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Application of off-specular polarized neutron reflectometry to measurements on an array of mesoscopic ferromagnetic disks

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Using off-specular polarized neutron reflectometry with neutron spin analysis, we determined the magnetic properties of a large array of in-plane magnetized ferromagnetic Co disks. Resonant peaks are clearly observed in the off-specular reflectivity, due to the lateral periodicity of the disk array. Using polarized neutrons, the intensity of the resonant peak in the off-specular reflectivity is studied as a function of the magnetic field applied in the sample plane. Spin analysis of the reflected neutrons reveals the magnetization reversal and saturation within the disks. © *2001 American Institute of Physics.* [DOI: 10.1063/1.1389764]

Recent advances in lithography and deposition techniques have created the possibility to produce high-quality micron and nanometer sized magnetic structures.[1] The motivation is twofold: first, new physical effects emerge when the mesoscopic regime is explored in which the size of the magnetic structures becomes comparable to some relevant physical length[2] or when the magnetic entities interact with each other,[3] or with a semiconducting layer,[4] or a superconducting layer.[5,6] The second reason is that the newly gained insight into the physics of mesoscopic magnetic structures is applied to large-scale industrial applications in, e.g., magnetic storage media, computer memories, and sensors. The most common experimental tools which are used in the study of these magnetic structures are typically magnetization measurements, electrical transport (magnetoresistance), and ever more frequently imaging methods like magneto-optical Kerr microscopy or magnetic force microscopy (MFM).

The structural properties of arrays of dots have been investigated by means of specular and off-specular x-ray reflectivity measurements.[7] By means of the magneto-optical Kerr effect (MOKE), different reflection orders can be probed to study the magnetic properties of magnetic arrays.[8,9] It has remained an interesting challenge, however, to exploit the huge potential of (polarized) neutron reflectometry (PNR)[10–14] in the study of the structural and magnetic properties of patterned magnetic elements.[15,16] In this letter, we report on the observation of resonant peaks in off-specular reflectivity using polarized neutrons with spin analysis to monitor the magnetization reversal in a huge array of ferromagnetic disks.

Periodic arrays of magnetic disks were produced by UV lithography and molecular beam deposition. A Si/SiO_2 wafer was covered with a resist mask, into which the film was deposited. A lift-off step removes the photoresist. Each disk consists of a Au (75 Å)/Co (200 Å)/Au (75 Å) trilayer. Co is evaporated from an electron beam gun at a rate of 0.45 Å/s, while Au is evaporated from a Knudsen cell at a rate of 0.25 Å/s, both at a working pressure of 10^{-10} mbar. X-ray diffraction indicates that the disks are polycrystalline. The total patterned area of the sample is 2×2 cm^2. Figure 1 shows an optical microscope picture of the sample. The disks have a diameter of 4 μm and are placed in a square pattern with sides of 10 μm. As indices for the directions on the sample surface (lower right inset of Fig. 1), {10} and {01} are used for the directions along the rows of disks, while {11} indicates the diagonal direction of the array. The upper left inset of Fig. 1 shows a more detailed picture of a single disk, obtained by atomic force microscopy (AFM). The AFM results show that the surface roughness of the disks equals 7 ±1 Å on an area of 1 μm^2, which is comparable to that of an unpatterned film. The magnetic domain structure within the disks was imaged by MFM, revealing that the disks are typically in a multidomain state. MFM images of the remanent state after saturation in a magnetic field parallel to the substrate plane reveal that all disks have a very similar domain state. This implies that the magnetic behavior measured

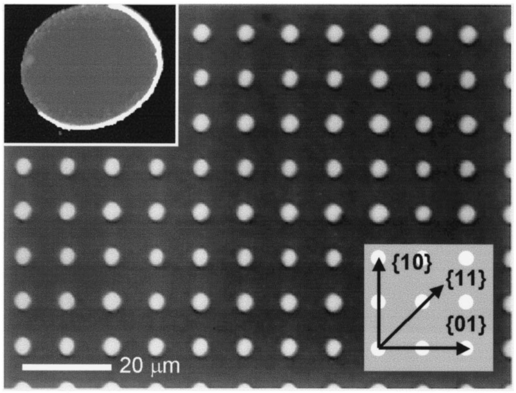

FIG. 1. Optical microscope picture of the sample, showing the arrangement of the Co disks in a square lattice with a 10 μm period. The length of the white bar corresponds to 20 μm. The inset (upper left corner) shows an AFM image of a single disk. In the lower right corner the in-plane directions are defined.

