Magnetic State of Template Released Isolated Nickel Nanowires

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Abstract—Surface magnetic state of a range of relatively thick isolated template released Ni nanowires (NW) has been thoroughly investigated. These investigations were employed using magneto-optical Kerr effect magnetometry. The angular dependence of the switching field was accomplished by applying the magnetic field at different angles with respect to the NW long axis. All the loops showed a high squareness ratio when the magnetic field was applied parallel to the NW long axis, indicating the dominance of shape anisotropy. This result is quite different from the measurements of high-density arrays of templated NW reported in literature due to the small number of nearest neighbors and hence a reduction in the magnetostatic interaction among these NW. Square loops were noticed, at higher angles of measurements, indicating that the easy axis of magnetization might rotate with the field applied or the magnetometry detecting both transverse and longitudinal components of magnetization. The magnetization reversal of such NW is well described by the non-uniform rotation of the curling model of domain reversal. Finally, the results of this article demonstrate a difference between the surface and the bulk magnetic states which required more examinations using micromagnetic simulations or/and magneto-transport measurements on such thick NW.

Index Terms—Curling model, Magnetization reversal, Magnetooptical Kerr effect magnetometry, Magnetostatic interactions, Switching field.

I. INTRODUCTION

The researches on the magnetic state in extended twodimensional (2D) arrays of ferromagnetic nanowire (NW) synthesized in several templates by electro-deposition technique have been the subject of increasing interest

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Corresponding author's e-mail: mussab_ahmmad@yahoo.com Copyright © 2018 Musaab Salman Sultan. This is an open access article distributed under the Creative Commons Attribution License. over the past few decades, due to their relative large magnetization and the opportunity of using a wide range of investigative techniques [1-6]. These studies were performed due to their potential industrial applications and for exploring the fundamental science. As an example, they have been proposed for several magnetic technology, including; spintronics, magneto-optical devices, sensors, logic circuits, and ultra-high-density magnetic storage media [1-10].

In these researches [5-7], the magnetostatic or dipolar interactions among the NW were found to dominate over the intrinsic magnetic state of single NW. Furthermore, the disparity of diameters, morphologies, orientations, circularity and the separation among the nearestneighbor NW may complicate the situation regarding the intrinsic magnetic state of the NW, as extensively discussed elsewhere [8,10]. Thus, the magnetic state and magnetization reversal behavior in such isolated NW is a significant and demanding problem.

Many research groups were trying to investigate the magnetic properties of such NW by releasing them from their templates in a dilute suspension and characterize them using micro SQIUD [11,12] or using further steps and different techniques to electrically connect these NW with the external circuitry and use magnetoresistance setup to discuss their magneto-transport behavior [13-16]. Recently, the Object Oriented Micromagnetic Framework packagehas been employed to investigate the magnetic properties of rectangular NW [17,18]. Most of these studies have been NW of the smallest dimensions that are likely to display single domain states and simpler magnetic behavior compared to larger dimension NW that can support more complex domain structure.

Magneto-optical Kerr effect (MOKE) magnetometry, on the other hand, has been widely used to discuss the magnetization behavior of ferromagnetic thin films, arrays of nanodots, microdots, and planar NW [19-22]. Such MOKE measurements are potentially interesting, as it provides a very sensitive probe that is proportional to the change in the nanostructure magnetization, depending on the rotation of the polarization of linearly polarized light on reflection and hence is sensitive to the surface magnetization to a depth in the order of skin-depth [20,22]. However, due to the difficulties arising from locating the position of these structures within the MOKE illumination, the very small size of these structures with respect to the laser spot size, and the curved surfaces which lead to the scattering of reflected light into various

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directions may have contributed to the lack of measurements of isolated cylindrical NW using this technique.

Thus, the surface magnetic state of an individual, a broad cluster of chains and a cluster of small number of poorly aligned template released Ni NW deposited from a dilute suspension onto pre-patterned/SiO₂/Si substrates have been investigated using highly sensitive MOKE magnetometry. The switching fields obtained from applying the magnetic field at different angles with respect to the NW long axis were argued and compared with the literature, as well as, with the theoretical calculations of curling model.

