the same amount of Co onto the flat and the stepped Cu crystal results in layers having a completely different surface morphology. The well separated islands of a 0.5 AL Co film on the flat crystal (Fig. 2a) have a preferential edge orientation along the [±110] direction (see also Fig. 5). The diameter of the islands varies from 10 Å up to 250 Å. On the stepped surface the islands appear to be round (Fig. 2b). They have a diameter ranging from 20 Å up to 50 Å. For this surface it is impossible to recognize the layer to which a single Co island belongs. The arrangement seems to be arbitrary and only the direction of the underlying Cu steps can still be recognized. We find that the topographs shown in Fig. 2 are typical for this coverage. Identical images have been collected at different spots on the surface and on other layers having the same thickness. The Auger spectra for the two samples are identical.
Fig. 2: $1000\text{ Å} \times 1000\text{ Å}$ STM topograph of a 0.5 AL Co film deposited onto a flat Cu(001) surface (a) and onto the stepped Cu(001) surface (b). Note the two steps crossing the image on the left side. The [-110] direction is along the horizontal axis of the images. From left to right: Gray scale range: 4.2 Å and 6.5 Å. Tunnel current: 0.1 nA and 0.2 nA. Sample voltage: 0.4 V.

Fig. 3: STM topograph of 2.0 AL of Co deposited onto a flat (a) and onto a stepped (b) Cu crystal. The [-110] direction is along the horizontal axis of the image. The image size is 1000 Å and 500 Å, respectively. Gray scale range: 5.4 Å and 6.9 Å. Tunnel current: 0.2 nA. Sample voltage: 0.6 V.
Increasing the coverage from 0.5 AL to 2 AL (Fig. 3a) results in a perfect Co film with almost no Co in the third layer. Evaporating the same amount of Co onto the stepped substrate gives the topograph shown in Fig. 3b. If the growth were layer by layer, the Co should reproduce the topographic properties of the substrate exactly. This is not the case. The topograph in Fig. 3b shows Co patches which are extended along the steps of the substrate. Identical Auger spectra are obtained for the Co layers shown in Fig. 3a and 3b. The non regular appearance of the layer grown on the stepped crystal is underlined in Fig. 4. It arises from the fact that straight Cu steps transform into meandering steps of the Co layer grown on top. Fig. 4 shows some isolated steps of the 2 AL Co film grown on the flat substrate. The steps are not straight but are meandering with an amplitude of about 70 Å. This fluctuating deviation between the Co step position and the position of the underlying Cu step results in a modulation of the film thickness in this region. Therefore the layer by layer growth is obscured by the presence of steps. Note that the distance between the steps of the second crystal is smaller than the modulation amplitude of the Co step position. Consequently the growth on this crystal is completely step dominated. The distance of the steps visible in Fig. 4 is becoming small in the upper left corner of the image. This region simulates the stepped crystal. The surface pattern in this region is very similar to the one shown in Fig. 3b.

A mean square Co step roughness of 62 Å was calculated from STM topographs of Co grown on Cu(1 1 17) [2, 7]. This findings are in agreement with the results described above.

Nothing new is found if the coverage on the flat crystal is increased. The same island pattern is very well reproduced for each new atomic layer. This means that a 2.5 AL film looks identical to a 3.5 AL film. An observation which is in agreement with Ref. [2, 8]. However for the stepped Cu surface the island shape and size changes with coverage. It is not possible to separate the Co belonging to different atomic layers. It is impossible to detect the completion of a layer by imaging the Co on the stepped surface. Just by depositing the same amount of Co onto the flat crystal gives the right calibration of the thickness.
Fig. 4: STM topograph of 2.0 AL Co deposited onto the flat Cu(001) crystal. The [-110] direction is along the horizontal axis of the image. The image size is 1000 Å. Gray scale range: 7.4 Å. Tunnel current: 0.2 nA. Sample voltage: 0.6 V.

2.3. The first layers

Much debate arose about the growth of the first two layers of Co on Cu(001). A perfect layer by layer growth was reported based on LEED and Auger measurements [2.9]. From angle resolved x-ray photoemission scattering experiments it was claimed that cobalt initially forms two layer thick islands [2.10]. A significant concentration of Co islands of bilayer height for a coverage above 0.5 AL was found using x-ray diffraction [2.11]. An STM experiment showed that 0.1 AL of Co are already in the second layer after having deposited 0.8 AL [2.8]. STM is the most direct method for studying initial growth modes. The covered area and the thickness of the islands is directly measured from a topographic image. Scattering methods require an absolute calibration of the thickness in order to correlate it with the onset of scattering from the second layer. A calibration done with an Auger or with a quartz microbalance can be off by a factor of two. Furthermore the presence of steps can result in a modulation of the film thickness (see previous section). All this could result in a wrong interpretation of the scattering experiment.
Fig. 5 shows STM topographs taken in the critical range from 0.6 AL up to 0.9 AL. This is the range where a significant bilayer growth mode should be observed. The height distribution of the STM image pixels is used to determine the amount of Co in the first and the second layer. The result of this analysis is shown in Fig. 5. Identical measurements in the thickness range from 0 - 2 AL are summarized in Fig. 6. The amount of Co found in the second layer is plotted as function of the amount of Co deposited. The solid line indicates the behavior which would be expected for an ideal layer by layer growth mode. Fig. 6 shows that the degree of bilayer growth observed is much smaller than the one reported in the literature [2, 10, 11]. Less than 10% of the Co atoms are found in the second layer for a film with a nominal thickness of 0.9 AL. This deviation from the ideal layer by layer growth mode is smaller but close to the one reported in Ref. 2.8.

![Fig. 5: STM topographs of 0.55 AL (left), 0.63 AL (middle), and 0.91 AL (right) of Co grown on the flat Cu(001) crystal. Black corresponds to the Cu substrate, gray to the first layer, and white to the second layer of Co. The image size is 500 Å, 400 Å, and 500 Å, respectively. The [-110] direction is along the horizontal axis of the image.](image-url)
Fig. 6: The amount of Co found in the second layer is plotted as a function of coverage. Each point was obtained by analysing the pixels height distribution of the corresponding STM image. The solid line corresponds to the ideal layer by layer growth mode.

2.4. Cu on the top of Co/Cu(001)

Only a minute amount of Cu on top of Co on Cu(001) rotates the easy axis of the magnetization [2.4]. Adding more copper eventually switches the magnetization back to its original direction. This behaviour was explained assuming the Cu to decorate the Co islands. In the following we will test this assumption. The result of this test is displayed in Fig. 7. Onto the Co layers shown in Fig. 7a and 7d we evaporate 0.2 and 0.6 AL of Cu respectively. The resulting surface topography is shown in Fig. 7b and 7e. These two pictures have to be compared with the topographs shown in Fig. 7c and 7f which show pure Co layers. (b) and (c) as well as (e) and (f) are indistinguishable. This finding corroborates the statement that Cu decorates the Co steps [2.12]. Depositing more Cu onto a Co layer results in a surface with irregularly shaped islands having round edges (Fig. 8a). Depositing Cu directly onto the Cu(001) surface gives rectangular islands (Fig. 8b). Therefore the underlying Co layer modifies the Cu growth over several atomic layers.
Fig. 7: Comparison between Co on Cu(001) and Cu on Co on Cu(001). The [110] direction is along the image diagonal.

