Magnetic force microscopy of a CoCr thin film

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We present high-resolution magnetic force microscopy (MFM) images of a Co₈₀Cr₂₀ film with a thickness of 230 nm. We clearly observe a stripe domain structure with a domain width of 220 nm, the highest resolution of a periodic magnetic structure measured by MFM to this date, by measuring forces as small as 0.1 nN. The micromagnetic structure is compared to the morphology of the sample, which consists of vertical columns with an average cross section of about 40×60 nm². The average surface roughness of this film did not exceed 12 nm on a 1- μ m lateral scale. A magnetic fine structure of typically 50 nm width and more than 200 nm length is observed. Theoretical calculations of force traces are in reasonable agreement with experiment. A nonuniform sample magnetization, correlated to the morphology, has to be assumed to simulate the observed fine structure of force traces. Calculated images characteristically depend on the effective domain structure of the last 500 nm of the tip.

I. INTRODUCTION

Polycrystalline CoCr thin films are promising candidates for ultra-high-density storage media 1,2 where densities of 100 Mb/cm² are expected.³ Micromagnetic domain structure information is necessary in order to improve the understanding of these films and to interpret macroscopic observations.4 Such microscopic investigations have been made on CoCr by the Bitter pattern technique, by means of the magneto-optic Kerr effect,5 or by Lorentz microscopy.6 Recently, force microscopy⁷ has been applied to the study of magnetic samples on a submicrometer scale.^{8,9}

The force sensor in magnetic force microscopy (MFM) consists of a sharp ferromagnetic tip mounted on a cantilever. Forces acting on the tip cause a deflection of the cantilver, which can be measured by electron tunneling, 7,9 or result in a change of resonance frequency, which can be detected by interferometry^{8,10} or piezoelectrically.¹¹ The measured interaction forces depend on the magnetic structure of the sample and the physical properties—such as magnetization and geometry—of the tip. 12 Measurable magnetic forces are a result of gradients of magnetization9 or are associated with local changes of the sample magnetic moments induced by the magnetic field of the tip. 13 Lateral magnetic resolutions of less than 10 nm has been reported for domain walls, a nonperiodic structure, in Fe-Nd-B.14

In this paper we present images of 220-nm-wide domains of Co₈₀Cr₂₀ thin films as measured by MFM. To elucidate the origin of fine structure with a width of less than 50 nm observed on MFM traces we simulated numerous images by calculating the tip-sample interaction. Resonable overall agreement between simulated and experimental traces was thus obtained. From simulations assuming a tilted sample magnetic structure it can tentatively be concluded that the observed magnetic fine structure might be due to a nonuniform sample domain structure.

II. METHODS

The instrument used in our experiments is described in detail in a previous paper.15 It was operated in the variable deflection mode in the regime of attractive forces when measuring magnetic structures and in the regime of repulsive contact forces when topographic information was needed. Images were obtained by scanning the sample in x and y and simultaneously measuring the cantilever deflection in z. All measurements were performed in ambient air on a passively dampened optical table. Magnetic force sensors were cut from 10- μ m Ni foil and electrochemically etched to a tip of less than 100-nm radius with an apex angle of 10° at one end. They were not subjected to any magnetic pretreatment. Typical force constants of the lever are 0.1 N/m. The angle between the force sensing tip and the sample was approximately 55°. Some topographic measurements were done with microfabricated SiO₂ force sensors.

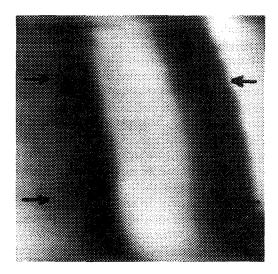
The 230-nm-thick Co₈₀Cr₂₀ sample investigated was sputtered on a polyimide (Kapton) support with a Ti underlayer. Cross-section TEM images clearly show a columnar structure with an average column diameter of about 50 nm. 16 Hysteresis measurements give a saturation magnetization $4\pi M_s = 4400$ G and a coercivity $H_c = 920$ Oe in the perpendicular direction. The in-plane remanent magnetization is 1.86 and the in-plane coercive field is 3.03 times smaller than the corresponding perpendicular components.16