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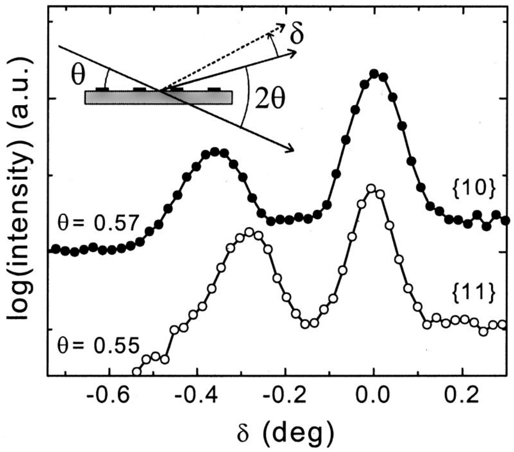

FIG. 2. Specular and off-specular reflectivity for a neutron beam along the {10} direction ($\theta = 0.57°$; closed symbols) and {11} direction ($\theta = 0.55°$; open symbols). The inset shows the scattering geometry. For clarity, the curves are shifted vertically. In both cases, the peak at $\delta = 0$ corresponds to the specular reflection, while the peak at $\delta < 0$ is due to the lateral periodicity of the disk array.

in the PNR experiments may be related to the properties of an individual disk. The in-plane saturation field H_s of the disks was determined from superconducting quantum interference device (SQUID) magnetization measurements to be about 400 Oe.

The PNR experiments were performed at the V6 reflectometer at the Hahn-Meitner-Institut, Berlin, using cold neutrons (wavelength $\lambda = 0.466\,\mathrm{nm}$). This reflectometer uses a graphite monochromator, a liquid-nitrogen cooled Be filter, a set of two diaphragms, and a polarizing supermirror, creating a polarized, monochromated, well-collimated neutron beam.[17,18] The experiments were carried out at room temperature and in ambient conditions. A magnetic field H parallel to the film plane (and perpendicular to the path of the neutron beam, i.e., perpendicular to the scattering plane) was applied using an electromagnet. The neutron spin was aligned parallel or antiparallel to the applied field by a Mezei-type flipping coil. The neutron intensities were measured using a position-sensitive detector, specifically, a multiwire proportional counter with an active area of $180 \times 180\,\mathrm{mm}^2$ and a spatial resolution of about 1.5 mm.

Specular reflectivity measurements with a polarized neutron beam did not provide information about the magnetic state of the disks, i.e., no splitting between the spin-up and spin-down reflectivities was observed. The specular reflectivity is dominated by the nonmagnetic substrate since only 12% of the sample surface is covered by magnetic material. We can obtain magnetic information, however, from the *off-specular* reflectivity which shows resonant intensity peaks resulting from the in-plane periodicity of the disk array, analogous to the interference pattern of a laser-illuminated optical grating. Figure 2 shows the reflected intensity for a neutron beam incident along the {10} and {11} directions. The angle of incidence θ was 0.57° and 0.55°, respectively. The inset schematically shows the scattering geometry. These data have been corrected for the background intensity from the directly transmitted beam. The peak at $\delta = 0$ originates from the specular reflection. Due to sample roughness, some intensity will also be scattered into the off-specular ($\delta \neq 0$) direction.[19] A patterned sample presents a special type of roughness: periodic roughness with a well-determined amplitude and lateral length scale. The off-specular reflectivity then shows a clear set of satellites, which can be attributed to the lateral periodicity.[7] These satellites are visible at the left of the specular peak (Fig. 2). Satellites at $\delta > 0$ can also be expected, but with a lower intensity, because of the larger magnitude of the scattering vector and hence a lower reflectivity. In this particular experiment, no satellites at $\delta > 0$ were observed. They were visible, though, in other scans with smaller θ. The position of the satellites scales with θ according to the formula $q_x = (2\pi/\lambda)[\cos(\theta+\delta) - \cos(\theta)]$ with q_x the in-plane component of the scattering vector, which is related to the real space periodicity $d = 2\pi/q_x$. The position of the satellites ($\delta = -0.36$ for {10} and $\delta = -0.28$ for {11}) reflects the periodicity $d = 2\pi/q_x = 10$ and 14 μm along {10} and {11}, respectively.