Fig. 8: STM topographs of 2.5 AL Cu grown on a 1.2 AL thick Co layer (a) and on a flat Cu(001) crystal (b). The image size is 1000 Å x 1000 Å. The [-110] direction is along the image diagonal (a) and along the horizontal axis for (b).
2.5. Calibration of the RFA and the CMA Auger

A film thickness calibration is often done with the help of Auger electron spectroscopy. Calculating a film thickness from an Auger spectra requires the knowledge of the mean free path of the electrons. Furthermore it has to be taken into account that a RFA Auger collects electrons which are emitted with an angle ranging from zero up 50 degrees measured from the surface normal of the samples whereas a CMA just accepts electrons emitted with 42 degrees. All these uncertainties together can result in a calibration being off by a factor of two.

The ratio of the Co Auger electron peak at 656 eV and the Cu Auger electron peak at 920 eV is plotted in Fig. 9 as function of the Co evaporation dose. The filled circles are measured with the CMA energy analyser [2.13] the open circles with the RFA analyser [2.14]. By counting the coverage with the help of an STM topograph we can transform the evaporation dose scale into an absolute atomic layer scale. This was done for a 0.5 AL and a 2 AL film. The two STM calibration points give the absolute thickness scale and confirm the linearity of the thickness versus evaporation dose. Setting for simplicity the mean free path of the Cu 920 eV Auger electrons equal to the mean free path of the Co 656 eV electrons leads to the following formula for the Auger ratio:

\[
\frac{I_{\text{Co}}}{I_{\text{Cu}}} = \frac{I_{\text{Co}}^0}{I_{\text{Cu}}^0} \cdot \exp\left(\frac{d}{\lambda'} - 1\right) \tag{2.1}
\]

Here \(d\) is the layer thickness, \(I_{\text{Co}}^0\) is the Co Auger signal from a very thick Co layer, and \(I_{\text{Cu}}^0\) is the Cu signal from a clean Cu surface. \(\lambda'\) is the only adjustable parameter. The solid lines in Fig. 9 are calculated using \(\lambda' = (4.3 \pm 2.1)\) AL and \((4.1 \pm 0.5)\) AL for the RFA and for the CMA Auger respectively. \(\frac{I_{\text{Co}}^0}{I_{\text{Cu}}^0}\) is 0.47 for the RFA and 0.40 for the CMA. The mean free path \(\lambda\) deduced from the CMA measurements equals \(\lambda' / \cos 42^\circ\). Therefore we get 5.5 AL (9.9 Å) for \(\lambda\). This value is smaller than the 6.5 AL and 7.8 AL obtained for the 656 eV and 920 eV electrons respectively using the commonly accepted “universal curve” [2.15]. It is difficult to deduce a mean free path from the RFA measurements since not all the emission angles have the same weight [2.16].
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Fig. 9: Intensity of the Co 656-eV Auger line divided by the intensity of the Cu 920-eV line is plotted as function of the evaporation dose. Filled circles are measured with a cylindrical mirror energy analyser, the open circles with a retarding field electron energy analyser. The lines are a fit to the measured points (see text). The evaporation dose was calibrated with the help of the STM at a coverage of 0.5 and 2.0 AL (indicated by the dotted lines).

2.6. Elevated substrate temperatures

The influence of the substrate temperature on film growth is investigated by depositing the same amount of Co onto the Cu held at 370 K, 430 K, and 540 K. The topographs of the corresponding layers are shown in Fig. 10a - c. The coverage equals to 0.7 AL. Small depressions evolve for a growth temperature of 370 K. They are 0.2 Å deep and are irregularly arranged. Shape and arrangement of the Co islands remain unaffected. Those depressions become more pronounced for a substrate temperature of 430 K. Note that Co and Cu can be distinguished in Fig. 10b since the depressions are found on the Co islands and not on the Cu substrate visible in between the islands. The depressions could be caused by missing Co atoms, by interdiffusion or by imperfections of the underlying substrate. We always observe empty Cu sites on a flat Cu surface. Under normal growth conditions these vacancies must be filled with Co atoms in order to allow the growth of flat Co islands. Those Co atoms repairing the substrate are embedded into a Cu layer and are therefore very attractive for interdiffusion. This could be the reason for the depressions visible in Fig. 10a and 10b. However the density of missing Cu atoms is not as high as the density of depressions. This can be seen in Fig. 10b since the uncovered regions appear to be flat.

Growing Co at a substrate temperature of 540 K gives a completely new surface morphology (Fig. 10c). We find a flat and almost island free surface. Co was proposed to diffuse into the Cu at a temperature of 670 K [2.10]. Heating a 3 AL Co film up to 490 K
resulted in a layer with a few big holes [2.17] but with an arrangement of Co islands being similar to the room temperature grown layer. The question which arises therefore is: Do we see a perfect Co layer or Cu covered Co in Fig.10c?
An identical amount of Co was deposited for the layers shown in Fig.10. Consequently the Auger peak ratio for Co and Cu should be constant for all three layers. The Auger peak ratio is defined as the intensity of the Co 656 eV Auger line divided by the intensity of the Cu 920 eV Auger line. For image 10a, b, and c we find a ratio of 0.086, 0.060, and 0.056. Extending formula (2.1) to the bilayer system and calculating the Auger ratio for a 0.7 AL film and a 0.7 AL film covered with Cu gives 0.082 and 0.066 respectively. Therefore the Auger spectrum suggests the top layer of Fig.10c to be Cu.
Annealing a room temperature grown Co layer during 450 seconds to 495 K gives the topograph shown in Fig.10d. After annealing the Co exhibits depressions similar to the layer grown at 430 K.

Fig.10: STM topographs of 0.7 AL of Co grown at 370 K (a), 430 K (b), and 540 K (c). The layer shown in (d) was grown at room temperature and subsequently annealed to 495 K during 450 s. The [-110] direction is along the horizontal axis of the image. The image size is 500 Å. The gray scale range is 5.6, 4.7, 3.6, and 3.2 Å.
2.7. Magnetic onset of Co on Cu(001) at room temperature

The onset of long range magnetic order is often used as a standard for the thickness calibration of a layer. In this section we investigate a Co layer at the onset of long range magnetic order with the help of the STM. During evaporation the magnetization $M$ is measured with magneto-optical Kerr effect (MOKE) [2,18]. The external magnetic field is applied along the [110] direction. As soon as the first hysteresis loops appear the Co evaporation is interrupted.

Fig.1a shows remanence (triangles) and saturation (circles) of the Co layer during evaporation. The hysteresis loop displayed in Fig.1b is taken at the onset after the evaporation was stopped. Fig.2 shows a STM image of a Co layer at the onset of long range magnetic order. The total Co coverage amounts to 1.70 AL.

![Fig.1: Magnetization $M$ versus evaporation time. Remanence (triangles) and saturation (circles) are shown in (a). Hysteresis loop at the onset of long range order (b).](image-url)
Fig. 2: STM topograph of Co on Cu(001) at the onset of long range magnetic order at room temperature. The Co coverage amounts to 1.70 ÅL. The image size is 700 Å x 700 Å and the [110] direction is along the horizontal axis of the image.
Section 3.

The Growth of Fe on W(110)

Due to the large lattice mismatch of bcc Fe on W(110) (\(a_{\text{Fe}} = 2.866 \text{ Å}; a_{\text{W}} = 3.165 \text{ Å}\)) only the first two layers grow pseudomorphic \([3.1,3.2]\). The third layer shows a reconstruction which is investigated with LEED and STM. The quality of in situ grown microstructures, e.g. the sharpness of the boundaries, is tested with STM. With the help of our evaporation technique it is possible to produce a Fe wedge on a micrometer length scale.