Calculations of the lines of force were done by the same method as presented in previous publications (see Ref. 12 and references therein). Analytical formulas describe the magnetic field above a sample of constant thickness with perpendicular anisotropy and a periodic domain structure. transitions between domains are modeled as being infinitely sharp, which is a sensible approximation as the domain period is comparable to force sensor tip dimensions. The tipsample interaction can then be calculated by assuming a truncated pyramid as a tip model. 12 The calculation is much easier if the magnetization direction is changed instead of the geometric axis of the model tip in order to account for any tilt of the force sensor with respect to the sample in the experiment. The calculated forces thus have a slightly smaller peak-to-peak force amplitude and a better lateral resolution than the results of the more complicated simulation with a

tilted geometric tip axis. This is due to the larger proportion of volume close to the sample in the latter case. In extension to previously published calculations we can incorporate a domain structure in our tip model in order to better simulate the measured lines of force and their distance dependence.

III. RESULTS

Typical images obtained with a Ni force sensor on a $Co_{80}Cr_{20}$ sample show a stripe structure with a periodicity of 440 nm [Fig. 1(a)]. It is not possible to determine the exact absolute value of the forces with the present instrument due to thermal drift and unavoidable piezo creep; the maximum force variation observed in this image is about 2.7 nN. The noise level during these scans was well below 0.1 nN, and the typical image acquisition time was 60 s.



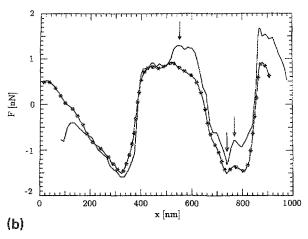


FIG. 1. (a) A stripe structure with a 440-nm period is typically observed on MFM image of a $\rm Co_{80}Cr_{20}$ thin film. The total gray scale range corresponds to a variation of 2.7 nN of the attractive force. The acquisition time was about 60 s for this image with a side length of 900 nm. (b) The two scans are separated by more than 300 nm in (a), where their position is indicated by arrows. They were shifted in x with respect to each other to compensate for the slant of the stripe structure observable in (a). Single traces clearly show a 440-nm periodicity, a slight asymmetry, and fine details (examples marked by arrows) that are typically at least 200 nm long, 50 nm wide, show a force modulation of less than 0.5 nN, and run parallel to the stripe structure of (a).

Single-scan lines demonstrate this 440-nm periodicity even clearer [Fig. 1(b)]. All traces show a slight asymmetry and fine details (marked by arrows) that are typically at least 200 nm long and 50 nm wide, show a force modulation between 0.1 and less than 0.5 nN, and run parallel to the stripe structure of Fig. 1(a). In some cases the length exceeds the total scan range of 900 nm; the structures observable between 700 and 850 nm are an example for this case. It is difficult to see the fine structure in Fig. 1(a) due to the limited number of gray scales available for encoding the large dynamic range of forces. This fine structure is magnetic in nature, as the interaction is long-ranged and the overall lateral dimensions are much larger than that of topography.

We tried to simulate these scans by choosing a model tip with dimensions well comparable to those of experimental force sensor tip. We considered a variety of magnetic structures of the tip as well as that of the sample (Fig. 2). The calculated force traces for both single- and two-domain tip model are a result of the interaction of the tip magnetization with the perpendicular and in-plane components of the magnetic sample stray field and its distance dependence as presented in Ref. 12. The force traces calculated for the CoCr sample used in the experiment show only slight changes of the order of pN if the model tip is made longer than 500 nm, we thus limited its length to this size. The tip sample separation in the simulation was set to 15 nm. van der Waals forces were included although at this separation their contribution is calculated to be much smaller than 0.1 nN.

