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Magnetic force microscopy of magnetic domain structure in highly ordered Co nanowire arrays

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Abstract

Highly ordered anodic alumina oxide (AAO) membranes with a diameter of about 50 nm are prepared by two-step anodization. Cobalt nanowires were obtained by ac electro-deposition of Co^{2+} into the pore of AAO membranes. Vibrating sample magnetometer (VSM) results show that such a sample has highly perpendicular anisotropy properties. Here, we first use magnetic force microscopy (MFM) to obtain a fine image of magnetic domain structure in Co/AAO film successfully. MFM images show that each as-prepared nanowire in the membrane show single domain structure. The nearest neighbor bits have opposite magnetization. The magnetic moment was aligned in the same direction after the sample was magnetized under a 1 T external field. Due to its high bit density (up to 100 $Gbits/in.^2$), such a material may be used as high-density perpendicular recording quantum magnetic disks (QMD). © 2001 Elsevier Science B.V. All rights reserved.

1. Introduction

Recently, preparing large area nanometer scale arrays of ferromagnetic nanowire has been the subject of intensive research [1–3]. Due to its large aspect ratio, the uniaxial anisotropy of such a material was very high, which gave such material a high perpendicular magnetic anisotropy. This is very important in providing definite magnetic states in perpendicular recording. Many research groups suggested that arrays of small magnetic islands can overcome super-paramagnetic limit and side track limit which exist in conventional longitudinal recording media and might be used as a high-density and low-noise magnetic recording medium [4]. Such a material was also called quantized magnetic disks (QMD) [5]. QMDs have discrete, single-domain magnetic elements uniformly embedded in a non-magnetic disk. Many investigations [6–8] have been undertaken on QMD fabricated by using electron beam lithography, nanoimprint lithography and electro-deposition methods. In such a case, a magnetic material such as cobalt or nickel was embedded in a non-magnetic material and each pillar could be defined as a bit. Due to its nanoscale size, each bit has only two stable states: equal in magnitude but opposite in direction. Each magnetization direction of a single-domain element can represent a bit of binary information, which can be used for high-

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density perpendicular recording. White et al. [9] had stated that QMDs having as many as 400 $Gbits/in.^2$ could be made in the case of 10 nm diameter dots with 40 nm pitch fabricated using nanoimprint lithography and lift-off.

Although there are currently substantial research efforts on magnetic nanowires, there is a paucity of investigation of magnetic domain structure in a highly ordered Co/AAO film. Unlike track etched membranes of polycarbonate or other membranes, the surface of AAO membranes is rather rough which make imaging of magnetic domain structure more difficult. In this Letter, we will report the preparation of highly ordered AAO membranes and Co nanowire arrays. A new method to polish Co/AAO film is also presented. Additionally, we demonstrate that each nanowire has a single domain structure and the nearest neighbor bits have opposite magnetization directions when the sample is demagnetized. The magnetic moment of nanowire was switched to the same direction when the sample was magnetized under a 1 T external field.

2. Experimental

2.1. Preparation of highly ordered AAO membranes

Ordered AAO membranes were prepared by two-step anodization [10]. An Al sheet ($0.1 \times 15 \times 10$ mm^3) with a purity of 99.9% was first pretreated by degreasing, electro-polishing and rinsing with distilled water, and then it was anodized at 15–50 V in 0.3 M $H_2C_2O_3$ for 10 h at room temperature. Then the oxide film was dissolved in 0.2 M H_2CrO_4, 0.4 M H_3PO_4 at 60 °C. The second anodization was carried out at the same condition as the first one, and the anodized time was 30 min.

2.2. ac electro-deposition of cobalt

The arrays of cobalt nanowires were fabricated by ac electro-deposition of an electrolyte containing the Co^{2+} ion using graphite sheets as counterelectrodes. The solution used in this experiment had the following composition: $CoSO_4 \cdot 7H_2O$ 10 g/l; H_3BO_4 40 g/l; ascorbic acid 15 g/l. The pH value of the electrolyte was maintained at 3.5–4.0, and then the electrolysis was conducted at 20 °C, 200 Hz, 11 V/ac and using graphite counterelectrodes. The time used in electro-deposition was 2 min.

2.3. Chemical polishing of Co/AAO film for observation of MFM image

In order to obtain a very fine magnetic force microscopy image (MFM) of Co/AAO, the surface of the sample should be very smooth in order to eliminate the topographical information from the magnetic image. After the sample was prepared, it was fixed into a graphite counterelectrode and dipped into the following solution: 0.1 M H_2CrO_4, 0.6 M H_3PO_4 at 25 °C to chemical polish. Then we take out the sample after 10–20 min and wash with distilled water several times to eliminate impurities.