3.1. W(110) Preparation

The W(110) single crystal is mechanical polished until it shows a perfect mirror-like surface. The orientation of the crystal was checked by x-ray diffraction with an accuracy better than 0.3 degrees. After load locking the sample into the UHV chamber the crystal is annealed in a O\(_2\) atmosphere of \(p = 5 \times 10^{-8} \text{ mbar}\) at 1900 K. This leads to the desorption of C through the formation of CO. The remaining WO\(_x\) is removed from the surface by flashing the crystal to 2500 K. The cleanliness of the crystal is checked by LEED and AES. Fig.1 shows a STM images of the clean W(110) surface. The size of the images is 2000 Å x 2000 Å. The average step to step distance is 400 Å.

![Fig.1: STM-image of the clean W(110) crystal surface (scan size 2000 Å by 2000 Å). The picture was taken with a tunnelling current of 0.3 nA and a sample voltage of 230 mV.](image)
3.2. Fe evaporation

Fe is evaporated from a Knudsen cell type evaporation source. The evaporation rate is between 0.02 and 0.2 AL/min. This is checked with AES and STM. During evaporation the base pressure is typically $2 \times 10^{-10}$ mbar and the temperature of the sample is 300 K.

3.3. Growth study

Data from several STM pictures are collected in Fig. 2. A histogram of the pixel heights distribution is derived (Fig. 2a) from every STM image taken on a flat part of the sample. Every peak in this histogram corresponds to a Fe layer. The first peak in Fig. 2a belongs to the first Fe layer, the second peak to the second layer. Due to the limited sharpness of the tip the number of image pixels between the two peaks is not zero. The pixels between two layers are added to the lower lying layer since they belong to the region the tip needs to adjust itself to the new surface heights. For example in Fig. 2a the first Fe layer is almost complete (98.9% of a AL) and the second layer covers 17.5% of the surface. This gives a total Fe coverage of 1.16 AL. Fig. 2b demonstrates a perfect layer by layer growth for the initial 1.5 Fe layers. A bilayer growth mode is observed with the beginning of the third layer.

![Diagram](image)

**Fig. 2:** (a): Histogram of an STM picture. The total Fe coverage is 1.16 AL with 0.99 AL in the first layer and 0.18 AL in the second layer. (b): The amount of Fe found in the corresponding layers plotted as function of the coverage. Each point was obtained by analyzing the pixel height distribution of an STM image. The solid line indicates perfect layer by layer growth.
Fig. 3 shows a 0.74 μm long Fe wedge starting from blank W(110) and ending at an Fe coverage of 2.5 AL. This wedge is the right boundary of a Fe stripe which was evaporated through a 91 μm wide GaAs slit (mentioned above). We use this wedge to visualize the growth mode of Fe on W(110) within one STM image. The first Fe layer is almost completed when the second layer starts to grow, i.e., an almost perfect layer by layer growth. The third layer starts (see Fig. 2b) before the second layer is completed. The Fe islands on top of the first layer are extended along the [001]-axis whereas the first layer islands have a square shape.

The transition from the first to the second layer is shown more detailed in Fig. 4. The boundaries of the first layer islands are oriented along the [-111] and the [1-11] directions and are equal in length. In contrast, the islands in the second layer are extended along [±110]. This change of growth mode occurring already in the second layer might be explained by the large lattice mismatch between Fe and W starting to stress the film.
Fig.4: 2000 Å x 4000 Å STM image showing the first and the second layer. The island in the first Fe layer prefer the [-111] and [1-11] directions and have a square shape. The second layer islands are extended along [001] and have an oval shape.

The growth of Fe between 1.7 AL and 2.8 AL is shown in Fig.5. The islands in the second layer cover large areas. Their length to width ratio is always larger than 1. Also the third layer exhibits islands which are extended along [001]. However the island size is reduced compared to the second layer. In addition, the islands in the third and fourth layer exhibit a chess-board like reconstruction.
Fig. 5: Fe growth study between the second and the forth layer. From the third layer a superlattice is observed. Image size: 2000 Å x 7600 Å.
This fact is illustrated in Fig. 6 where we zoom onto an island in the fourth layer. This island shows a chess-board shaped superlattice with the following lattice vectors

\[ \mathbf{a}_1 = (40,0) \ \text{Å} \quad \text{and} \quad \mathbf{a}_2 = (20,40) \ \text{Å}. \]

The difference in heights within this superlattice is about 1 Å. We transfer the vectors \( \mathbf{a}_1 \) and \( \mathbf{a}_2 \) into reciprocal lattice vectors. Using the following formula

\[ b_1 = \frac{2\pi \cdot a_1 \cdot (a_2)^2 - (a_2 \cdot (a_1 \cdot a_2))}{(a_1)^2 \cdot (a_2)^2 - (a_1 \cdot a_2)^2} \]

This gives

\[ b_1 = (0, \pi/20) \ 1/\text{Å} \quad \text{and} \quad b_2 = (\pi/20, \pi/40) \ 1/\text{Å} \]

\( b_2 \) is derived from formula (1) by interchanging the indices. The new primitive reciprocal vectors are given by

\[ b_1^* = b_2 \quad \text{and} \quad b_2^* = b_1 + b_2 \]

This calculation leads to the LEED picture shown in Fig. 7 (right hand side). Comparing the calculated picture with the LEED picture taken from a 3 AL Fe film (left hand side) shows a good agreement.
Fig. 6: Stepwise magnification of a Fe island in the fourth layer. The size of the first image is 1000 Å x 1000 Å, second 500 Å x 500 Å, third 250 Å x 250 Å, fourth 150 Å x 150 Å. The superlattice begins from the third Fe layer and has the lattice vectors $a_1 = (40,0) \text{ Å}$ and $a_2 = (20,40) \text{ Å}$.

Fig. 7: LEED picture of 3 AL Fe on W(110) (left hand side) in comparison to the calculated picture (right hand side).
3.4. Magnetic onset of Fe on W(110)

Growing Fe on W(110) and stopping the growth at the onset of long range magnetic order gives a sample with a Curie temperature close to room temperature (which is the substrate temperature during growth). Such layers were found to follow the phase transition of the two-dimensional Ising model which belongs to the universality class of a two-dimensional system with a one dimensional order parameter [3.3]. In this section we will define thickness and structure of the Fe layer at the onset of long range magnetic order.

Fig. 8 shows remanence (squares) and saturation (circles) during film growth. As soon as the first hysteresis loop appears (Fig. 8b) the evaporation is stopped and the sample is investigated with the STM. Fig. 9 shows a typical STM image of the sample belonging to Fig. 1. It consist out of one complete Fe layer and a two-dimensional network of irregular patches. Some iron is already visible in the third layer. The total Fe coverage is 1.82 AL. LEED measurement show a sharp p(1x1) pattern indicating a growth in registry with the substrate.

![Graphs showing remanence and saturation vs. evaporation time, and hysteresis loop at the onset of long range magnetic order](image)

**Fig. 8:** (a): Remanence (squares) and saturation (circles) versus evaporation time. (b): Hysteresis loop at the onset of long range magnetic order at room temperature.
Fig. 9: STM topograph of a Fe layer at the onset of long range magnetic order. The coverage is 1.82 ÅL. The image size is 2000 Å x 2000 Å. The [001] direction is along the horizontal axis of the image.
Section 4.

