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Multiprobe magnetoresistance measurements on isolated magnetic nanowires

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We present magnetotransport measurements on single cobalt nanowires fabricated by electrodeposition into nanoporous membranes and contacted by an electron beam lithography technique. Isolated nanowires (up to 20 μm long) are extracted from the nanoporous template and spread over a silicon substrate. Electron-beam lithography allows us to make nanosized multiprobe connections on isolated wires and perform measurements on parts of the nanowire as small as 500 nm. Measurements of different segments of a single 60 nm Co nanowire show the inhomogeneity of magnetization reversal processes along the same wire.
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Magnetic nanowires fabricated by electrodeposition into cylindrical pores of a porous media have proved to be interesting systems for fundamental studies of nanomagnetism and spin transport.[1] Using track-etched polycarbonate membranes as templates, it has been demonstrated that it is possible to grow cylindrical ferromagnetic nanowires with diameters down to 30 nm and extremely large aspect ratios (up to 1000).[2] Whereas measurements of several parallel nanowires embedded in a nanoporous membrane are relatively easy, studies of a single isolated nanowire constitute a real challenge. Moreover, the magnitude of the magnetization of such small magnets is of the order of 10^{-11} emu and thus measurement of single wires can be performed using only a few techniques. So far, micro-superconducting quantum interference device (SQUID),[3] magnetic force microscopy (MFM),[4,5] and magnetoresistance measurements[6–9] have been reported on single Ni and Co nanowires.

Up to now, connecting a single nanowire for magnetotransport measurements was made directly during the electrodeposition process.[6–9] To this end, metallic electrodes are evaporated on both sides of the membrane prior to electrodeposition. During the plating process, the emergence of the first nanowire from the membrane surface that comes into contact with the top electrode exposed to electrolytic solution is detected by a sharp increase in the deposition current. The plating process is then immediately interrupted to avoid the emergence of other nanowires. Using this procedure, electrical contacts are therefore established on the two extremities of a 6–20 μm long nanowire thereby allowing two-probe measurements.[6–9] Thanks to the extremely large aspect ratio of the nanowire, the contact resistance is usually small compared to the sample resistance.

In this work, we report a method that combines electrodeposition in a nanoporous medium and electron-beam lithography (EBL) to connect a single nanowire. Attempts were made on Co nanowires of various diameters ϕ in the range of 35–100 nm as well as on Co/Cu multilayered nanowires ($\phi = 80$ nm). The advantage of this technique is that multiprobe connections are established along the isolated wire enabling one to perform electrical measurements on different segments of the wire. From these measurements, we are able to point out the inhomogeneous character of magnetic anisotropy and the reversal process in a single 60 nm Co nanowire. The nanowires were grown by electrodeposition from a sulfate bath into 22 μm-thick nanoporous track-etched polycarbonate membranes. The pore sizes were 35 and 60 nm and the pore densities were close to $10^9/\mathrm{cm}^2$. While the electrodeposition process in such nanoporous media is now well established,[1,2] optimization of the dissolution of the membrane and cleaning of the wires before the lithography process are not easy tasks. The objective is to obtain clean nanowires spread over a silicon substrate which are sufficiently isolated from each other to allow multiprobe connection. Dichloromethane (CH_2Cl_2) was used to dissolve the polycarbonate membrane. The dissolution process was assisted by ultrasonic agitation. A few drops of this solution were then spilled onto the substrate. We observed that, after drying the solution, the wires are strongly stuck on the substrate surface probably due to van der Waals forces. This is of importance because it allows us to perform all the nanofabrication steps (resist spinning, lithography, liftoff in liquid solutions, etc.) with very high accuracy. A regular cleaning of the silicon substrate and wires is then performed by ion beam etching (IBE), in order to ensure low contact resistance to the nanowires. Auger Nanoprobe PHI 680 and time of flight-secondary ion mass spectrometry (TOF-SIMS) experiments were used to check if there was surface contamination of the wires and substrate and to optimize the cleaning process (see Fig. 1). The Auger nanoprobe allows one to locally probe a surface 20 nm in diameter with resolution of a few nanom-

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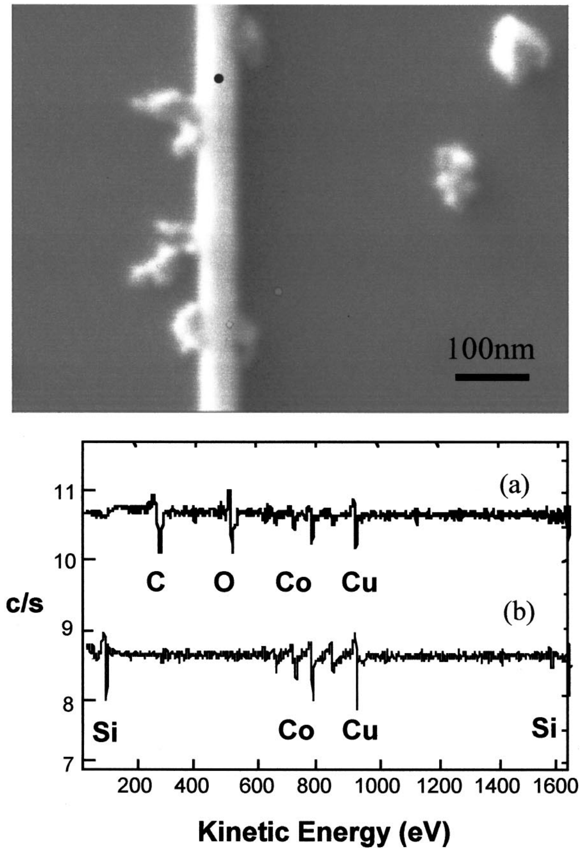

