Contacting individual Fe(110) dots in a single electron-beam lithography step

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Received 5 March 2009, in final form 7 May 2009
Published 23 June 2009
Online at stacks.iop.org/Nano/20/285302

Abstract

We report on a new approach, entirely based on an electron-beam lithography technique, to contact electrically, in a four-probe scheme, single nanostructures obtained by self-assembly. In our procedure, nanostructures of interest are located and contacted in the same fabrication step. This technique has been developed to study the field-induced reversal of an internal component of an asymmetric Bloch domain wall observed in elongated structures such as Fe(110) dots. We have focused on the control, using an external magnetic field, of the magnetization orientation within Néel caps that terminate the domain wall at both interfaces. Preliminary magneto-transport measurements are discussed demonstrating that single Fe(110) dots have been contacted.

1. Introduction

Contacting individual nanostructures to probe their intrinsic transport properties has been a major technological challenge within the last few years. Recently, nanowires have been widely studied for their quantum properties [1–3] and also as promising candidates for next-generation nanoelectronic transistors and devices [4, 5]. Most of these works have opted for a top-down approach mostly based on electron-beam (e-beam) lithography to contact electrically randomly distributed nanostructures. This route requires two technological steps that consist first in localizing the nanostructures to be electrically probed and then contacts are made to individual wires using a lift-off technique. The first step is clearly time-consuming as AFM or SEM pictures are needed. Another approach has been developed for nanowires embedded in a membrane and obtained by electrodeposition [6]. Membrane holes are partially plugged using a physically deposited top Au layer. The growth of nanowires is stopped abruptly when the first structure emerges from the membrane, leaving the other wires unconnected. The growth termination is detected by the sharp change in the deposition current. This simple low-cost technique is, however, restricted to a very specific system. Alternative routes have been developed using local probe microscopy techniques such as AFM [7] or STM [8]. The former makes it possible to indent accurately in depth and lateral position a photoresist layer. The indented void then serves as a mask to create the top contact. The latter avoids any technological process as it defines per se a tunnel junction between the tip and the probed nanostructure. These local probe techniques are nevertheless limited by their low throughput.

In this paper, we report on a new technique, entirely based on e-beam lithography, to carry out four-probe magneto-resistance measurements of self-assembled Fe(110) dots. We opted for a simple approach that can be applied as a general procedure to contact other self-assembled nanostructures randomly distributed over the sample surface. Our innovative approach makes it possible to circumvent the technical difficulties which consist in localizing precisely self-assembled structures and in contacting selected structures. In our method, we addressed these two crucial issues in a single e-beam lithography step. Preliminary magneto-resistance (MR) measurements of individual Fe(110) dots are then presented as proof of the reliability of our approach.

2. Fabrication process

Fe(110) dots are obtained by pulsed-laser deposition (PLD) under ultra-high vacuum conditions. These structures grow...
epitaxially on an Mo(110) buffer layer previously deposited on a sapphire(1120) substrate. Self-assembly results from the so-called Stranski–Krastanov growth mode, occurring for a deposition temperature in the range ∼600–850 K (figure 1(a)) [10]. For our study, the Fe dots have been deposited at ∼800 K on a 50 nm Mo(110) buffer layer and covered by a ∼0.7 nm Mo layer and a 5 nm Au layer to prevent oxidation.

The sample has been processed using a field emission gun scanning electron microscope (FEG-SEM) LEO 1530 remote-controlled by the Elphy plus system (Raith GmbH) for e-beam lithography (typical e-beam voltage of 20 kV). A schematic view of the pattern is presented in figure 1(b). The first step of our process is based on a routine procedure for the fabrication of the bottom electrodes in the Mo(110) buffer layer by Ar ion beam etching using a Shipley UVN2® chemically amplified negative resist as a mask. The 230 nm resist mask is thick enough to protect ∼140 nm high Fe(110) dots upon the etching process of the 50 nm Mo(110) buffer layer. The UVN2 layer is removed using EKC® LE® solution and a 55 nm SiO2 layer is then deposited on the sample surface using an RF magnetron sputtering technique. Note that the width (10 μm) of the constriction of the bottom electrodes has been determined considering dot surface density (controlled by Fe deposition temperature) to ensure that statistically 1–10 dots would be sitting on the 10 μm × 10 μm intersection area of the top and bottom electrodes. Among these few dots, a single nanostructure will be contacted later on.