Mobile atomic scale depressions in ultra-thin Co- and Cu-films

4.1. Introduction

The most elementary defects in solids are monoatomic vacancies. Their existence and mobility has been established for semiconductor surfaces using scanning probe microscopies[4.1,4.2]. At metal surfaces the smallest defects observed so far are sputtering induced monolayer deep vacancy islands [4.3,4.4], their diameter being typically larger than 3 nm.

Here we report on the first observation of mobile atomic-sized depressions in atomically thin Co- and Cu-films grown on Cu(001). As much as 10% of the top atomic layer of a Co film can consist of monoatomic depressions, their density growing with thickness. Covering these films with Cu results in a surface having the same kind of defects. The observed monoatomic defects are mobile like ad-atoms on clean metal surfaces [4.5], although our first attempts to control their movement with the STM tip failed. We argue these depressions to be missing Co or Cu atoms, respectively. This observation demonstrates that such ultra-thin epitaxial metal films are far from being defect-free on an atomic scale. As defects on all length scales are known to govern many physical properties, our discovery identifies a new potential player in determining e.g. the magnetic and transport properties of metallic multilayers [4.6-4.12] and their evolution with time.

4.2. Sample preparation

We grow Co films by evaporation from a Knudsen-cell type of source. The evaporation rate is typically 0.1 AL per minute. The ambient pressure during evaporation is less than 5x10^{-10} mbar and the substrate temperature is typically 45 °C. STM imaging is done in a vacuum better than 2x10^{-11} mbar and at room temperature.
4.3. Mobile atomic scale depressions

Fig.1a (500 Å x 500 Å) illustrates the large scale morphology of Co films on Cu(001). Growing Co on Cu(001) results in Co layers with regularly shaped and flat islands [4.13,4.14] which repeat at the completion of each AL. The monoatomic steps of the Cu-surface are replicated by the Co-film [4.13] (see section 2.). In this STM topographic image of a 3.2 AL thick Co film light-gray (black) corresponds to the Co in the forth (third) layer. The overall large scale properties of the Co-films, as well as the preferential boundary orientation of the Co-islands along [±110] direction are in agreement with previous findings [4.13,4.14]. In contrast to previous findings, however, new features appear on a smaller scale, consisting of a large amount of tiny defects: they are already visible as fine structure in Fig.1a and become clearly resolved in the 150 Å x 150 Å image of Fig.1b. Their density is ≈ 1.5 per nm². Their shape is round.

![Fig.1](image)

We observe that both diameter and depth of the defects are very sensitive to the sharpness of the tip. This fact is illustrated in Fig.2a-2c, which shows cross sections through such defects taken with three different tips. A measure of the tip-sharpness is derived from a line scan over a monoatomic step. The derivative of such a line scan gives a bell-shaped curve, its full width at half maximum (FWHM) being representative of the tip-
sharpness. This procedure yields 5.1 Å, 5.5 Å, and 8.0 Å for the tips used to obtain the defect line scans (a), (b), and (c), respectively. In agreement with the deteriorating sharpness, the defects become more shallow in going from (a) to (c). We conclude that the status of the tip is one crucial parameter for resolving atomic-scale defects, which might remain unresolved if the apparent resolution is not truly atomic.

The observation of these atomic-scale features leads to the question whether their origin is electronic or structural. Whatever the tunnelling parameters are the observed features always appear as a depression. In the line scans of Fig.2d-2g different tunnelling parameters are used to image the defects, which corresponds to changing the tip-sample separation. All line scans show the defect as being a depression with a resolution depending on the tip-sample separation.

**Fig.2:** Single line scans through defects found in Co layers. Tip radius in Å, sample voltage in V and tunnel current in nA are as follows. a: (5.1, 0.28, 0.2); b: (5.5, 1.0, 0.5); c: (8.0, 0.8, 0.7); d: (5.1, 1.0, 0.2); e: (5.1, 3.5, 0.2); f: (5.1, 1.0, 0.4); g: (5.1, -1.2, 0.4). Scans (d) and (e) as well as (f) and (g) have been measured in double scan mode. The dashed lines correspond to the surface level of the layer. The profiles of the defects have been shifted for clarity.
The observed defects are mobile, see Fig. 3. This figure shows two images taken at the same spot on the Co layer. The defect labeled with “A” has the same position in both images. Defect “B” moved $(6.2 \pm 1.1)$ Å, the uncertainty of $1.1$ Å arising from the positioning of subsequent image frames, from the calibration of the STM and from the uncertainty in finding the centre of the defect. Direction and distance of “B”’s-displacement are close to the nearest neighbor distance of Cu along the [3-10] direction which amounts to 5.6 Å. The split feature labeled with “C” (Fig. 3b) corresponds to a defect jumping during the scanning of image 3b. The movement of the defects observed in 34 images collected at the same surface spot is summarized in Fig.4. Along the [110] direction the most frequently observed displacement amounts to $(3.2 \pm 0.5)$ Å. The most frequent displacement along the [100] direction amounts to $(3.7 \pm 0.5)$ Å. The corresponding Cu-nearest neighbor lattice distances are 2.6 Å and 3.6 Å, respectively. The large difference between defect displacement and Cu lattice constant along [110] might be due to a more efficient stress relaxation in the Co layer along this direction. The shorter movements along the close packed [110] direction are about five times more frequently observed than movements along [100]. It can not be excluded that a movement along [100] consists of a step along [110] plus one step along [1-10]. This could account for the different probabilities observed. We find a rate of 0.05 jumps per defect and per minute at room temperature.

**Fig.3:** 50 Å x 50 Å STM topographs taken at the same surface spot of a 3 AL Co film grown on Cu(001). The defect labelled with “A” has the same position in image (a) as in (b). Defect “B” moved 6.2 Å between the collection of image (a) and (b). The split feature labelled with “C” corresponds to a defect jumping during the scanning of image (b). The grey scale range is 0.6 Å, the sample voltage 0.06 V, and the tunnelling current 0.1 nA for (a) and for (b). The images represent raw data. The defects are labelled on their right hand side.
The origin of these mobile atomic-scale depressions is not yet established beyond doubts. One possible source of this atomic-sized contrast could be the interchange of Co with Cu atoms interdiffusing through the Co-film from the substrate. In this case the different density of states of Co and Cu could appear in a tunneling experiment as an electronic contrast [4.15,4.6], thus simulating under certain conditions, a depression. Following experimental facts speak against the observed depressions being due to inter-diffused Cu-atoms.

- The defects are mobile. In a densely packed island, moving Co or Cu atoms would require one atom to move out of the island (into the next layer) in order to provide the necessary space for its neighbor to change place. This process is energetically highly unfavorable and has never been observed with the STM [4.6].

- The density of the defects in the topmost layer increases as a function of film thickness, see Fig.5, i.e. as function of the distance of the topmost layer from the substrate.

- Submonolayer amounts of Cu deposited on top of a Co film are found to increase the lateral size of the top two-dimensional islands, indicating that the Cu atoms wet the edge of the Co-islands. Yet, neighboring Cu and Co atoms situated at the edge of a Co-island show no contrast at all.
Fig. 5: Defect density for different Co-topmost surface layers. The bars result from the statistical variations of the density within one layer.

Covering a Co film with Cu results in a surface having the same kind of mobile depressions as described above. The surface of a sample consisting out of 17 AL of Cu grown on 7.8 AL of Co grown on top of Cu(001) is shown in Fig. 6a. We find a density of \((0.9 \pm 0.2)\) defects per \(\text{nm}^2\). The Auger electron spectra of this system (Fig. 6b) shows no traces of Co. We conclude that the occurrence of depressions seems to be a common feature in Cu/Co/Cu(001) metal epitaxy as well and cannot be accounted for by interdiffusion of Cu into Co or of Co into Cu.

