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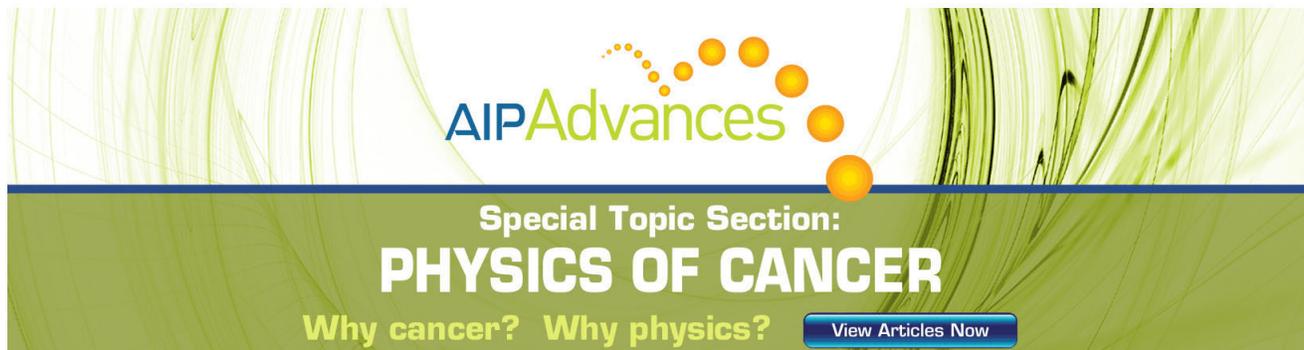
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Magnetic properties of planar arrays of *Fe*-nanowires grown on oxidized vicinal silicon (111) templates

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Planar arrays of *Fe* nanowires (NW) grown on oxidized self assembled Si templates are shown to be ferromagnetic at room temperature with wire width down to 30 nm as revealed by magnetometry and x-ray magnetic circular dichroism studies. The atomic terrace low angle shadowing (ATLAS) method used to produce these NW arrays allows one to grow planar arrays that are several nanometers thick as opposed to monolayer thickness attained with step flow and step decoration methods. These NW arrays possess much smaller width fluctuations along the wire length owing to the highly periodic nature of the step-bunched templates. Magnetic anisotropy of the NW array is dominated by the shape anisotropy which keeps the magnetization in-plane with easy axis along the length of the wires. © 2011 American Institute of Physics. [doi:10.1063/1.3554264]

There has been a considerable amount of interest in studying magnetic nanowires (NWs) over the span of the last two decades, due to their application potential in spin electronic devices and interesting fundamental physics issues.^{1,2} Realization of magnetic nanowire is achieved either through the bottom up or top down approaches. For the bottom up approach, template mediated synthesis of NWs is an attractive option, owing to faster throughput.^{3–8} In commonly used anodized alumina templates, the NWs grow along the thickness of the pores of the templates leading to out-of plane array of NWs. However, there are relatively few reports related to the production of planar NW arrays based on self assembled templates.^{9,10} Planar NW arrays on self assembled templates could be realized using step flow, step decoration^{9,11,12} and reactive deposition epitaxy (RDE)¹³ methods on vicinal templates. The small thickness achieved using these methods (leading to superparamagnetism) and material selective nature (applicable only for certain NW material and substrate combinations) restricts their scope for future applications.

To overcome the obstacles related to the small thickness of NWs in planar arrays one can use a shallow angle deposition on vicinal surfaces.^{14,15} This method was demonstrated to produce *Fe* NW arrays down to 45 nm NW width, that are ferromagnetic at room temperature (RT).¹⁴ In this paper we report on the growth of ferromagnetic planar NW arrays of *Fe* using the atomic terrace low angle shadowing (ATLAS) technique.^{16,17} These planar NW arrays are ferromagnetic at RT down to 30 nm wire width and show an in-plane uniaxial anisotropy related to the shape of the NWs.

The vicinal *Si* templates used in the present study were prepared by performing a dc-current annealing¹⁸ under ultra-high vacuum (UHV) conditions on n-type doped *Si* (111)

with resistivity 1–10 Ω.cm. The *Si* substrates had a miscut of 2.5 or 4 degrees along the (11 $\bar{2}$) crystallographic direction. The samples were annealed at 1120 °C or 920 °C with a direct current perpendicular to the step edges, with current being either in the ascending (uphill, UH) or descending step (downhill, DH) directions. Annealing at 1120 °C with uphill current leads to highly periodic templates with 3.2 μm periodic array of regular step-bunches (1.1 μm) and terraces (2.1 μm). Annealing at 920 °C with UH (DH) current produces arrays of 75 (1100) nm periodic arrays with step heights being 35 (2.7 nm). These annealed *Si* templates were oxidized (about 100 nm oxide thickness) using standard thermal oxidation procedure carried out at 830 °C for a duration of 15 hrs.