2.4. Experiment instrument

The microstructure and magnetism of the samples used in this work are characterized by transmission electron microscopy (TEM, Hitachi 600), vibrating sample magnetometer (Japan, VSM-5s-15) and scanning probing microscopy (SPM, P47-SPM-MTD, made in Russia), respectively.

3. Results and discussion

An AFM micrograph of the surface view porous aluminum in oxalic acid solution at 30 V for 30 min is shown in Fig. 1a. The pore diameter is estimated to be about 50 nm and pore density is about 10^{10}–10^{11} cm^{-2}. The diameter and the interval of the pores are based on the applied voltage. Adjusting the applied voltage and using different electrolytes can prepare AAO membranes with different diameters and lengths. We can confirm that the holes were ideally arranged hexagonally within domains of microsize, which were separated from neighboring aluminum oxide domains with a different orientation of the pore lattice by grain boundaries. Thus, a polycrystalline pore structure is observed. Shown in Fig. 1b is the bottom view of porous aluminum after the first

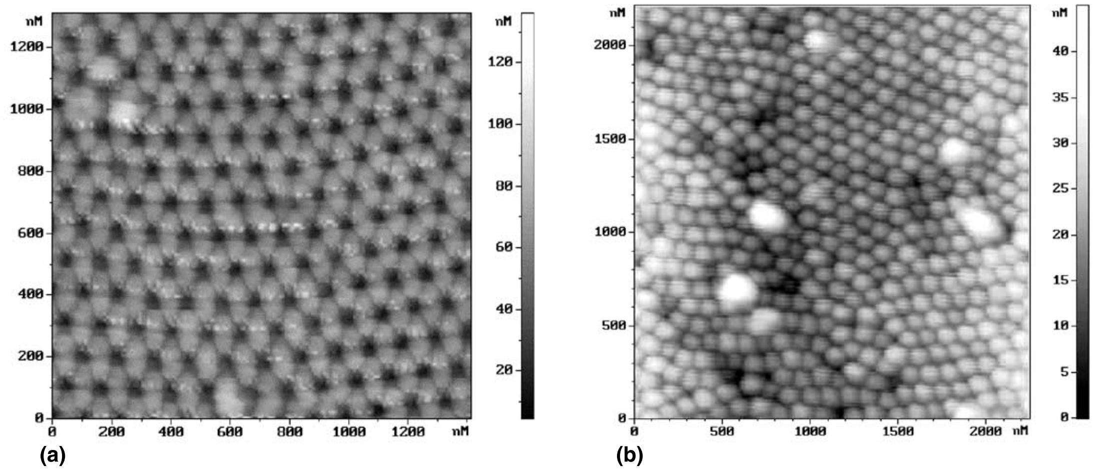


Fig. 1. (a) AFM image of AAO membranes fabricated using two-step anodized process, (b) a bottom view of AAO membranes after it was anodized the first time and taken off from the Al sheet.

anodizing process. It is clear that every aluminum oxide particle shows a hexagonal structure, which pre-textures the pore into the pattern structure after the second anodizing process.

Fig. 2 shows a representative TEM micrograph of the Co nanowires. Clearly, the Co nanowires are both very regular and uniform, with an average diameter of about 40 nm and length ca. 1.6 μm (aspect ratio of over 40). Studies on the detailed structure of the wires by electron diffraction patterns also show that each nanowire is made up of many microcrystals, which may result from the deposition of Co in a semi-circle during the ac electro-deposition. Due to its very large aspect ratio, it is reasonable to consider each Co dot in our nanowires as a single magnetic domain.

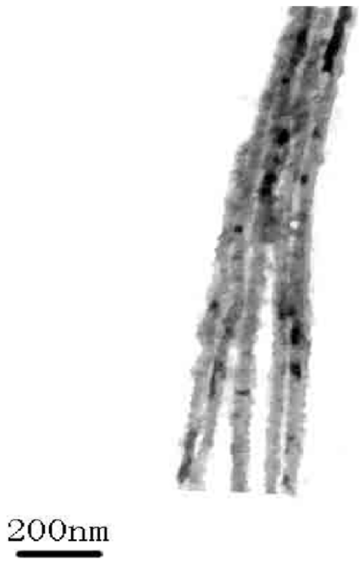


Fig. 2. TEM image of 40 nm diameter and 1.6 μm length Co nanowire arrays.

