First-Order-Reversal-Curve Analysis of Nanoscale Magnetic Materials

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ABSTRACT

Magnetic nanowires, nanodots and nanoparticles are an important class of nanostructured magnetic materials. At least one of the dimensions of these structures is in the nanometer (nm) range and thus, new phenomena arise in these materials due to size confinement. These structures are ideal candidates for important technological applications in spintronics, high density recording media, microwave electronics, permanent magnets, and for medical diagnostics and targeted drug delivery applications. In addition to these technological applications, these materials represent an experimental playground for fundamental studies of magnetic interactions and magnetization mechanisms at the nanoscale level. When investigating the magnetic interactions in these materials, one of the most interesting configurations is a periodic array of magnetic nanowires, because both the size of the wires and their arrangement with respect to one another can be controlled. Inter-wire coupling is one of the most important effects in nanowire arrays because it significantly affects magnetization switching, and microwave and magneto-transport properties. The magnetic characterization of materials is usually made by measuring a hysteresis loop, however it is not possible to obtain information of interactions or coercivity distributions from the hysteresis loop alone. First-order-reversal-curves (FORC) provide a means for determining the relative proportions of reversible and irreversible components of the magnetization in arrays of magnetic nanowires [1]. In this paper we will discuss the synthesis of magnetic nanowire arrays, the FORC measurement and analysis technique and present results for an array of nickel (Ni) nanowires.

Keywords: first order reversal curve, FORC, magnetic nanowire

1 SYNTHESIS OF MAGNETIC NANOWIRE ARRAYS

The strength of magnetic interactions in magnetic nanowire arrays can be controlled effectively by varying the interwire spacing. The spacing provided by the polymer membranes is difficult to control due to the randomness of pore location obtained through the combination of the charged-particle bombardment (irradiation) and chemical etching [1]. Despite recent progress in fabrication of ion track nanochannels that allows a better control of the number of the pores in a membrane, their precise location is still problematic. Templates obtained by aluminum anodization provide for effective control of pore structures through a combination of voltage, acid concentration, oxidation reaction time, and post-anodization processing [2]. These methods can produce membranes with highly symmetric pores structures with a range of sizes. Fig. 1 shows electron microscopy images of a series of alumina membranes prepared with different pores diameters.

Nanowires grown in such membranes have differing magnetic behavior. This can be easily observed from their hysteresis loops (Fig. 2), where the field applied parallel and perpendicular to the 1000 nm nanowire axis show distinct variations in curve shape. As the nanowire diameters increase, for the same distance between their centers, the magnetic interactions increase due to the increased proximity between the wires. As a result, the hysteresis loops measured with the applied field parallel to the nanowires axis becomes more sheared with a reduced squareness (i.e., ratio between remanent and saturation magnetizations).
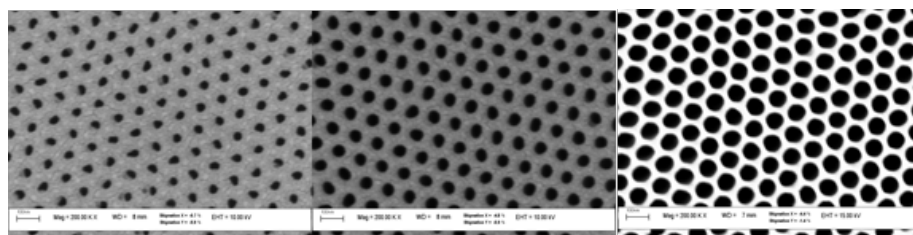
Fig.1 Scanning Electron Microscopy images of the top surfaces of AAO templates with pore diameters of (a) 40, (b) 60, and (c) 80 nm, and interpore distances of 100 nm

(a) (b) (c)
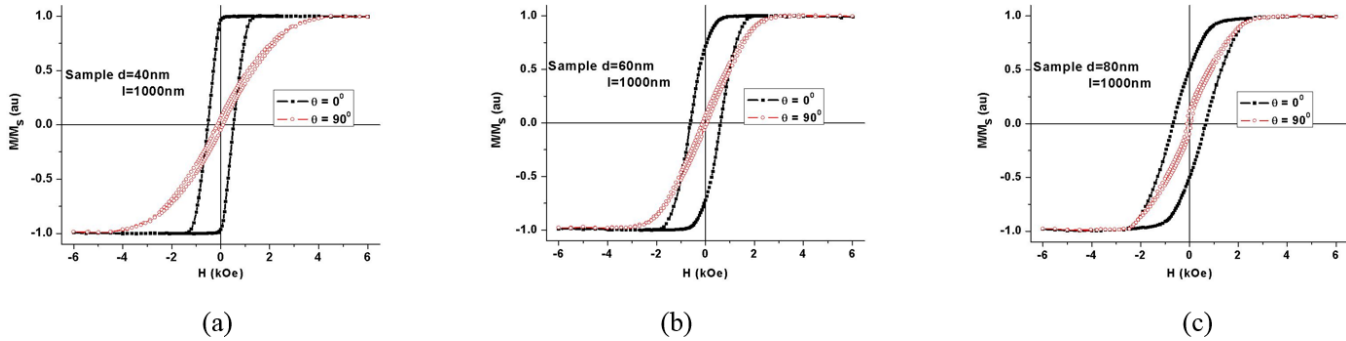
Fig.2 Hysteresis loops measured with the applied field parallel (black) and perpendicular (red) to the nanowires. The (a) 40, (b) 60, and (c) 80 nm nanowires were grown in the membranes shown in Fig. 1, respectively, with a length of 1000 nm.