Growth of *Fe*-NWs was carried out at RT by depositing *Fe* (0.02–0.04 Å/s) onto the oxidized templates at a small angle (0.5–5°) using a multi-pocket e-beam evaporator (Telemark, USA) in a UHV chamber with a base pressure of better than 5×10^{−10} Torr. Details of the ATLAS method are given elsewhere.^{16,17} For magnetization studies, the *Fe*-NWs samples were capped with a 5 nm *MgO* layer. The topography and morphology of the templates and NW arrays was checked with an atomic force microscope (AFM), (Solver Pro, NT MDT) and scanning electron microscopy (SEM) (Ultra Microscope, Zeiss).

Magnetic properties of the NW arrays were examined using a vibrating sample magnetometer (PPMS, Quantum Design) with a sensitivity of 5 × 10^{−7} emu. Uncertainty in the NWs volume determination could be as large as 10% arising mostly from the statistical fluctuations in the terrace coverage that are related to the distribution of step-terrace periodicity of the template, whereas the thickness of the deposited material is accurate within ~2%.

The element specific x-ray absorption (XAS) and x-ray magnetic circular dichroism (XMCD) experiments were carried out at the ESRF's ID08 beamline.

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Figure 1 shows the AFM images of planar arrays of *Fe*-wires (micro- and nano-) grown on oxidized templates of three different periodicities ranging between $3.2\ \mu\text{m}$ to $75\ \text{nm}$. Deposition of NW arrays is realized by driving the deposition flux of *Fe* toward the downhill direction of the step-bunched surface. It can be seen from the figure that depending on the deposition angle and miscut strength, one can change the coverage of the terraces (i.e. wire width) by modifying the extent of shadowing of the deposited *Fe* flux caused by the step-bunches. Figure 1(a) shows an AFM phase image of $\sim 1\ \mu\text{m}$ wide *Fe* micro-wire array prepared at a deposition angle of 1.2° on $3.2\ \mu\text{m}$ periodic template. Average terrace coverage in this case is 50%. Figure 1(b) is a *Fe* NW array grown at a deposition angle of 1.8° on $1.1\ \mu\text{m}$ periodic template, leading to an average terrace coverage of 80% corresponding to an average wire width of $400\ \text{nm}$. The average thickness of the *Fe*-NWs determined from the height profile (not shown) for this sample is $4.8\ \text{nm}$. Figure 1(c) shows an AFM height image of a *Fe*-NW array with average periodicity of $75\ \text{nm}$ (4° miscut and oxidized vicinal Si (111) template) and $30\ \text{nm}$ wire width. This array was grown at a deposition angle of 1.9° which corresponds to an average terrace coverage of 50%. Height profile corresponding to the dashed line marked on Fig. 1(c) is shown in Fig. 1(d). One notices that the periodic arrays of *Fe* are quite regular with wires remaining straight up to several μm in length even for $30\ \text{nm}$ wire width arrays leading to a large aspect ratio (length/width ratio). Another noticeable feature is an island type of morphology of the wires, more clearly visible in Fig. 1(b). This island type morphology is related to the fact that *Fe* grows with an island type growth mode on oxidized silicon. These islands are isolated in the initial stages of the *Fe* growth forming discontinuous chains of aligned nano-islands on the terraces. With increasing thickness, the density of these islands is enhanced leading to coalescence of the islands and forming NWs of coalesced islands. The crystal structure of *Fe* in the NW arrays was found to be *bcc*, as revealed by the transmission electron microscopy investigations. Interfaces ($\sim 1\ \text{nm}$ thickness) of *Fe*-NWs, formed with the SiO_2 substrate and the cap layer of MgO possess a crystal structure that resembles *fcc Fe-O* structure.

Figure 2 shows magnetization hysteresis loops of a *Fe*-NW array having $30\ \text{nm}$ average wire width (50% coverage grown on $75\ \text{nm}$ periodicity templates at a deposition angle of 1.2°) measured at $300\ \text{K}$ and capped with $5\ \text{nm}$ MgO layer.¹⁹ The thickness of the *Fe*-NW array is $2.8\ \text{nm}$ in this case. From the magnetization hysteresis M-H loop measured at $300\ \text{K}$, it is clearly evident that irrespective of the magnetic field direction i.e., either parallel (H_{\parallel}) and perpendicular (H_{\perp}) to the length of the wires, the magnetic hysteresis demonstrates that the wires are ferromagnetic at RT. The approach toward magnetic saturation is easier for H_{\parallel} as compared to H_{\perp} . No sign of out-of plane anisotropy was found from the out of plane M-H loops measured at $300\ \text{K}$ and $10\ \text{K}$. Temperature (T) dependent studies (not shown) showed an increase in the coercivity (H_C) and remnant magnetization (M_R) with decrease in T . Values of H_C and M_R are found to be $60(675)$ Oe and $31(61\%)$ at $300(10)$ K for H_{\parallel} .