II. EXPERIMENTAL PROCEDURE

Here, an extended 2D array of ferromagnetic Ni NW was prepared using a DC electro-deposition technique. The deposition process was carried out in a (Metro-Ohm) conventional three electrodes electrochemical cell. The NWs were synthesized within the pores (~300 nm diameters) of commercially available alumina template (Anodisc Whatman Inc.) after coating one side with a 100 nm pure gold layer using the sputtering technique. The alumina template was then used as a working electrode in the electrochemical cell which contains an electrolyte solution of about 0.57 M of NiSO₄ and \sim 31 g/l of H₃BO₃. The pH of the solution was controlled to be around 3.5 by adding few drops of a dilute ammonia solution. The electro-deposition was carried out under a constant voltage of about 0.9 V following a linear volumetric sweep results. After filling the nanopores with the required length of ~20 µm NW, the template was removed from the cell and washed very well with acetone followed with Iso-Propanol Alcohol. Further information on the fabrication procedure can be found in Refs [3,9,10,23].

The template was then dissolved and entirely removed in approximately 2 M NaOH solution for a time of ~48 h at room temperature. The suspended NW were washed successively very well with distilled water, followed by IPA, to remove any template residues. Finally, the released NW were left suspended in IPA solution. Single drops of low concentration suspension were then placed onto the center of a gold pre-patterned SiO₂/Si substrate. To overcome the random orientation of the deposited NW, electromagnetic alignment assembly was used [9,10,23]. This procedure, however, creates a distribution of isolated, clusters and chains of NW spread across the whole area of the substrates.

To locate these NW, gold patterns of several micrometers in size were pre-patterned on the SiO_2/Si substrates using conventional electron beam lithography and lift-off techniques, before the dispersion of NW on it. The position of NW relative to these patterns was then carried out using optical or/and scanning electron microscopy (SEM). In this work, high-resolution field emission SEM column on an FEI-Helios Nanolab dual focused ion (Focused Ion Beam)/ electron beam (SEM) system with electron beam energy of 10 keV and 30 keV is used for imaging and pattern production, respectively. Further details on the electron beam lithography and lift-off techniques are available elsewhere [24,25].

Magnetic hysteresis loops of the isolated NW were obtained using a highly sensitive longitudinal MOKE magnetometry. The MOKE setup is well suited for measuring Kerr signal as a function of the applied magnetic field, which is directly proportional to the magnetization of the NW. Helium-Neon laser light with a wavelength of 658 nm has an elliptical spot size of approximately 5–6 μ m. An electromagnet with a maximum field of around ±450 Orested (Oe) was used with a lock-in amplifier to switch the magnetic hysteresis of the NW at a frequency of approximately 21 Hz. The MOKE setup allows for a rotation of the substrate around its surface normal by an angle up to ~180° to apply a magnetic field in the plane of the substrate at different directions. More details on MOKE setup can be found in Refs [20,26].

III. RESULTS AND DISCUSSION

Fig. 1 show examples of normalized hysteresis loops obtained from MOKE measurements of (a) an individual, (b) a broad cluster of chains, and (c) a cluster of small number of poorly aligned template released Ni NW. In each case, the external magnetic field was nominally applied parallel to the NW long axis. These loops were plotted without any corrections. The associated scanning electron micrographs showing the NW interrogated by MOKE laser spot are presented in the figure insets. Histograms of the switching field distributions obtained from repeating measurements on these NW at multiple locations are also presented in the figure insets. The diameters and lengths of the NW investigated here are



Fig. 1. Examples of normalized hysteresis loops of (a) an individual, (b) a broad cluster of chains, and (c) a cluster of poorly aligned template released Ni nanowire (NW) obtained using magneto-optical Kerr effect magnetometry by applying the magnetic field nominally parallel to the NW long axis. The insets are the scanning electron micrographs of the measured NW and the histograms of switching field distribution obtained from repeating measurements on these NW at multiple locations.

approximately 300 nm and 7 μ m, respectively, as measured using SEM.

In general, the MOKE signal is quite poor, indicating the sensitivity required to make these measurements on an individual and small number of clustered NW. From these loops, the magnetic curves show high squareness ratio ~1 (squareness ratio is the remanent magnetization to the saturation magnetization), indicating a characteristic magnetic easy axis along the wire long axis. The loops as shown in Fig. 1b and c show more "structure" in the reversal behavior, where the switching is not sharp as for single NW but has steps in it. This behavior may be expected, as the Kerr signal is likely to arise from more than one NW, which may have different switching fields, leading to two or more switching steps. Alternately, it is well known that MOKE setup is a surface sensitive technique which can probe ~30 nm skin depth [20,22], and since the NW diameter is much larger than this depth, so this behavior might be due to the response of any internal magnetic states or domain structure in the NW. This result may need more investigations using bulk magnetization sensitive techniques such as micromagnetic simulations or/and magnetotransport measurements. These measurements were already performed, and their results will be published in another work.