Fig. 3 shows the magnetization *loop* curves for the Co/alumina at room temperature (the applied

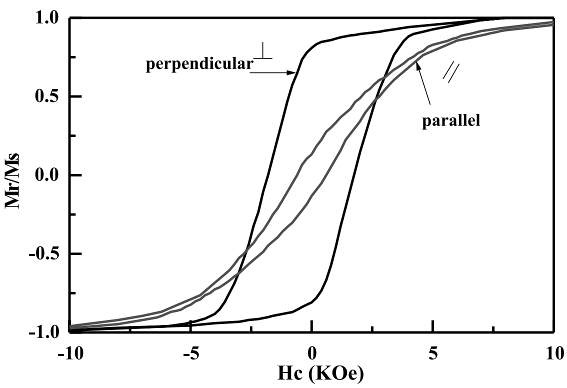
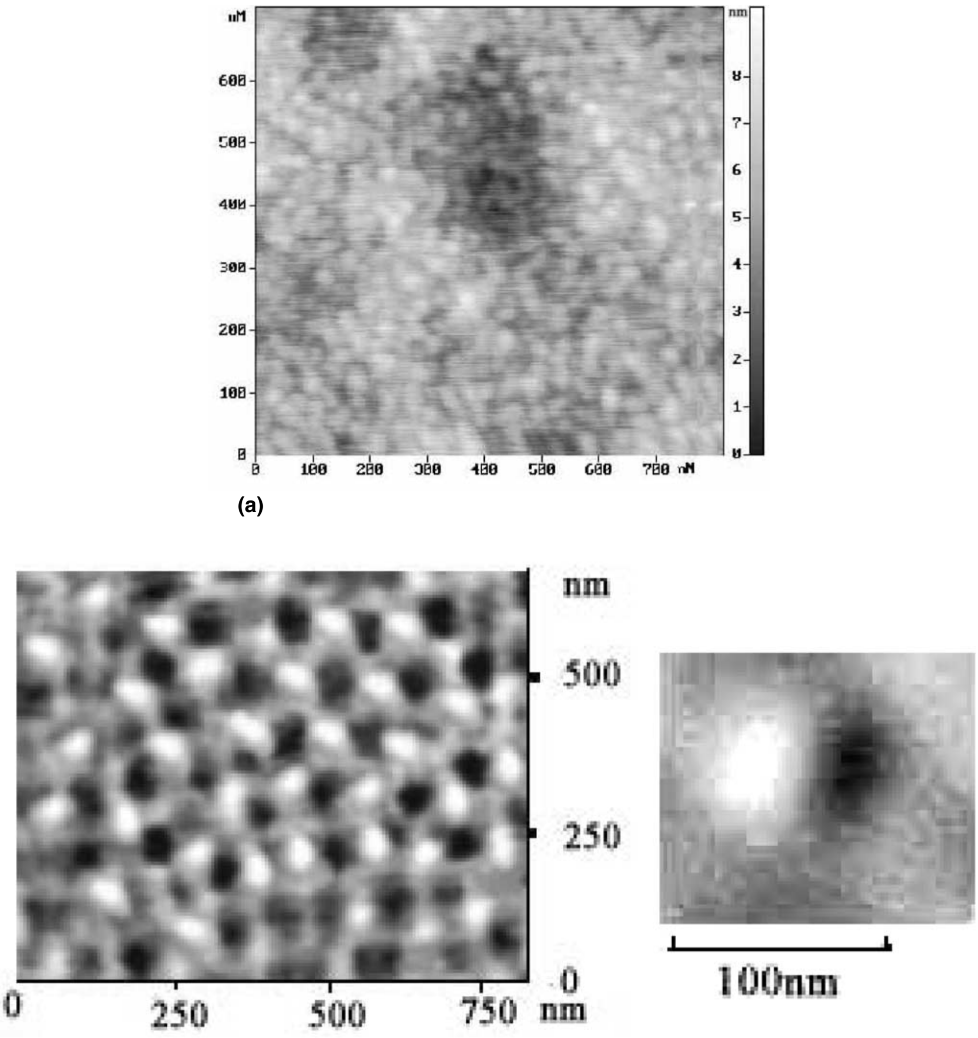


Fig. 3. Hysteresis loops of Co nanowires in AAO membranes at room temperature (the applied field was 16 kOe) ∥ with the magnetic field parallel to the wire; ⊥ with the magnetic field perpendicular to the wire.

field was 16 kOe). The squareness (Mr/Ms) is as high as 0.92 and the coercivity of array is 2000 Oe when the applied field is parallel to the axis of nanowire arrays, which suggests that the Co/AAO has superior perpendicular magnetic characteristics. The magnetic moment of Co dot in the nanowire was then deduced to be oriented along the nanowire axis, which is consistent with the result from other workers [11]. Because the shape anisotropy of our Co nanowires (aspect of 40) is much higher than crystal anisotropy, we then can conclude that the shape anisotropy and interactions dominate the magnetic behavior of our arrays. The easy magnetization direction is along the long axis of the wire and the hard magnetization direction is along the short axis.