Contamination by Carbon or Hydrogen could be a further source for the observed contrast. Carbon, the most prominent candidate, is not present in the Auger electron spectra shown in Fig. 6b [4.16]. A density of 0.9 defects per \(\text{nm}^2\) corresponds to a Copper to defect ratio of 100 to 6.2. The CMA electron energy analyzer is 40% more sensitive to C than to Cu (920 eV) [4.17]. Therefore we would expect a Carbon Auger signal having a heights of 8.6% of the Copper Auger signal. This is not observed in Fig. 6b. From this Auger electron spectrum we can estimate an upper limit for the C signal of 1% of the Copper signal and therefore exclude Carbon as being responsible for the contrast. Hydrogen is the only contaminant not visible in an Auger electron spectra. However no adsorption of \(\text{H}_2\) occurs on the low index Copper surfaces under usual UHV conditions [4.18], the adsorption of molecular hydrogen has to be thermally activated.
The only features left to account for the depressions are single atomic vacancies (holes). We are aware that this identification contains some difficulties. First, the observation of Fig.5 is against the intuitive thought of vacancies being filled after subsequent evaporation. Whether empty Co sites are left within the film remains an open question which can be definitely answered only by e.g. cross sectional STM imaging [4.20,4.21]. However, we point out that the growing of thin films requires allocating a large number of atoms. Whether all of them can find a lattice place in the time scale of the growth is a subtle, yet unresolved question. Second, the observed mobile depressions do not seem to coalesce so to form vacancy islands or to annihilate at step edges, as one could expect if the defects are holes. However, understanding the formation of large islands and the annihilation at steps requires a model taking pair bonding, strain energies, as well as the vibrational entropy into account [4.22]. This represents a future challenge to theoreticians. We notice that the large pin holes 50 Å x 50 Å observed in Ref. [4.23] and [4.24] might occur due to the clustering of holes at elevated temperatures. Third, the geometrical size of a vacancy in a Cu-lattice is (touching spheres) 2.6 Å, its depth 1.8 Å. Instead, we observe broader and shallower features. However, tip-sharpness and screening certainly play an important (albeit quantitatively unknown) role in determining the appearance of holes.
These experiments demonstrate that the top layer of Co and Cu/Co ultrathin films contains atomic-sized defects and thus are not flat on an atomic scale, as suggested by previous findings [4.14,4.23,4.13,4.24]. We have argued by exclusion that these defects are missing atoms. Their density can be as high as 1.6 per nm², which is 10% of an AL. Whether these holes are detectable in STM images strongly depends on the sharpness of the tip and the tunnelling parameters. The depressions are found to be mobile, their movement being apparently thermally driven. In fact, our preliminary attempts to move these holes by “kicking” them with the tip failed. Rather, we find that the movement of the defects is independent of tunnelling parameters and tip position. Even positioning the tip onto the defects or onto the defect boundary and using a voltage flash, a current flash or both together had no reproducible effect on the defect position. The observed contrast has been found using different Cu(001) single crystals as a substrate for the epitaxial growth. The used crystals are flat prior to the deposition of Co and Cu [4.13] and do not show anything like the depressions found on the adlayers. The parameters used for the growth are the commonly used ones [4.7,4.19], the magnetic properties of the layers are in agreement with published results [4.7,4.8,4.19]. In fact, many of these observations have been done while obtaining some of the published magnetic properties. Therefore these images represent standard Co/Cu multilayers used for standard magnetic measurements. The presence of large amounts of atomic defects might not be restricted to Co and Cu/Co ultrathin metallic films. As metallic multilayers like those described in this report are an important class of materials in science and technology (see e.g. Ref. [4.6-4.12,4.19]), the discovery of mobile atomic-sized defects might help to a deeper understanding of these systems.
Section 5.

Magnetic Investigations of Microstructures

5.1. Introduction

Magnetic order is a collective phenomenon. As such, the dimensionality of the system plays a crucial role. In fact, epitaxial atomically thin films of magnetic atoms grown on top of a non-magnetic substrate, have magnetic properties which are very much different from their three dimensional counterparts [5.1-5.3]. Because of the collective nature of magnetism, we suspect that limiting the lateral size of the samples is bound to modify as well the very magnetic functioning of the system. It is crucial, to our opinion, to reduce the size of samples below the typical size of the magnetic domains, which is in the 5 μm range for epitaxial films [5.4].

The investigation of structures in microscale offers several challenges. First, there is the difficulty of how to fabricate the microstructures with the possibility to control their size and thickness. Second, there is the problem of how to measure the magnetic properties of such small islands. Their magnetic signal is extremely small. Ideally one would like to study one individual island in order to avoid effects resulting from variations in the island size and from interactions between the islands.

In order to meet these challenges we have developed a techniques which allows us to evaporate stripes with a width as small as 5 μm or islands with a diameter of 1 μm being as thin as one atomic layer. For the magnetic measurements we built a UHV Scanning Kerr Microscope (SKEM) capable of probing the magnetism of atomically thin epitaxial films with a lateral size limited to the μm-range.

Dipolar interactions within a magnet can give rise to a shape anisotropy. The anisotropic part of the dipolar energy is given by

\[ E_{Dip} = \frac{(g\mu_B)^2}{2a^3} \sum_{\substack{i,j \neq j}} \left( \frac{3 \cdot (\mathbf{s}_i \cdot \mathbf{r}_{ij}) \cdot (\mathbf{s}_j \cdot \mathbf{r}_{ij})}{|\mathbf{r}_{ij}|^5} \right) \]  \hspace{1cm} (5.1)

where \( a \) is the lattice constant, \( \mathbf{s}_j \) is the spin-magnetic moment at the lattice site \( r_j \), and \( r_{ij} \) is \( (r_i - r_j) \). For a small particle or for a low dimensional system \( E_{Dip} \) can be calculated...
with equation (5.1). For a macroscopic ellipsoidal sample equation (5.1) can be evaluated with the help of the Lorentz construction (see Ref. [5.5] for an overview). This leads to the following expression for the anisotropy energy

$$E = -\frac{1}{2} \cdot (N_a \cdot M_a^2 + N_b \cdot M_b^2 + N_c \cdot M_c^2)$$

(5.2)

where $N_{a,b,c}$ are the demagnetizing coefficients along the principal axis of the ellipsoid and $M_{a,b,c}$ represent the magnetic moment along the corresponding axis. $N_a$, $N_b$, and $N_c$ can be calculated for any given ellipsoid [5.6].

Evaluating (5.2) in the limit of a 5 μm wide 3000 μm long and 0.0002 μm thick stripe results in a strong shape anisotropy favouring the long axis of the stripe. This confirms the intuitive picture of a long needle being preferentially magnetized along its axis. However calculating (5.1) for the very same stripe gives almost no preferential orientation for the in-plane magnetization. In the following we will give an experimental test for the existence of an in-plane shape anisotropy in ultrathin films.

5.2. Fe-Microstructures on W(110)

5.2.1. Sample preparation

Fe microstructures are grown by evaporating Fe through a mask or a diaphragm in front of the W(110) surface. The evaporation rate was typically 0.01 AL/min. The structures were grown at room temperature.