In order to extract magnetic information about the disk lattice, experiments with polarized neutrons were carried out with spin analysis of the reflected neutrons. θ was fixed at 0.5° (i.e., $q_z \approx 0.024\,\text{Å}^{-1}$), while H was increased from 0 to about 200 G. The magnetic state of the disks was studied by monitoring the integrated intensity of the off-specular satellite. The intensity of the four polarized beam cross sections (the four possible combinations of incident and reflected neutron spin) was measured as a function of H. The nonspin flip (NSF) intensities (I^{--} and I^{++}) are generated by magnetization components (anti)parallel to the neutron spin, while the spin flip (SF) intensities (I^{-+} and I^{+-}) are generated by magnetization components perpendicular to the neutron spin. Figure 3 shows the intensity of the off-specular satellite as function of H for two different initial configurations obtained after saturating the disks in a field H_{prior} along different directions. In Fig. 3(a) the neutron beam was in the {11} direction, and H was in the sample plane, perpendicular to the neutron beam. The sample was initially in the remanent state after saturation with H_{prior} opposite to H. The SF contribution is almost zero at all H, indicating that there are negligible magnetization components perpendicular to the neutron spin. At the smallest H, $I^{--} > I^{++}$, indicating that a certain fraction of the disks has a remanent magnetization component parallel to H_{prior}. As H is increased and magnetization reversal within the disks sets in, I^{++} increases while I^{--} decreases. This splitting between I^{++} and I^{--} saturates when the disk magnetization is saturated in the direction of H. Note that the off-specular intensities do show clear magnetic splitting, in contrast to our observations of the specular reflectivity. Due to the small film thickness, the disk magnetization is confined to the sample plane and can be reversed by domain wall motion and/or coherent rotation. No increase of the SF intensities is observed, indicating that there is no coherent rotation of the magnetization (during which magnetization components perpendicular to the neutron spin would arise). Rather, it can be inferred that the reversal takes place by domain wall motion, starting at domains that are already favorably oriented. Figure 3(b) shows the magnetization reversal in the disks after saturation with H_{prior} perpendicular to H and to the neutron spin. At $H = 0$, there is now also a SF contribution, confirming that there is a remanent fraction of

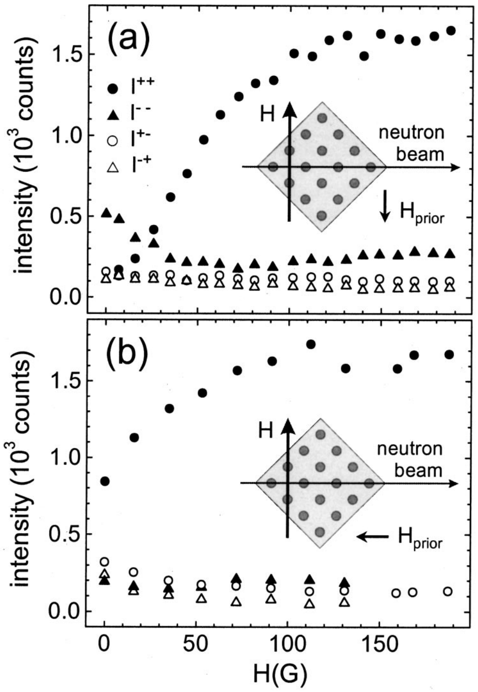

FIG. 3. Intensity of the resonant peak in the off-specular scattering as a function of the field H applied along the {11} direction. The four possible combinations of incident and reflected neutron spin are shown: I^{++} and I^{--} correspond to nonspin-flip scattering while I^{+-} and I^{-+} correspond to spin-flip scattering. In (a) the sample was initially in the remanent state after being saturated in a field H_{prior} antiparallel to H. In (b) the sample was initially in the remanent state after being saturated in a field H_{prior} perpendicular to H. The insets show the scattering geometry and the direction of H_{prior}.

magnetization oriented perpendicular to the neutron spin. As the field is increased, the magnetization of the disks is forced into the direction of the external field, reducing the magnetization component perpendicular to the field and, hence, also the SF scattering, as can be seen most clearly in the I^{+-} and I^{-+} signals. At the same time, the NSF intensity I^{++} increases and maximal splitting between I^{++} and I^{--} is achieved when the disks are saturated in the direction of H.

In conclusion, we have demonstrated that off-specular reflection experiments of polarized neutrons combined with spin analysis can be carried out on patterned magnetic structures. Both, off-specular reflectivity and the four polarized beam cross sections are used to determine the magnetization reversal process and the approach toward saturation within the magnetic disks. An important advantage of this technique is that simultaneously both in-plane components of the magnetization vector are probed by measuring the SF and NSF intensities. In order to evaluate the data more quantitatively, a theoretical framework for the calculation of off-specular polarized neutron reflectivity from magnetic islands would be very desirable. This type of measurement can be extended to complete "mapping" of the reciprocal space near the origin, and could provide information about magnetic order in the plane of the disk array as well as along the growth direction of the magnetic islands. The success of this experiment implies that the specific advantages of neutron reflectometry (noninvasiveness, depth sensitivity, complete vectorial determination of the magnetization directions) are applicable to investigate the manifestation of important magnetic effects such as anisotropy, antiferromagnetic coupling in layered nanostructures, dipolar coupling between nearby entities, exchange bias, etc., in *patterned* magnetic structures.

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