To obtain the MFM image of Co nanowire arrays, we examined them using MFM. The magnetic force images were taken using a lift mode process. The imaging was done with a two-step lift mode process, with the first scan being used to determine the topography, and the second scan



(b) (c)

Fig. 4. (a) TMAFM image of Co/AAO film, (b) MFM image of Co/AAO membranes, (c) MFM image of two neighbor bits. The black region represents the attractive force between the tip and the sample and the white region represents the repulsive force.

passing at a fixed height above the known surface and recording the magnetic response of the tip, which eliminates the topographical information from the magnetic image.

TMAFM and MFM images taken simultaneously on the same area of Co arrays are shown in Figs. 4a and b, respectively. The TMAFM image of a 800×700 nm^2 section of the Co arrays shows that the topology of the Co pillars is indistinguishable from that of AAO membranes. The roughness of the surface is 9 nm. The corresponding MFM image, on the other hand, clearly shows that each bit has a quantized magnetization and the magnetic image of each pillar of this section can be resolved. We suppose that the wire's shape anisotropy is the primary cause of forming single domain in the Co nanowires. The number of south poles (black) is almost the same as that of north pole (white) in the section researched. Every single domain is surrounded by several single domains with opposite magnetized direction. Fig. 4c is a two neighbor bit with opposite magnetized direction: one is the south pole (black) and the other is north pole (white), the diameter of which is about 60 nm. The corresponded recording density is up to 100 Gbits/in.^{2} fabricated using AAO membranes. Due to the defects existing in the sample, some of the nearest neighbors show the same magnetized direction. In the case of nanowire arrays, the major features of the film are attributed to a new interplay between the magnetostatic energy and exchange energy. In order to reduce the exchange energy, it favors the alignment of all magnetic domains in the same direction, forming a single domain. However, to reduce the magnetostatic energy, it favors breaking the material into multiple domains to cancel out magnetic poles. In order to minimize the total energy of Co arrays, the nearest neighbor bits should have opposite magnetization energy, and our experiment results conform to this very well.

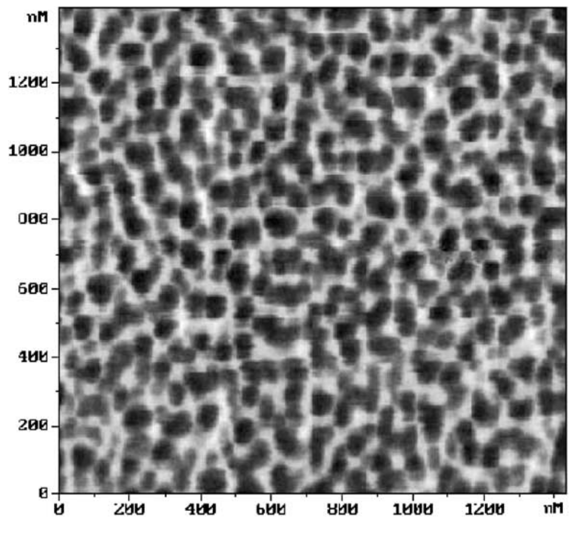


Fig. 5. MFM image of Co/AAO film after the sample was magnetized at 1 T external field.

As we know, the maximum magnetostatic energy is proportional to the wire length. When the wire length becomes so long that the maximum magnetostatic energy becomes larger than the exchange energy, the coherent switching can no longer be maintained and the vortex state may appear, leading to incoherent switching, which needs high external field to switch the magnetic moment of Co nanowire. As we know, the field of MFM tip is not larger than 50 Oe, much lower than the switching field of Co nanowire (2000 Oe). So we cannot use MFM to write information to such a sample. Fig. 5 shows the MFM image of Co nanowire after the sample was magnetized at a 1 T external field. Clearly, only black dots can be seen, which means that almost all the magnetic moments of the nanowires switch to the same direction. If high enough external field were applied, information can be written into a Co/AAO film.

4. Conclusion

We have prepared highly ordered and high-density AAO membranes. Co nanowires were electrodeposited into the pore of the AAO template. MFM observation shows that all the as-fabricated Co nanowires are single domain. After the sample was magnetized in a 1 T external field, almost all the moments were reverse to the same direction, which means that we can read/write information in such a sample. The corresponding recording density is up to 100 Gbits/in.^{2} if a single domain can represent a binary bit, which is very attractive in perpendicular recording.

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