FIG. 1. Nano-Auger spectrum recorded on the surface of a Co/Cu multilayered nanowire. The black spot shows the area observed on the nanowires before (a) and after (b) etching with argon ions. After etching carbon and oxygen are removed from the surface of the wire.

eters in depth. Carbon and oxygen coming from the polycarbonate membrane were found at the surface of the wire. These were removed by *in situ* IBE without any damage to the nanowires. From the TOF-SIMS experiments, surface contamination by hydrocarbons species was also found. This can be easily removed using acetone and alcohol.

The lithography process is the second important step for multiprobe connection on an isolated nanowire. It is based on work by Naud *et al.*[10] on carbon nanowires. EBL was performed using a JEOL 5DIIU nanowriter. The first step of the process is the deposition of Ti/Au alignment marks on the Si substrate to pinpoint the wires. The nanowires are then spread over the sample in the manner described above. Optical microscopy and scanning electron microscopy (SEM) investigations are then used to select and locate isolated nanowires. In this way we can design the probe setup adapted to the geometry of a particular nanowire using homemade computer-assisted design (CAD) software. EBL is then performed after spin coating a 4500 Å thick layer of PMMA resist. After development, a 1000 Å thick metallic layer is evaporated and lifted off. Both Ti/Au or Al can be used as metallic pads. Using the results of the nano-Auger study, IBE is made *in situ* in the evaporator chamber just before metal deposition. The quite thick layer of PMMA ensures good liftoff of the metallic patterns even if the IBE step is done before evaporation. The micrograph in Fig. 2 shows the final multiprobe connection of a 35 nm Co nanowire.

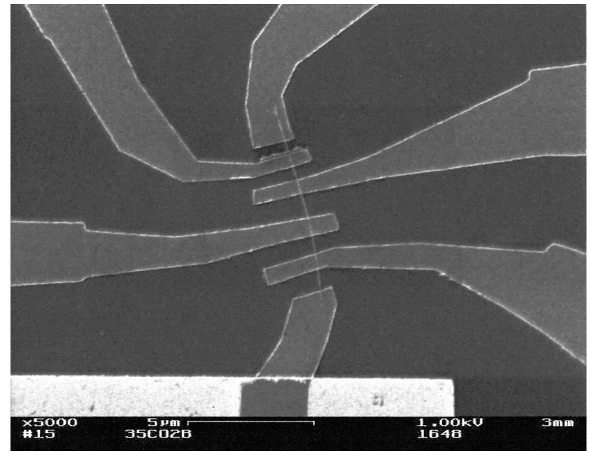

FIG. 2. SEM picture of a connected Co 35 nm diam wire. The typical pad width and pad separation are 0.5 μm.

Magnetoresistance measurements were performed using a PAR Keithley lock-in amplifier with a typical ac current of 10 μA. It is well known that for a transition metal ferromagnet the most important part of the magnetoresistance comes from the anisotropic magnetoresistance (AMR). In the saturated state, the AMR can be expressed as a function of the angle θ between the magnetization and the current[11] as follows:

$$R(H) = R_0 + \Delta R \cos^2 \theta. \quad (1)$$

Figure 3 shows the magnetoresistance recorded in two different parts of a 60 nm Co nanowire for both parallel and perpendicular directions of the applied magnetic field with