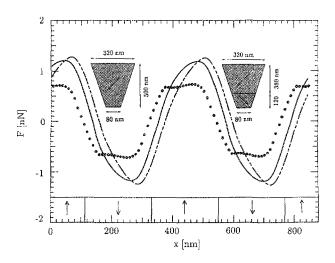


FIG. 2. Force traces for two different tips, modeled as truncated pyramids, and simulated for a sample with perpendicular anisotropy, periodic stripe domain structure of 440-nm period, and magnetic parameters corresponding to the measured ${\rm Co_{80}Cr_{20}}$ film. Alternatively, the direction of the sample magnetization was tilted by 20°. The domains are indicated in the lower part of the figure. The trace with the amplitude of 2.4 nN (full line) arises from the single-domain model tip (left inset). The angle of 202° for the magnetization was chosen to account for the experimental tilt of the force sensor tip with respect to the sample. The second trace (dotted) of the 1.4-nN peak-to-peak force amplitude, showing a distinct substructure, results from a model tip with a domain structure (right inset). The tilt of the domain with a length of 120 nm is 140°, the second, larger domain is tilted 260°. The third trace (dashed) is the result of the interaction of the single-domain tip and the sample with the tilted magnetic structure and leads to a peak-to-peak amplitude of 2.8 nN.

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(a)

The magnetization direction of the tip with a single domain (dimensions indicated in the inset of Fig. 2) was chosen to account for the measured angle between tip and sample in the experiment. An asymmetric, structureless trace is calculated for this case with a peak-to-peak force amplitude of 2.4 nN and a good overall quantitative similarity to the experimental traces of Fig. 1(b).

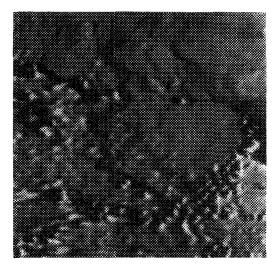
If the magnetic tip is modeled with two domains (dimensions indicated in the inset of Fig. 2), the peak-to-peak force amplitude becomes smaller (for the case plotted 1.4 nN), the resulting traces less asymmetric, more square in shape, and a substructure of less than 0.1 nN amplitude appears. In order to enhance the amplitude of the substructure, this trace was calculated at a tip sample separation of 5 nm. Changes in the length or the angles of the two domains alter the amplitude or position of this substructure, as does a change in the number of domains taken to model the effective tip magnetization. Although we simulated numerous tip domain structures, we could not obtain a force amplitude of close to 2.7 nN with a substructure amplitude above 0.1 nN. These model domains do not have to correspond to physical real domains; they should rather be regarded as the mathematical representation of a nonuniform tip magnetization, which is a result of the physical domain structure of the tip, but also of the geometry-dependent demagnetization field and very local, surface-roughness-dependent stray fields.

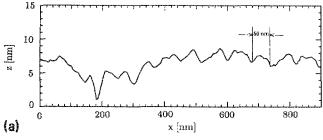
If the sample is modeled with at tilted magnetization, the peak-to-peak force amplitude can change by more than 0.2 nN when compared to the results obtained with the identical tip model and a perpendicular magnetized sample. Furthermore, a slight change in the position of the extrema is observable, the symmetry of the trace is however unchanged. The corresponding simulation for the multidomain tip differs insignificantly from the result for the perpendicular magnetized sample.

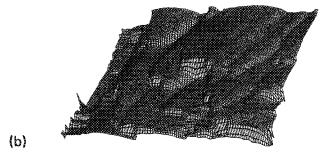
In constrast to the magnetic structure, the typical sample morphology measured in the repulsive contact regime seems to consist of individual grains with an average diameter of 50 nm. The sample is fairly flat, and a maximum roughness of 12 nm is observable. No similarity to the images and traces acquired in the attractive regime (Fig. 1) is observable. Figure 3(a) shows a typical image of 900×900 nm² obtained by measuring the lateral variation of the repulsive contact forces with a sample loading of some 10 nN and thus is a good measure of surface topography. The contribution of magnetic forces to this image, however, cannot be totally neglected as the identical force sensor as in the images of Figs. 1 and 4 was used.