5.2.2. SKEM and its spatial resolution

Fig.1a shows a map of the surface magnetization. An island with a diameter of 1.6 μm consisting out of 5 atomic Fe layers is clearly visible as bright area in the centre of the image. The size of the image is 10 μm x 10 μm with a pixel resolution of 40 x 40 pixels. For each image pixel the reflectivity is measured in an applied magnetic field of +600 Oe and -600 Oe. According to [5.7], the difference of these two measurements is proportional to the surface magnetization within the laser focus. This difference is plotted in Fig.1a. The sum is proportional to the reflectivity and is plotted in Fig.1b. A reflectivity image is a measure of the structural homogeneity of the sample surface. As such it does not show any trace of the Fe island.
The spatial resolution of the apparatus is given by the size of the laser focus and can be estimated from the sharpness of the boundary between magnetic and non-magnetic areas. Fig. 2 shows a Kerr effect line scan through the island shown Fig. 1a (open circles) and an exponential curve fitted to the measurement (broken line). The derivative of such a line scan (solid line) gives a peak for each transition from a magnetic to a non-magnetic region. The full width at half maximum (FWHM) of these peaks is a measure of the effective spatial resolution (boundary sharpness + laser focus). For the line scan in Fig. 2 we find a FWHM of 1.17 μm, which is an upper limit for the laser focus. Notice that Secondary Electron Microscopy with Polarization Analysis (SEMPA) [5.8-5.10] has a better spatial resolution (typically 40 nm). However, this technique does not generally allow measurements in an applied external field.

Fig. 1: 10 x 10 (μm)² Kerr effect (a) and reflectivity (b) image of a W(110) surface with a 5 atomic layer thick Fe island having a diameter of 1.6 μm. The Fe is visible as a bright dot in the Kerr effect image. The Fe island is not recognizable in the reflectivity image. Both images are collected simultaneously.
Fig. 2: Kerr effect line scan through the Fe island shown in Fig. 1 (open circles). The broken line indicates an exponential curve fitted to the Kerr effect data points. The solid line indicates the derivative of the curve. The full width at half maximum is an upper limit for the diameter of the laser focus.

5.2.3. Stripes of Fe

5.2.3.1. 91 μm wide Fe stripe along the easy axis and its visibility in a reflection image

Fig. 3a shows a surface magnetization map obtained with SKEM. The Fe stripe (visible as a bright area) has a width of 91 μm and a thickness of 3.5 AL. The Kerr signal was measured in an applied field of ±150 Oe. The stripe appears curved and not as straight as it was grown. This is coming from the limited reproducibility of the scanning with the inertial sliders. This can produce a drift which is induced through small scratchers on the sliding face or through thermic drift. A hysteresis loop taken in centre of the stripe is shown in Fig. 3b. The coercive field of the hysteresis is 91 Oe and the shape is square. Accordingly, the stripe is in a single domain state [5,11]. Fig. 3c shows the corresponding reflectivity map. The images (a) and (c) are collected simultaneously. Although the reflectivity for Fe and W are almost the same the Fe stripe is visible in the reflectivity image as well (dark area in the centre of the image). The reflectivity for bulk Fe and bulk W is calculated with the Fresnel formulas [5,12]. For E perpendicular to the plain of incidence we find:
\[ e_\perp = \frac{E_0}{E_0} = \frac{\frac{n \cos \alpha - \left( \frac{\mu}{\mu'} \cdot \sqrt{n^2 - (n^2 \cdot (\sin \alpha)^2)} \right)}{n \cos \alpha + \left( \frac{\mu}{\mu'} \cdot \sqrt{n^2 - (n^2 \cdot (\sin \alpha)^2)} \right)} \]  

(5.3)

and for \( E \) parallel to the plain of incidence:

\[ e_\parallel = \frac{E_0'}{E_0} = \frac{\mu}{\mu'} \cdot \frac{n^2 \cdot \cos \alpha - (n \cdot \sqrt{n^2 - (n^2 \cdot (\sin \alpha)^2))}}{(\mu/\mu') \cdot n^2 \cdot \cos \alpha + (n \cdot \sqrt{n^2 - (n^2 \cdot (\sin \alpha)^2))}} \]  

(5.4)

The reflectivity \( R \) is given by:

\[ R = \frac{|e_\perp^2 + e_\parallel^2|}{2} \]  

(5.5)

\( E \) is the electric field vector of the incident beam and \( E' \) of the reflected beam. \( \alpha \) is the angle between surface normal and incoming laser beam. We set \( \mu/\mu' = 1 \) and use for \( n = N + i \kappa \) the following values; Fe: \( N = 2.92, \kappa = 3.46 \) and W: \( N = 3.82, \kappa = 2.91 \) (photon energy 1.8 eV) [5.13]. For a 45° linear polarized laser beam (\( E_\perp = E_\parallel \)) with \( \alpha = 45^\circ \) equation (5.5) yields \( R_{Fe} = 0.55 \) and \( R_W = 0.50 \). Taking the small difference between \( R_{Fe} \) and \( R_W \) as well as the probing depth of 100 Å into account it is very surprising that the 3.5 Å thick Fe layer is visible in the reflectivity image. We conclude that SKEM has a lateral resolution of only 1 μm but a z-‘resolution’ of a few Å! The resolution in the z-direction depends on the flatness of the sample and more important, on the difference of the reflectivity coefficients. For Co on Cu the effect is more pronounced (see section 5.3.).
Magnetic field [Oe]

Fig. 3: 216 x 216 (µm)² and 64 x 64 pixel Kerr effect (a) and reflectivity (c) image of a W(110) surface with a 3.5 AL thick and 91 µm wide stripe of Fe along the magnetic easy axis (the [1 -10] direction). The iron is visible as a bright stripe in the Kerr image and as a dark stripe in the reflectivity image. Both image are collected simultaneously. (b) shows a hysteresis loop taken in the centre of the stripe.

5.2.3.2. 15 µm wide Fe stripe oriented along the hard axis

A 15 µm wide and 3.5 AL thick stripe of Fe is grown along the hard axis for Fe on W(110). For the measurements in Fig. 4 the external magnetic field is applied along the easy axis and therefore perpendicular to the stripe. Applying an external field up to 500 Oe along the stripe does not induce any measurable magnetization along this axis. The shape anisotropy arising from the stripe's aspect ratio of 200 : 1 is not strong enough to compensate the crystallographic anisotropy and to induce an easy axis along [001]. Fig. 4a shows a Kerr image of the stripe. The Kerr measurement is done in remanence. The bright area corresponds to a positive remanence and the dark area corresponds to zero remanence. The whole stripe is in a single domain state and does not collapse into domains. A hysteresis loop taken in the centre of the stripe has a square shape and a coercive field of 190 Oe (Fig. 4b). Fig. 4c shows the reflectivity image corresponding to Fig. 4a. In contrast to Fig. 3c the Fe stripe is not visible in Fig. 4c. This is due to large reflectivity variations from the substrate itself which are caused by scratches and holes. The black dots on the bottom part of Fig. 4c indicate such imperfections of the substrate. Their large reflectivity signal overshadows the minute difference between Fe and W.
Fig. 4: 36 x 36 (µm)$^2$ and 30 x 30 pixel Kerr effect (a) and reflectivity (c) image of a W(110) surface with a 3.5 AL thick and 15 µm wide stripe of Fe along the magnetic hard axis, the [001] direction. The iron is visible as bright stripe in the Kerr image. In the reflectivity image, the iron is not visible. This is due to the large intensity variations within the image (see text). The hysteresis loop (b) is taken in the centre of the stripe and has a coercive field of 190 Oe.