The next step of the process consists in patterning the SiO2 layer on top of selected Fe(110) dots (φ = 100 and 200 nm holes) and on top of the bottom contact pads using a Microchem PMMA positive resist layer as a mask. The key aspect of our process has been to develop an exposure procedure to locate and contact individual Fe(110) dots in a single e-beam step. This approach is based on the imaging of the central area of the pattern covered with a PMMA resist layer. A single Fe(110) dot per pattern is then selected and exposed with a view to creating a hole in the resist layer in the very same process step. The technical difficulty of this step has been to optimize the quality of the image used to determine the structure to be connected without exposing the electron-sensitive PMMA resist in a too significant manner. The procedure has been optimized using a standard 180 nm PMMA 3% layer spin-coated on a so-called test sample exhibiting ∼120 nm Fe(110) dots (figure 2). In our process, two images are required to shift e-beam writefields with sufficient accuracy (<150 nm) on top of the selected structure. The sample surface is imaged under the same e-beam voltage conditions as that used for exposure steps (20 kV). The key parameters of the imaging conditions are the size (in μm) and the resolution (in px) of each image. These parameters determine, knowing the beam current and the dwell-time per pixel, an equivalent exposure dose (EED). SEM imaging of Fe(110) dots is possible because the PMMA surface is not perfectly flat on top of Fe(110) dots. A 10 nm Al layer deposited on top of the resist layer is also used to allow imaging of the surface of the sample covered with an insulating PMMA layer. Typically, 30–40 nm high prominences have been observed using AFM in contact mode. The thickness of the PMMA layer on top of Fe(110) dots is thus ∼80 nm.

The effect of the SEM image acquisition step on the final PMMA resist thickness and the accuracy of the e-beam writefields’ shifts have been quantified by contact AFM images (figures 2(b) and (c)). It turns out that the first 15 μm × 15 μm SEM picture (256 px × 256 px), labelled 1 in figure 2(b), induces a loss of only 3–4 nm of the resist layer while in the overlapping area with the second SEM image, labelled 2 in figure 2(b), ∼30 nm of PMMA have been removed. The remaining thickness of the PMMA layer on top of each exposed Fe(110) dot is thus 50 nm, which turned out to be sufficient to protect the rest of the exposed Fe(110) dot as detailed below. The EED of each image is ∼27 μC cm⁻² so that the total EED of ∼54 μC cm⁻² remains significantly below typical exposure dose values of 250–300 μC cm⁻² used for PMMA resist layers. Figure 2(c) highlights the fact that an exposure position accuracy better than 150 nm is obtained. From this image, the thickness of the resist layer on top of an Fe(110) dot can be estimated to be ∼70 nm.

This thickness is, however, underestimated owing to the finite AFM tip radius (typically 10 nm) and the full tip cone angle (typically 40°). This is consistent with the estimate of a resist layer thickness of 80 nm, meaning that the resist has been completely exposed on top of the selected Fe(110) dots. It also turns out that the FWHM of the exposed area is of the order of 205 nm, which is in good agreement with a nominal diameter of 200 nm.

For the final sample exhibiting Fe(110) dots of typical height ∼140 nm, we have used a thicker resist layer (PMMA 4%, 240 nm). Although the PMMA 4% layer has been observed to be more sensitive to e-beam exposure in the imaging procedure, similar protection conditions of the
resist layers we have used exposure doses of 450–600 μC cm⁻² to ensure that the resist layer atop Fe dots would be exposed.

Once developed, the PMMA resist layer serves as a mask for a reactive ion etching of the SiO₂ layer using a CHF₃ RF plasma (P_{CHF₃} = 2 × 10⁻² Torr, P_{RF} = 50 W). The etching has been monitored using laser reflectometry performed on a Si/SiO₂ reference layer deposited together with the covering layer of the Fe(110) dots. For the test sample exhibiting ∼120 nm Fe(110) dots, the etching process has been intentionally extended. Figure 3(a) clearly demonstrates that only the area corresponding to the exposure pattern is affected by the etching process, leaving the area imaged using SEM unaffected.