Higher-resolution images measured with a microfabricated SiO_2 force sensor such as Fig. 3(b) indicate a slightly elongated shape with typical grain dimensions of 40×60 nm [Fig. 3(b)]. This can be quantified best by comparing single-scan lines taken in orthogonal directions [Fig. 3(c)]. The grain morphology and dimension correlates very well with the columnar structure as observed by cross-section TEM images. ¹⁶

The distance between sample and force sensor can be changed by applying a suitable dc voltage to the sample z-piezo. Figure 4 shows traces of images acquired at different







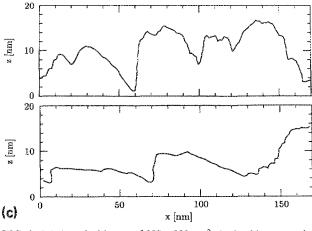


FIG. 3. (a) A typical image of $900\times900~\text{nm}^2$ obtained by measuring the lateral variation of the repulsive contact forces and thus is a good measure of surface topography. A maximum roughness of 12 nm is observable, encoded with a gray scale, and measurable from the trace. Grains about 50 nm in diameter can be detected. (b) The sample topography on a $170\times170~\text{nm}^2$ scale as observed with a nonmagnetic microfabricated SiO_2 force sensor. Clearly different, slightly elongated grains are observable. The grain dimension and morphology corresponds very well to the columnar structure as observed by cross-section TEM (Ref. 16). (c) Single traces from (b), allowing the precise determination of grain dimensions. The top trace is taken in x, while the lower trace is in the y direction, demonstrating the slightly elongated grain morphology.

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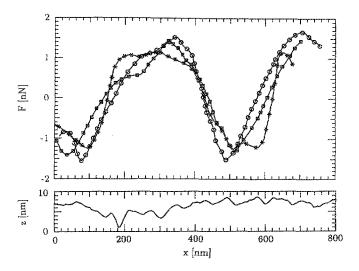


FIG. 4. Three sinusoidal traces in the top part of the figure are from images measured fairly far from the sample surface. The trace marked by circles (reference) was measured about 80 nm closer to the sample than the one marked with stars; the reference is about 20 nm closer than the trace marked by squares. The absolute distance between the sample and the reference could not be determined due to problems mentioned in the text. The trace in the lower part of this figure was acquired in the repulsive contact force operation mode of the microscope and thus is a measure of topography. It is from Fig. 3(a) and clearly demonstrates that the topography and magnetic structure of this sample are very different.

relative force sensor to sample distances. The three sinusoidal traces in the top part of Fig. 4 are from images measured fairly far from the sample surface in the attractive force regime. At first sight these three traces, representative for images obtained at relative distances 60 and 80 nm closer to the sample, look similar. This z independence is expected if the main contribution to the force corrugation is magnetic in nature, as the magnetic force gradient is very small and of the order of 10^{-4} N/m at a distance of 100 nm from the sample. These three traces differ significantly from the fourth trace in the lower part of Fig. 4. This trace corresponds to surface topography as it was obtained by measuring repulsive contact forces.

If the three sinusoidal traces are analyzed more closely, they reveal subtle differences. The peak-to-peak amplitude of the trace obtained closest to the sample (and marked by circles) is 3.3 nN and changes to 2.7 and 2.4 nN for increasing tip-to-sample separation. This corresponds to a 30% difference in force corrugation for a change in the interaction distance of 80 nm.

The relative lateral displacement of the individual traces was corrected for as exactly as possible. This correction is difficult as the determination of thermal drift and piezo creep in the direction parallel to the stripes is inaccurate due to a lack of characteristic features in this direction. The absolute force sensor-to-sample distance is unknown, as the force sensor suddenly becomes unstable when approaching the sample. Such instabilities are expected when the sample force gradient is larger than the lever constant. The point of instability depends on the lateral position of the force sensor with respect to the sample, as the total sample force gra-

dient is the sum of both van der Waals and magnetic force gradients, the value and sign of the latter showing a large lateral variation. The force sensor is finally stabilized again by repulsive contact forces. As a result the difference in sample to force sensor distance between the trace marked by circles and the sample surface (lower part of Fig. 4) cannot be determined precisely.

IV. INTERPRETATION AND DISCUSSION OF RESULTS

It is possible to observe by MFM the micromagnetic structure of a CoCr thin film with a stripe domain structure and a domain width of about 220 nm with very high resolution difficult to achieve by other techniques on this sample. The observed magnetic fine structure with a 50 nm width and a length of at least 200 nm is sample specific. By comparison with image simulations we tentatively attribute this magnetic fine structure to a local variation of the sample magnetization direction. We observe a mean column dimension of 40×60 nm², which is substantially smaller than the domain width of 220 nm.