5.2.3.3. 5 µm wide Fe stripe oriented along the hard axis

In order to increase the shape anisotropy and to compensate the unidirectional anisotropy we reduce the width of the Fe stripe to 5 µm. The aspect ratio is now 600 : 1. The stripe is 12 AL thick and oriented along the [001] direction. This is the magnetic hard axis. Fig.5a shows a 30 x 30 (µm)$^2$ Kerr image. The iron is visible as bright stripe. For the collection of Fig.5a a magnetic field of ±150 Oe is applied along the easy axis. No measurable magnetization is found along the hard axis (along the stripe) for external fields up to 500 Oe applied along this axis. Even an aspect ratio of 600 : 1 is therefor not sufficient to induce an easy axis along the [001] direction. The crystalographic anisotropy of Fe on W(110) can not be overcome by the shape anisotropy. The hysteresis loop displayed in Fig.5b is taken in the centre of the Fe stripe. It has a coercive field of 122 Oe and a square shape. The stripe is in a single domain state. Notice the stripe’s faint visibility in the reflection image (Fig.5c).

Fig.6 shows an array of hysteresis loops taken across the iron stripe shown in Fig.5. The distance between the loops is 0.75 µm.
Fig. 5: 30 x 30 (μm)$^2$ and 60 x 60 pixel Kerr effect (a) and reflectivity (c) image of a W(110) surface with a 12 AL thick and 5 μm wide stripe of Fe oriented along the magnetic hard axis, the [001] direction. The iron is visible as the bright stripe in the Kerr image and as dark stripe in the reflectivity image. The hysteresis loop (b) taken in the centre of the stripe shows a coercive field of 122 Oe.

Fig. 6: Array of hysteresis loops taken across the 5 μm stripe of iron shown in Fig. 5. The distance between the loops is 0.75 μm the coercive field is 122 Oe.
5.2.3.4. Fe cross along the hard and easy axis

Next we evaporated a cross consisting out of 15 μm wide stripes. One is oriented along the hard axis and one along the easy axis. Both stripes are 3.5 AL thick. Fig.7 shows a Kerr effect (a) and a reflectivity (b) image of the cross. A magnetic field of ±120 Oe is applied along the easy axis. Hysteresis loops taken in the centre of the cross (Fig.7c), where 7 AL of Fe have been grown, show twice as much Kerr signal as the hysteresis loops taken on the arms of the cross (Fig.7d). The shape of all measured loops is square and the Kerr image which is taken in remanence shows no contrast within the stripe. Therefor we conclude that the whole cross is in single domain state and does not split into domains. Domains could be induced into the stripe oriented along the hard axis by the dipole field of the stripe along the easy axis. An imperfection of the W substrate which is visible in the reflectivity as well as in the Kerr image is indicated by a circle. The change in the reflectivity caused by this defect is larger than the Kerr signal itself. Therefore the defect is visible in the Kerr image (Fig.7a) as well.

**Fig.7:** 45 x 45 (μm)^2 and 30 x 30 pixel Kerr effect (a) and reflectivity (b) image of a W(110) surface with a 3.5 AL thick and 15 μm wide cross of Fe. The centre of the cross consists out of 7 AL Fe. The hysteresis loop taken in the centre of cross (c) shows twice as much magnetic signal than the hysteresis loop taken in one of the arms. Both hysteresis loops have the same coercive field (86 Oe) and the same square shape.
5.2.3.5. Two parallel 91 μm stripes along the easy axis

Two parallel 91 μm stripes oriented along the easy axis can be controlled independently. The two stripes are 3.5 AL thick and 60 μm apart from each other. Fig. 8a shows a Kerr image measured with an applied field of 250 Oe along the easy axis. Both stripes are magnetized parallel to the external field. This is in contrast to the behaviour observed in Fig. 8b where the stripes are magnetized antiparallel with respect to each other. Fig. 8b was measured in an applied field of 170 Oe and shows the same area as Fig. 8a (the different shape of the stripe is caused by instrumental drift). Fig. 8c and 8d show line scans through Fig. 8a and 8b, respectively. A positive (negative) signal corresponds to a magnetization parallel (antiparallel) to the external field. The unexpected behaviour of the first stripe in Fig. 8b can be explained with the slow magnetization reversal process observed for Fe on W(110) (see section 5.2.5) and the slightly different coercive field for the left (see Fig. 9a) and the right stripe (Fig. 9b). For each image pixel the external field is applied 10 ms before the collection of the data starts. For the left-hand stripe of Fig. 8b a field of 170 Oe (which is 43 Oe above the coercive field) applied during 10 ms is not enough to complete a magnetization reversal. The reversal occurs after the measurement.

![Images of Kerr effect images](image1)

Fig. 8: 400 x 150 (μm)² and 64 x 24 pixel Kerr effect images (a & b) from the same area. The applied magnetic field was ±250 Oe (a) and ±170 Oe (b). Fig. 8c and 8d show a line scan belonging to Fig. a and b, respectively.
Fig. 9: Hysteresis loops taken in centre of the stripes shown in Fig. 8. The hysteresis loop shown in (a) belongs to the left stripe and (b) to the right iron stripe. Note the different coercive fields.
5.2.4. Microsized ultrathin Fe-islands.

In this experiment we have evaporated 4.3 Å thick Fe islands of different lateral size on a W(110) surface. The evaporation technique is explained in section 1. Fig.10 shows Kerr images of three islands which are arranged on one line. Their centres are 10 μm apart from each other. For the collection of the Kerr images the magnetic field was applied along the magnetic easy axis. The hysteresis loop belonging to each island is displayed below the corresponding Kerr image. Island 1 (Fig.10a) is round, has a diameter of 2.4 μm and a coercive field $H_{c1}$ of 465 Oe. Island 2 (Fig.10b) is oval, 2.6 μm wide, 5.3 μm long, and has a coercive field $H_{c2}$ of 400 Oe. Island 3 (Fig.10c) is 2.6 μm wide, 4.7 μm long, and has a coercive field $H_{c3}$ of 450 Oe. The different coercive fields allow an ‘independent’ switching of the islands. Island 2 can be controlled with a field higher than 400 Oe but smaller than 450 Oe. Island 2 and 3 can be reoriented with an external field between $H_{c3}$ and $H_{c1}$. An example of this ‘single island switching’ is given in Fig.11.

![Kerr images of three Fe islands with different lateral size](image)

**Fig.10:** 10 x 10 (μm)$^2$ and 20 x 20 pixel Kerr images of three Fe islands with different lateral size which are arranged on one line. The hysteresis loops belonging to each island is shown below the image. Note the different coercive fields of the islands.
The coercive field \(H_c\) of Fe on W(110) is very sensitive to film thickness and to contamination. After the onset of long range magnetic order at 1.7 AL the coercive field shows a steep increase from zero up to more than 500 Oe around 2 AL. Between 2 AL and 2.5 AL \(H_c\) decreases to 100 Oe and remains almost constant for a coverage exceeding 3 AL. The large value around 2 AL could be induced by the lattice strain in the first two Fe layers which arises from the lattice mismatch between Fe and W. With the beginning of the third Fe layer a lattice relaxation is observed and this could cause the decreasing of \(H_c\). The adsorption of carbon on the top of the Fe layer is found to increase the coercive field as well. Generally we observe that a reduction of the microstructure’s size induces an enhancement of the coercive field. Moreover, the sweeping rate of the
external field has a strong influence on the size of $H_c$. Fig.12 shows two plots of $H_c$ versus the sweeping rate for a 4.3 AL thick Fe film (Fig.12a) and for the 4.3 AL thick Fe island shown in Fig.10b (Fig.12b). The coercive field increases as function of sweeping rate for the film as well as for the island. The best fit for $H_c$ is proportional to $x^{0.52}$. Therefore the observed behaviour does not depend on the size of the sample. A sweeping rate dependent $H_c$ was found by Pommier et al. [5,14] for Co on Au(111) and was explained with the dynamics of the nucleation process of reversed domains and with domain wall propagation.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figures/fig12.png}
\caption{$H_c$ as a function of the sweeping rate of the external field for a 4.3 AL thick film (left hand side) and a 4.3 AL thick island (see Fig.10b).}
\end{figure}
5.3. Co-Microstructures on Cu(001)

5.3.1. Sample preparation

Co microstructures are grown by evaporating Co through a mask or a diaphragm in front of the Cu(001) surface. The evaporation rate was typically 0.01 AL/min and the structures were grown at room temperature.