The last step of the process is a standard lift-off process of a Ti/Au (10 nm/100 nm) layer using an automatic realignment procedure and a PMMA positive resist (figure 3(b)). The structured Ti/Au layer finally defines the top electrodes and connects a single Fe(110) dot per pattern and the bottom electrodes.

Fe(110) dots have been contacted to observe in real time the reversal of an internal component of a magnetic domain wall and demonstrate experimentally its associated hysteresis. Indeed, we have recently demonstrated that the magnetization orientation within the two Néel caps (NCs) that terminate an asymmetric Bloch wall at both interfaces in elongated structures (in our case self-assembled Fe(110) dots) can be controlled using an external magnetic field [9, 11]. The signal used to probe the NC reversal is the anisotropic magnetoresistance (AMR) which yields to two different resistance states for a transverse (i.e. along the width of the Fe(110) dots) current component.

Magneto-transport measurements have been carried out in a variable temperature (2.2–300 K) pumped He cryostat. An in-plane magnetic field up to 7 T is provided by a superconducting split-coil magnet. The sample holder can be rotated continuously in the plane of the sample. In our measurements, we have used two applied field configurations with respect to Fe(110) dots’ orientation: along the [001] (called longitudinal hereafter) direction and along the [110] (called transverse hereafter) direction. Typical field scans in the range ±1.2 T have been recorded in 10 min. Low-noise measurements are required as, according to our RT micromagnetic simulations [11], the transverse magnetization averaged over the 3D structure is expected to decrease by 4% upon NC reversal. Furthermore in [12], the authors determine the RT AMR signal for an epitaxial Fe(110) layer with a current applied along the [110] direction (i.e., an equivalent direction to our transverse configuration) of the order of 0.3%. This yields an RT-MR signal of the order of 10⁻⁻⁻ which, in the case of an impedance of ∼1 Ω, is a severe experimental difficulty. Our low-noise/low-impedance set-up is based on a Stanford Research Systems SR830 lock-in device and a high-impedance polarization charge (1 kΩ) as a current source (figure 4). The four-probe sample voltage is then detected at the lock-in frequency (1013 Hz). In our set-up, peak-to-peak relative noise amplitude has been reduced down to ∼7 × 10⁻⁵ at 1013 Hz which is close to the lock-in limit measured at ∼5 × 10⁻⁵ in this frequency range.

The zero-field resistance of a pattern is plotted in figure 5 as a function of sample temperature. It clearly exhibits a
of \( \sim \) is expected in the Au layer yielding a total extra resistance of the same order of magnitude. A lead resistance of the same order of magnitude arises from the fact that current flows into low-impedance current-crowding loops of an assembly of Fe(110) dots [14]. The two maxima observed at \( \sim \pm 0.12 \) T are thus likely to be related to the NC's reversal, in qualitative agreement with micromagnetic simulations [11]. From figures 5(d) and (e), no clear evidence of the existence of a hysteresis associated with NC reversal can be drawn. An improvement would be to reduce the current crowding effect by etching trenches in electrodes around the selected nanostructure as suggested in [15]. Superconducting electrodes also constitute a promising alternative route as lead resistances would be suppressed. In this perspective, Nb appears as a high potential candidate as Nb(110) buffer layers have been already obtained in our deposition chamber.

4. Conclusion

In conclusion, we have developed a new approach to contact self-assembled nanostructures, by nature randomly distributed on a surface. This procedure makes it possible to locate and expose individual structures in a single e-beam lithography step. It can be used as a general route to connect structures randomly distributed over the sample surface. This technique...
has been developed to study the field-induced reversal of an internal degree of freedom of an asymmetric Bloch wall (i.e. the orientation of NC’s) in individual Fe(110) dots. Preliminary MR results obtained confirm that individual Fe(110) dots have been connected and that magnetic processes can be monitored through magneto-transport measurements based on the contacting process.

Acknowledgments

We are grateful to P David and V Santonacci (sample elaboration), T Crozes (nanofabrication), D Dufeu and D Maillard (magneto-transport), and D Lucot for technical support and/or fruitful discussions.

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