We conclude that magnetic forces are measured from the differences observed when imaging repulsive, topographic forces. A further confirmation of this interpretation is the first-order distance independence of the measured forces, which points to a small force gradient of the measured interaction. Thus the forces measured are predominantly magnetic in nature, as topographic (van der Waals) forces have a large force gradient. ^{9,13,18} A similar stripe domain structure for certain CoCr samples has previously been observed by Kerr^{4,5} and Lorentz microscopy. ¹⁹

The overall correlation of the measured force traces [Fig. 1(b)] with the simulation assuming no tip domain structure (Fig. 2) is good. The observed general shape and magnitude of force is well reproduced in the realistic theoretical calculation. An indirect approach has to be chosen to understand the observed magnetic fine structure. We will proceed as follows: first, we will discuss the simulations involving a nonhomogeneous tip domain structure, leading to the conclusion that the observed magnetic fine structure is sample specific. We then assume that the correlation of the characteristic width of the fine structure of about 50 nm with the typical column diameter is not a coincidence. This points to a possible origin of the fine structure as being due to a local variation in the sample magnetization direction, as simulations show that a tilt of the sample magnetization leads to a change in amplitude and position of the extrema of the force traces. The currently employed simulation method is based on a periodic sample magnetic structure model (see Ref. 12 and references therein). Thus deviations from this periodicity, such as magnetic defects, cannot presently be simulated directly.

As indicated above, we will now first discuss the simulations attributing the origin of fine structure to a tip domain structure (dotted line in Fig. 2). Such a nonhomogeneous tip magnetization might explain some details, but we are convinced that most of the fine structure is really a sample specific magnetic property, as the effect of a tip magnetic structure should manifest itself on the traces of all domains and have the same length as these. This is evidently not the

case; in Fig. 1(b) the fine structure in the two minima of the force traces is different and the observed length is sometimes limited to 200 nm. We also could not simulate the correct peak-to-peak force amplitude and simultaneously a fine structure amplitude above 0.1 nN assuming a multidomain tip. Furthermore, there is no reason why the width of the fine structure, observed with different magnetic force sensors, should be similar and often about 50 nm.

As simulations show (dashed line in Fig. 2), a tilt of the whole sample magnetization direction leads to a shift of the position and a change in the amplitude of the force trace. It is thus reasonable to assume that a magnetic defect (i.e., a local tilt of the sample magnetization direction) will give rise to fine structure. Referring to Fig. 2 and assuming a tilt of the sample magnetic structure between 500 and 550 nm, the resulting force trace can be visualized to zeroth order by following the full line in Fig. 2 and skipping to the dashed line between 500 and 550 nm. This produces a smooth trace with a fine structure due to a magnetic defect. The correlation of the characteristic fine structure width of 50 nm with the typical dimension of a single grain would then be no concidence, as the easy axis of magnetization is along the c axis of the hcp CoCr crystallites. The texture of the CoCr thin film is not absolutely perfect; the c-axis orientation might however show some degree of local correlation, possibly more along a certain direction due to external parameters such as sputter direction, magnetic fields, or substrate stress during film deposition. As argued above, this would locally modify the magnetic forces and possibly explain the observed fine structure. Furthermore, the existence of small reversal domains at the surface, known as spikes, cannot be excluded (see Ref. 20 and references therein). Such reversal domains would also locally modify the measured force traces.

Simultaneous measurement of topography and magnetic forces would help to verify this interpertation. The magnetic contrast observed might further be enhanced by the magnetic "anisotropy" force, ^{13,21} as CoCr is very anisotropic. From the theoretical simulation we see that the localization of the domain transition region is dependent on many parameters, among them the model sample magnetic structure. Domain walls are expected to be very narrow (to the authors knowledge no direct determination has been published) and difficult to observe experimentally by MFM as the domain period is comparable to the physical tip dimension. ²² Domain walls thus probably cannot be directly correlated to the observed fine structure.