The average switching field obtained from repeating MOKE measurements on individual Ni NW was found to be ~140 Oe (see the inset of Fig. 1a), whereas the switching field obtained from a cluster of chains was 135–185 Oe (see the inset of Fig. 1b) and for the cluster of poorly aligned NW was 185–200 Oe (see the inset of Fig. 1c). The switching field distribution is very complex and was investigated intensively in other published works [9,10], and it was attributed to the magnetostatic interaction or the disorientation or misalignment of these NW with each other and the applied field. Small distribution of the switching fields (around 10 Oe) is obtained from repeating measurements on the same NW at multiple locations. This was attributed to the disattributed of these NW with each other misalignment of these NW or/and the misalignment of these NW or/and the misalignment of these NW with each other and with the field applied [9,10].

Lupu, *et al.* used MOKE magnetometry to probe the magnetic state of a range of isolated $Ni_{80}Fe_{20}$ NW with diameters ranging from 35 to 300 nm [22]. The estimated switching field of 300 nm diameter from their data was found to be around 100 Oe. As reported elsewhere, increasing the Fe content in the NiFe alloys, decreases the switching field. Comparing this value with the value presented here, a reasonable agreement is clearly obtained,

since the composition of NW investigated here is different from Lupu's NW.

Rheem, *et al.* studied the magnetic behavior of individual $Ni_{85}Fe_{15}$ [13] and individual $Ni_{80}Co_{20}$ [14] cylindrical NW with 200 nm diameters and more than 5 µm lengths using magnetotransport measurements. The estimated switching fields from their data were found to be around 15 and 28 Oe, respectively. Comparing these values with the switching field obtained here, a large difference is clearly seen. This difference may also reflect the variance between the surface and bulk magnetic states in such NW. This comparison indicates that further studies are required to use both surface and bulk magnetic sensitive techniques.

An important finding of all these measurements is the observation of a high squareness ratio. This is quite different from the results reported in literature obtained from measurements of extended 2D arrays of ferromagnetic NW [5-7] which has been measured using other techniques that often display sheared hysteresis loops due to the strong magnetostatic interaction among these NW. Otherwise, it may again reflect the surface compared to the bulk magnetic measurements.

To understand the magnetization reversal behavior and the underlying physical mechanism in such NW, systematic measurements were performed to study the angular dependence of the switching fields. These measurements represented the most challenging part of this work. Typical examples of normalized hysteresis loops obtained from MOKE measurements of an individual Ni NW by applying the magnetic field at different angles (0°, 10°, and 40°) with respect to the NW long axis are shown in Fig. 2. These loops were plotted without any corrections.

In many cases, the hysteresis loops were clearly distorted with additional magneto-optical effects at higher magnetic fields and high angles. From all these measurements, the loops generally show well-defined switching behavior with high remanent magnetization. Square hysteresis loops are expected to occur when the magnetic field is nominally applied parallel to the NW long axis, due to their high aspect ratio (length to diameter/thickness of the NW) and the dominance of shape anisotropy. However, theoretically the shape of the hysteresis loops should vary with increasing NW angle, therefore the square loops at higher angles indicates either that; the easy axis of magnetization rotates with the angle of applied field, or the difficulty in recognizing the change in the shape of these loops due to the distortion occurred with other magneto-optical effects. A further



Fig. 2. Examples of normalized hysteresis loops of an individual template released Ni nanowire (NW) obtained using magneto-optical Kerr effect magnetometry when the magnetic field was applied at different angles: (a) 0°, (b) 10°, and (c) 40° with respect to the NW long axis.

possibility is that the MOKE setup was actually detecting two signals arising from the transverse component in addition to the expected longitudinal component of the magnetization within these NW. The transverse component, perhaps, gives rise to the square hysteresis loops at higher angles. This result may also support the difference between surface and bulk magnetization behavior. Thus, bulk magnetization sensitive techniques are highly recommended to use for probing the magnetization behavior of thick cylindrical NW [27,28].