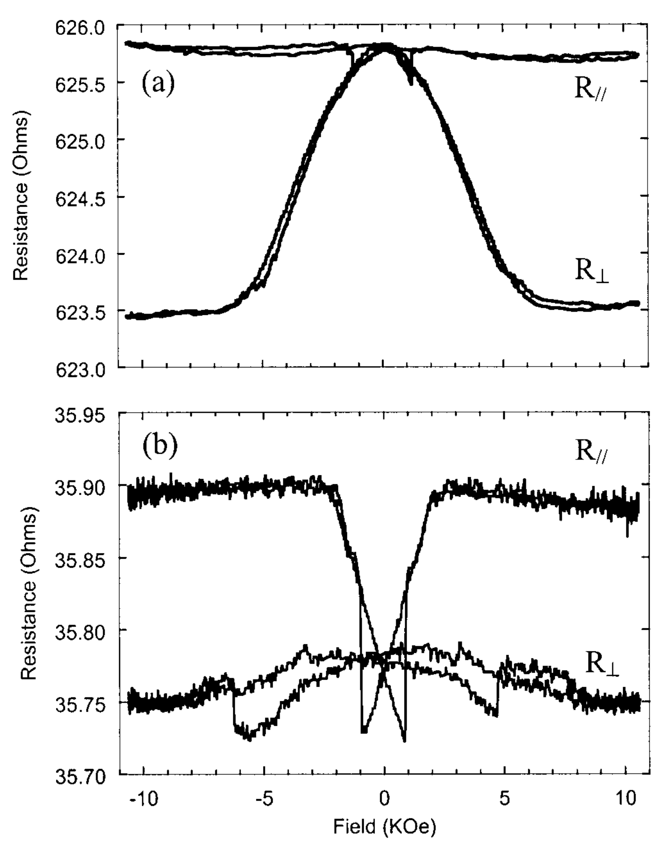

FIG. 3. Measurements of two segments of the same 60 nm cobalt nanowire. The lengths of the segments are 5 (a) and 0.5 μm (b).

respect to the wire axis. Quite different behavior is observed for the two different parts of the same unbroken wire. Whereas for the 5 μm long segment we observe large magnetoresistance in the perpendicular configuration [Fig. 3(a)], the magnetoresistance signal is much larger in the parallel configuration for the 0.5 μm long segment [Fig. 3(b)]. This suggests that the remanent state of magnetization, which does not depend on the direction of the field applied, is different in two parts of the same wire. For 5 μm contact separation, the almost vanishing magnetoresistance for the parallel direction indicates that the magnetization in the remanent state lies mostly along the axis of the wire. It should be noted that the perpendicular resistance value at zero field coincides with that obtained for the parallel direction. To the contrary, for the 0.5 μm long segment, the magnetoresistance behavior indicates that the magnetization at zero field is in a direction close to perpendicular to the axis of the wire.

In previous studies of these Co nanowires we saw evidence of the influence of strong, diameter dependent magnetocrystalline anisotropy. The total magnetic anisotropy can be expressed as

$$E_{\mathrm{an}}=(K_1+\pi M_s{}^2)\sin^2\theta, \qquad (2)$$

where K_1 is the magnetocrystalline anisotropy coefficient and the term $2M_s{}^2$ comes from the shape anisotropy. Both transmission electron microscopy (TEM) and x-ray diffraction (XRD) have demonstrated a change in the crystalline texture from c axis perpendicular to the wire axis ($K_1<0$) for diameters of 80–150 nm, to c axis mostly parallel to the wire axis ($K_1>0$) for diameters smaller than about 40 nm.[5,12] Previous MFM studies of 150 nm Co nanowires have shown a clear signature of a large transverse component of the magnetization.[4,5] The nanowire appears to be made of several magnetic segments that exhibit blockwise varying MFM contrasts. The length of the magnetic segments is of the order of 1 μm which is significantly smaller than the length of the grains.[12] In contrast, magnetization states parallel to the wire axis are favored for diameters smaller than 40 nm.[5] Measurements of anisotropic magnetoresistance performed both in the small diameter limit ($\phi\leqslant 40$ nm) and in the large diameter limit ($\phi\geqslant 80$ nm) corroborate these different behaviors.[13] In addition, using micromagnetic calculations, Ferre and co-workers[13] have shown the influence of magnetocrystalline anisotropy on the magnetic reversal mechanism. For a negative large magnetocrystalline anisotropy (K_1) they found a reversal mechanism with domain nucleation, whereas for a positive value of K_1 a reversal mode with vortex nucleation and propagation by winding up around it was predicted.

For the 60 nm Co nanowires considered in the present study, we expect mixing of the two orientations gives rise to local inhomogeneity of the magnetic anisotropy along the wire. The results obtained on the sample with 0.5 μm contact separation are thus consistent with a local texture (0001) preferentially oriented perpendicular towards the axis of the wire. In that case, the magnetocrystalline anisotropy due to the local perpendicular orientation of the c axis overcomes the shape anisotropy. Due to the small distance between contacts, we expect to measure a single crystal region. However, although the overall behavior of the magnetoresitance curves agrees qualitatively with this simple picture, anomalies in the curves, such as the small jump in resistance at 5 kOe when the field is perpendicular to the wire [Fig. 3(b)], reveal a more intricate process for the reversal mechanism. In contrast, for the 5 μm contact separation, the results are consistent with remanent magnetization states mostly along the axis of the wire.

In conclusion, multiprobe connections were made on isolated magnetic nanowires using electron-beam lithography, and magnetoresistance measurements were performed. The main attraction of our method comes from the possibility to probe transport properties on parts of single nanowires as small as 0.5 μm. The results show the inhomogeneous magnetic behavior of the reversal process along a single 60 nm Co nanowire. This can be ascribed to the intermediate diameter for the Co nanowire that allows local to magnetocrystalline anisotropy either to compete or to add to the shape anisotropy. Our technique can also be useful to investigate other interesting phenomena such as domain wall magnetoresistance and magnetization reversal by injection of spin-polarized current.[14,15] Another area of interest is the possibility of combining electrical measurements with near field or TEM studies since the wire is freed of the porous template.

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