5.3.2. 5 μm wide Co stripe oriented along the [010] direction

Co grown on a flat Cu(001) surface shows a fourfold crystallographic anisotropy along the [±110] directions. Grown on a stepped surface, Co exhibits a uniaxial anisotropy along the steps [5.15-5.17]. This demonstrates the sensitivity of the magnetic properties to the symmetry and the weak crystallographic anisotropy of Co on Cu(001) system. Thus, it is more likely to find shape induced anisotropies in Co structures than for Fe on W(110).

Fig. 13a shows a Kerr image of a 5 μm wide and 2.7 AL thick Co stripe oriented along the [010] direction which is the hard axis for a Co on Cu(001). The external magnetic field is applied along the stripe. Co appears as a bright area. The hysteresis loop shown in Fig. 13b is taken in the centre of the stripe. Its shape is square and the coercive field is 6.6 Oe. The stripe is clearly visible in the reflectivity image (Fig. 13c) as well. In contrast to Fe on W(110) the reflectivity of bulk Co and bulk Cu are very much different. The Fresnel formulas (see section 5.2.2.1., formulas (5.3) - (5.5)) [5.12] for the reflectivity yield \( R_{Co} = 0.66 \) and \( R_{Cu} = 0.90 \). We set \( \mu / \mu ' = 1 \) and use for \( n = N + ik \) the following values; Co: \( N = 2.61, k = 4.45 \) and Cu: \( N = 0.21, k = 4.04 \) (photon energy 1.8 eV) [5.13]. The calculations were done for a 45° linear polarized laser beam and for an angle \( \alpha = 45^\circ \). \( \alpha \) is the angle between surface normal and incoming laser beam.
Fig. 13: 25 x 25 (μm)$^2$ and 40 x 40 pixel Kerr effect (a) and reflectivity (c) image of a Cu(001) surface with a 2.7 AL thick and 5 μm wide Co stripe oriented along the [010] direction. The external magnetic field is applied along the [010] direction, i.e. along the stripe’s axis. The Co is visible as a bright stripe in (a) and (c). The hysteresis loop (b) is taken in the centre of the stripe and shows a coercive field of 6.6 Oe.

More pronounced is the reflectivity contrast between Co and Cu in Fig. 14c which is collected in a flat area. Co appears as a bright stripe, the reflectivity signal from Co is as high as the Kerr signal (Fig. 14a). The external magnetic field for collecting the Kerr image (Fig. 14a) is applied perpendicular to the stripe, i.e. along [100]. The hysteresis loop in Fig. 14b is taken in the centre of the Co stripe and shows a coercive field of 10.4 Oe. However Fig. 14b does not show an easy axis loop since it has a linear background and is not square shaped. This fact is illustrated in Fig. 15 where Hysteresis loops are taken in an external field up to 250 Oe. For the first loop (Fig. 15a) the external field is applied parallel to the stripe, i.e. along the [010] direction. Its shape is square and the saturation is equal to the remanence. Such a loop is typical for a magnetic easy axis. Perpendicular to the stripe an external field of 250 Oe has to be applied in order to saturate the sample (Fig. 15b). The same behaviour is found in Fig. 15c where the external field is applied along the [110] direction (easy axis for a Co film!). The curves in Fig. 15b and Fig. 15c are typical for intermediate loops and we can conclude that the shape anisotropy of the stripe is strong enough to induce an easy axis along the stripe.

For a stripe oriented 25° off the [010] direction we do find the same behaviour, the stripe is magnetized along its axis and not along the crystalographic easy axis (see Fig. 16). Note that for the stripe shown in Fig. 13 the [010] direction is the easy axis and for the stripe in Fig. 16 the [010] is an intermediate axis.
Fig. 14: 25 x 25 (μm)^2 and 40 x 40 pixel Kerr effect (a) and reflectivity (c) image of the same Co stripe shown in Fig. 14. The external magnetic field is applied perpendicular to the axis of the stripe, i.e. along the [100] direction. The hysteresis loop (b) is taken in the centre of the stripe and has a coercive field of 10.4 Oe. The saturation is not equal to the remanence.

Fig. 15: Hysteresis loops taken in the centre of the Co stripe up to a field of 250 Oe. The applied magnetic field is along the [010] direction for (a), the [100] direction for (b), and the [110] for (c). The loop shown in (a) exhibits an easy axis loop in contrast to the other loops which show intermediate loops.
Fig. 16: Hysteresis loops taken in the centre of the 25° stripe. The external magnetic field is applied along the Co stripe axis (a) and along the [010] direction (b) & (c). The loop shown in (a) is an easy axis loop in contrast to (b) and (c) which are intermediate loops.
Section 6.

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Danksagungen

Jetzt wird gedankt! Und zwar als erstes Danilo Pescia, der mir als mein Doktorvater viel Freiheit während meiner Diss gewährt hat. Auch für seine Geduld mit mir und für die vielen anregenden Diskussionen und Gespräche möchte ich mich hier bei ihm bedanken.

Ganz herzlich danken möchte ich an dieser Stelle auch Prof. Dr. H. C. Siegmann für die Übernahme des Koreferats. Auch erinnere ich mich gerne an die Zeit zurück, in welcher ich in seiner Forschungsgruppe meine Diplomarbeit fertigen durfte und meine ersten Gehversuche in der Experimental-Physik machen konnte.

Ein ganz besonderes Dankeschön bin ich Andreas Vaterlaus schuldig. Er hat als mein Oberassistent die schwere Bürde der Verantwortung auf sich genommen und mich vorzüglich durch meine Diss geführt. Unvergesslich bleiben die etlichen Labortage, die wir zusammen in unserem Messeifer verbracht haben und vor allem unvergesslich bleibt die Ionenpumpe-Ab-und-Aufmontierungs-Stress-Nacht.

Für die hervorragenden Leistungen in Sachen Technik, STM und UHV-Aparatur möchte ich mich bei Urs Maier bedanken. Dass die Aparatur überhaupt so schnell Zustände gekommen ist und solch interessante Resultate gemessen werden konnte, war zu großem Teil sein Verdienst. Ausserdem hat er meinen musikalischen ‘Wortschatz’ um diverse Bands vergrössert (Yeah).

Bei Christoph Würsch möchte ich mich als erstes dafür bedanken, dass er mich ertragen hat. War wohl nicht ganz so einfach! Zweitens für die ständige Hilfsbereitschaft, sei es nun in LabView Programmierung oder in physikalischen Theorien. Auch unsere gemeinsame Konferenz-Zeit in den USA wird mir in angenehmer Erinnerung bleiben, vor allem der New York-Florida Marathon. Danken möchte ich Dir auch für Deinen netten Dank in Deiner Diss! (Wirklich, ganz nett!)