An important issue is whether it is possible to observe magnetic structures 50 nm in size when the tip has an extended geometry and is 100 nm away from the sample. Recent theoretical simulations show that it might be difficult to achieve a resolution comparable to the dimension of the observed fine structure with realistic tip geometries at a distance of 100 nm. ¹² Experiments on FeNdB, however, demonstrate a 10-nm resolution with similar force sensor tips as employed in this experiment, ^{14,22} thus pointing to the possibility that the effective tip magnetization volume is smaller than the geometrical tip dimensions as determined by scanning electron microscopy. A slight variation of about 10% in domain width is observable. The variations, however, do not

seem to correlate spatially with typical grain dimensions. All the domains observed are about 4 times wider than the mean grain size of 50 nm. As this number is significantly larger than 1, this suggests that there is significant exchange coupling across column boundaries. Domain widths several times larger than the average grain dimension are reported for certain CoCr thin films in the literature. 4.5,19,23

One of the open problems is the interpretation of the experimentally observed distance dependence (Fig. 4), which is closely linked to the problem of determining the absolute force sensor to sample distance. If this distance is small, then the magnetic force gradients are large; the sample roughness will lead to a local variation of the separation between tip and sample and thus of the magnetic force that should not be neglected in a realistic simulation. Furthermore, the contribution of attractive van der Waals forces (topography) to the measured total force can be substantial at very small separations. If we assume a small and constant separation of 15 nm between force sensor and sample and calculate the force traces, we get a peak-to-peak value of 2.4 nN for the tip geometry chosen. The contribution of van der Waals forces at this distance is already very small and amounts to less than 0.1 nN. The distance dependence, however, of the peak-to-peak force is much larger than the experimentally observed 30% for a change in distance of 80 nm. On the other side, assuming a large separation of about 100 nm between force sensor and sample also leads to an inconsistency with experiment: The 30% relative change in peak-to-peak force amplitude for a 80-nm change in interaction distance is now reasonable, not so, however, the large absolute value of 2.7 nN.

It is currently not possible to answer this question and thus not possible to discuss the experimental observed distance dependence. Either the simulation lacks an important contribution to the force modulation ("anisotropy" force?) or the sample position measurement uncertainty is much larger than the estimated 10 nm.

V. CONCLUSIONS

We conclude from the results and discussion presented above:

- (1) The stripe domain structure of a $\rm Co_{80}Cr_{20}$ thin film with a domain width of 220 nm is observable by MFM in air with very high resolution without any special sample preparation.
- (2) Magnetic fine structure with dimensions smaller than 50 nm is observable. This fine structure is probably sample specific and might be attributed to local variations of the sample magnetization direction resulting from variations in the c-axis orientation of the columns characteristic for this film.
- (3) The morphology of the film consists of grains with an average dimension of $40 \times 60 \text{ nm}^2$ and a surface roughness not exceeding 12 nm on a 1- μ m scale.
- (4) A statistical correlation of micromagnetic structure to topography is possible; the domain width is about four times larger than the average grain size, suggesting that there is a significant exchange coupling across column boundaries.
 - (5) In order to interpret MFM measurements, image

simulation is very helpful. Reasonable overall agreement could be obtained when comparing experiment and theoretical calculations. Discrepancies are probably due to the assumptions of static magnetic moments in the sample (thus neglecting "anisotropy" forces 13,21), the approximation of the sample domain structure used to calculate the sample stray field, the assumed identity of the geometric and effective magnetic volume of the tip, and the uncertainty in the absolute sample position. It would be very helpful if magnetic defects could be simulated.

(6) The correct interpretation of the distance dependence of the observed force traces is open.

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- ²¹This "anisotropy" force has nothing to do with the normal magnetic anisotropy interaction arising from spin-orbit coupling. It is a result of the orientation dependence of the reaction of the magnetic moments of the sample to the presence of an external magnetic field, which is identical to the force sensor stray field. This force is always attractive and can be related to the magnetic susceptibility of the sample; it only has a nonzero value if the elements of the susceptibility tensor are not all equal, i.e., are orientation dependent or "anisotropic." The susceptibility tensor might show some local variation, too.
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