Even with standard alumina membrane growth, the average distance between the pores cannot be varied over a large range. An alternative to this problem is to use alumina membranes in which the wires are allowed to grow only in some channels, by selectively obstructing others. One way to affect this pore blockage in alumina membranes is to use photolithographic techniques [3]. This allows one to obtain complex nanowire arrays where extensive patterns can be obtained over a large area. An alternate method to control interwire spacing is through a multistep aluminum anodization scheme that utilizes standard techniques (mild anodization) and those involving high voltage (hard anodization) [4]. In this approach one can obtain alumina membranes with more complex configurations that can be used to prepare nanowires with varying interwire distances and/or modulated diameters. An example of a membrane prepared by multistep anodization is shown in Fig. 3(a). In the transition from the mild region (lower right) to the hard region (upper left), approximately half of the channels remain continuous while others terminate at the mild-hard interface. Wires grown in the hard side of these membranes are 110 nm in diameter and are readily obtained within all available pores. Wires fabricated in the mild side of the AAO template, however, only grow in selected pores. The different wire configurations that can be fabricated using this approach are presented in Fig. 3(b).

2 FORC CURVES AND FORC DIAGRAMS

Any experimental method used to quantify the interactions in magnetic systems must be simple to implement. This can be achieved by starting from a reproducible and easy to achieve initial state, by requiring a reasonable number of measurement points, and by providing, directly or after some preferably simple data processing, a meaningful parameter characterizing the strength of interactions. For ferromagnetic materials the major hysteresis loop (MHL) is the distinctive fingerprint and the simplest measurement protocol, obtained by cycling the applied magnetic field and recording the ensuing change of magnetization of the specimen along the field direction. Unfortunately, through its main parameters of coercive field, remanent and saturation magnetizations, MHL cannot provide an adequate description of magnetic interactions. In spite of some attempts to consider more subtle features of the MHL as its shape (squareness, shear, etc.) [5, 6], it has proved not to be a suitable alternative to quantitatively characterize magnetic interactions, a situation stemming from exactly MHL's main advantage, simplicity. This can be easily observed from the loops presented in Fig. 2 where only general qualitative differences between samples can be discerned.
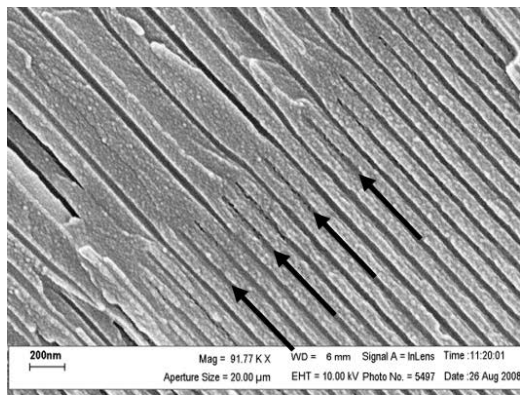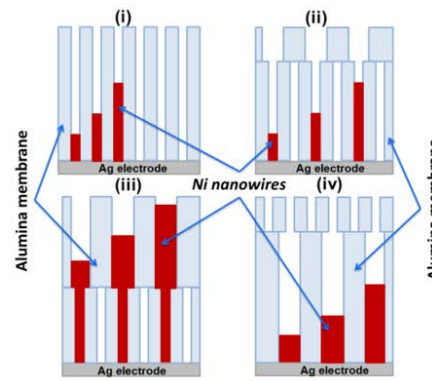
(a) (b)

Fig. 3 (a) FESEM of modulated membrane—arrows indicate closed pores (b) Schematic representation of the four series of samples considered (i) normal membrane (Mi) (ii) modulated membrane with increased interwire distance (Mi-Ha), (iii) modulated wires (SM Mi-Ha) and (iv) larger diameters wires (Ha-Mi).