Significantly, these loops demonstrate that there is a strong correlation between the switching field and the angle of applied magnetic field to the NW long axis. The extracted switching fields from these loops were plotted as a function of the NW long axis angle with respect to the applied field, as shown in Fig. 3a. Distributions of the switching field values were obtained from repeating measurements at more than 20 multiple locations on the same wire at each angle are included in the plots as an error bar. These error bars (around 3 Oe) are smaller than the data points. Clearly, the switching field increases slowly with increasing NW angle up to about 25° and then increased rapidly at higher angles.

There are various distinct mechanisms that are able to describe the magnetization reversal processes in such NW. For instance; coherent rotation of the Stoner-Wohlfarth model appears when the nanostructures are smaller than the exchange length, whereas curling and buckling modes occur when these structures are larger than the exchange length [29-31].

The magnetization in buckling mode appears as a wave configuration along the planar thin NW, whereas curling mode shows spiral magnetization state along the cylindrical NW. The applicability of these mechanisms to a system, however, depends on various parameters including geometries, shapes, defects, morphology, and instantaneous magnetic state of the nanostructures [29-34].

To understand and explain the angular dependence of the switching fields presented here, it should first be noted that since the diameters (~300 nm) of these NW are much larger than the exchange length, therefore one can expect a deviations of the micromagnetic structure away from the uniform rotation behavior that is described by the Stoner-Wohlfarth reversal model [33,34]. Since buckling behavior is expected to occur



Fig. 3. (a) Average switching fields extracted from magneto-optical Kerr effect loops as a function of nanowire (NW) long axis angle with respect to the applied magnetic field for an individual template released Ni NW. The error bars (3 orested) are smaller than the data points.(b) Normalized switching fields as a function of NW long axis angle with respect to the field applied for an individual template released Ni NW.

The dashed line represents the theoretical curling model.

when the NW diameter is comparable to the exchange length (reduced radius around unity), buckling may also be excluded from the applicability here. The shape and aspect ratio of the NW investigated here is far from being a chain of spheres which would reverse as in the fanning model [32].

Therefore, the angular dependence of the switching fields investigated here is likely to be the consequence of an incoherent rotational process following the curling model of reversal with a maximum reversal field obtained along the hard axis at perpendicular direction. Accordingly, the experimental results obtained here were compared with the theoretical calculations of curling model after normalizing the switching fields to the minimum values using the equations presented elsewhere [33,34]. This is plotted in Fig. 3b. Clearly, at low angles, a good agreement is obtained, whereas there is a very slight deviation at higher angles. The agreements between the experimental and theoretical calculations at low angles suggest that the curling mode of reversal is a reasonable analytical representation of the magnetization reversal processes in such individual NW. The deviation at higher angles may be due to the more complex magnetization structure. These results are also in agreement with the results reported in literature using other characterizing techniques [11,13]. As examples, individual Ni and Ni₈₅ Fe₁₅ [13] NW with 200 nm diameters were investigated using magnetoresistance measurements at 10 K and room temperature, respectively. Their data were found to fit very well with the curling model of reversal, and according to their calculations, the size of curling reversal was found to be much smaller than the NW diameter.

IV. CONCLUSIONS

The surface magnetization behavior of individual, broad clusters of chains and a cluster of small number of poorly aligned template released Ni NW was investigated using a highly sensitive MOKE magnetometry. These measurements were performed by applying the magnetic field at various angles with respect to the NW long axis. Square hysteresis loops with high squareness ratios were obtained from all the angles of measurements. Square hysteresis loops were obtained, when the magnetic field was applied parallel to the NW long axis, indicating that the shape anisotropy is dominating on the behavior whereas at higher angles, indicating that the easy axis of magnetization rotates with the field applied or due to the detection of both transverse and longitudinal components of magnetization. The transverse component might give rise to the square hysteresis loops. There was a strong correlation between the switching field and angle of applied field with respect to the NW long axis. Although the angular dependence of switching field was adequately fitted to the non-uniform rotation of the curling model of domain reversal at low angles, the magnetization reversal within these NW may occur in a different way. The results reported here demonstrate that more investigations are required to use both surface, and bulk magnetization sensitive techniques, such as micromagnetic simulations or/and magnetoresistance measurements to ascertain from the results described here.

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