More complex magnetization curves covering states with field and magnetization values located inside the MHL, as first order reversal curves (FORC) [7, 8], can give additional information that can be used for magnetic interaction characterization. A FORC is measured by saturating a sample in a field H_{sat}, decreasing the field to a reversal field H_a, then sweeping the field back to H_{sat} in a series of regular field steps H_b. This process is repeated for many values of H_a yielding a series of FORCs. The measured magnetization at each step as a function of H_a and H_b gives $M(H_a, H_b)$, which is then plotted as a function of H_a and H_b in field space. The FORC distribution $\rho(H_a, H_b)$ is the mixed second derivative, i.e., $\rho(H_a, H_b) = -\partial^2 M(H_a, H_b)/\partial H_a \partial H_b$, and a FORC diagram is a contour plot of $\rho(H_a, H_b)$ with the axis rotated by changing coordinates from (H_a, H_b) to $H_c = (H_b - H_a)/2$ and $H_u = (H_b + H_a)/2$, where H_u corresponds to the distribution of interaction fields, and H_c the distribution of switching fields.

The most commonly employed techniques for measuring FORCs are vibrating sample magnetometry (VSM) and alternating gradient magnetometry (AGM). Since the second derivative, $-\partial^2 M(H_a, H_b)/\partial H_a \partial H_b$, significantly amplifies measurement noise present in the magnetization data, the sensitivity of the measurement technique is important for magnetically weak samples, such as magnetic nanowire arrays. And, a typical sequence of FORCs may contain thousands of data points which can be unwieldy and cumbersome if the measurement is inherently slow; therefore, measurement speed is very important.

3 FORC MEASUREMENT RESULTS

Fig. 4 shows a series of FORCs measured using an AGM [9] for a periodic array of Ni nanowires with a mean diameter of 70 nm and an inter-pore distance of 250 nm. The FORC curves consist of 4640 points, and the data was recorded in 20 minutes. Analysis [10] of these FORC curves yields the local interaction H_u and coercive H_c field distributions shown in Fig. 5. This measurement protocol and analysis provide additional information regarding irreversible magnetic interactions or processes in this array of nanoscale wires, which cannot be obtained from the standard hysteresis loop measurement.
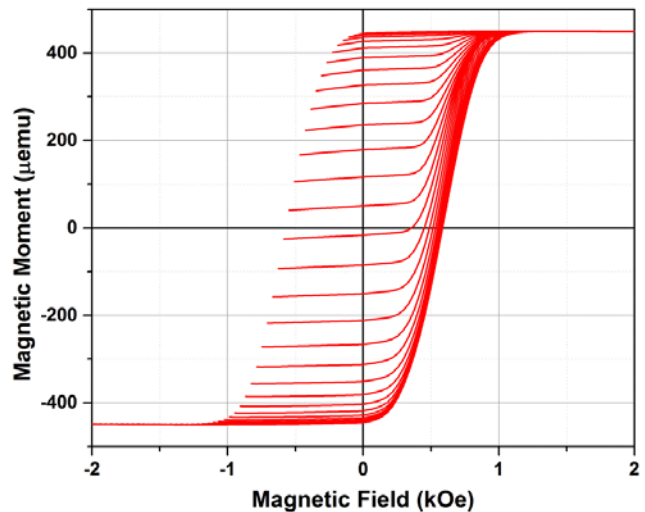

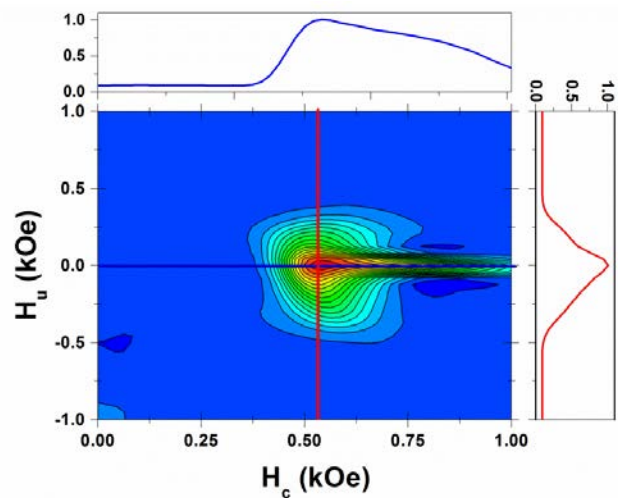**Fig. 4** First order reversal curves (FORCs) for an array of magnetic nanowires.

Fig. 5 Distribution of interaction fields as determined from FORC analysis

4 CONCLUSION

FORCs are indispensable in characterizing interactions and coercivity distributions that reveal insight into the relative proportions of reversible and irreversible components of the magnetization in magnetic nanowire arrays. In this paper we have discussed the FORC measurement technique and subsequent analysis which leads to the FORC diagram, and presented measurement results for a sample consisting of an array of Ni nanowires.

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