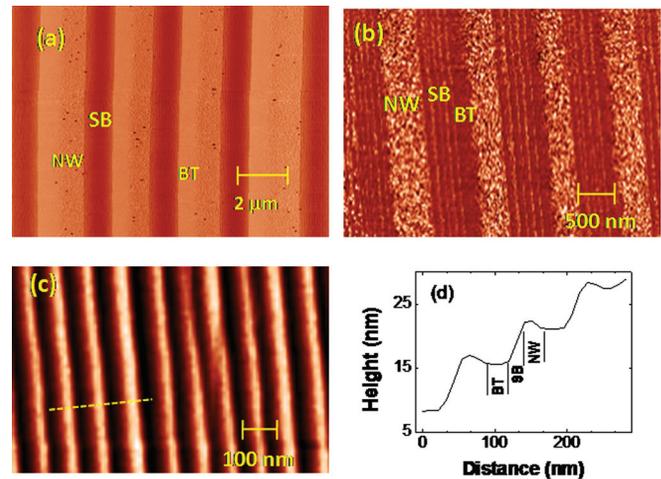


FIG. 1. (Color online) (a) AFM phase image of a *Fe* wire array grown at a deposition angle of 1.2° on oxidized *Si* template (miscut 2.5°) $3.2\ \mu\text{m}$ periodic template with an average wire width of $1\ \mu\text{m}$. (b) Phase image of a *Fe*-NW array grown on $1.1\ \mu\text{m}$ periodic oxidized *Si* template (miscut 2.5°) template at a deposition angle of 1.7° , leading to 80% terrace coverage ($400\ \text{nm}$ wire width). (c) AFM height image of a *Fe*-NW array grown on $75\ \text{nm}$ periodic oxidized *Si* template (miscut 4°) template at a deposition angle of 1.9° . Average width of NWs in this case is $30\ \text{nm}$. A height profile corresponding to the dashed line marked on the Fig. 1(c) is shown in Fig. 1(d). Labels BT, NW and SB on the Figs. 1(a), 1(b) and 1(d) denote bare terrace, nanowire and step bunch respectively. Direction of deposition flux is from left to right for all the three samples.

Corresponding values for H_{\perp} at $300(10)$ K are found to be $50(500)$ Oe and $8(56)\%$ respectively. Only a marginal increase ($\sim 5\text{--}8\%$) in the magnitudes of saturation magnetization was observed with a decrease in temperature from $300\ \text{K}$ to $10\ \text{K}$ for both samples. Enhanced H_C for H_{\parallel} as opposed to H_{\perp} is consistent with the shape anisotropy origin of the enhanced H_C as discussed within the Stoner-Wohlfarth description. The uniaxial anisotropy related to the shape anisotropy of the wires is preserved at low temperatures (down to $10\ \text{K}$). The magnetization results suggest that the wires are continuous and show expected anisotropy related to the large aspect ratio (length to width ratio) of the wires.

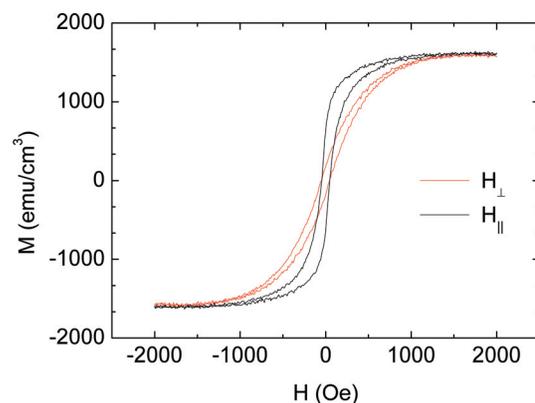


FIG. 2. (Color online) Magnetization hysteresis loop of *Fe* NW array ($2.8\ \text{nm}$ thickness, $30\ \text{nm}$ width) deposited on an oxidized *Si* template of $75\ \text{nm}$ periodicity measured with an in-plane magnetic field directed either along or across the length of the NWs.

Figure 3 shows the XAS spectra measured at the $Fe L_{3,2}$ edge for a Fe -NW array with average wire width and thickness being 40 nm and 2.7 nm respectively, and capped with 5 nm MgO layer. The spectra were normalized to the incident flux and pre-edge. XAS line shape is typical of metallic Fe but shows sign of oxidation, owing to the presence of oxidized interface with the cap layer of MgO and hybridization of $O2p$ - $Fe3d$. XAS spectra show a much reduced signal corresponding to hybridization of oxygen with Fe after 15 min Ar^+ ion etching. XMCD signal ($\mu^+ - \mu^-$) determined from the two XAS spectra is also shown in the lower panel. Here, μ^+ (μ^-) refers to the absorption coefficient for the photon helicity parallel (antiparallel) to the $Fe 3d$ majority spin direction. The XMCD sum rules^{20,21} were applied to extract information on the spin and orbital magnetic moments of the Fe -NW arrays both at RT (300 K), and low temperature (10 K). The calculated values of magnetic moments at 300 (10) K are found to be; spin magnetic moment (m_s) = 2.68 (3.15) μ_B /hole, orbital magnetic moment (m_l) = 0.01 (0.04) μ_B /hole. For the evaluation of m_s and m_l , we assumed n_h (number of holes in the 3d states) = 3.4 for iron,²² and negligible value of spin dipolar term. We also notice that the dimensionality effects that are known to enhance m_s and m_L in ultrathin Co atomic chains¹⁰ are absent in these NW arrays of Fe due to relatively large thickness of the wires.

Our results show a reasonable improvement in reduction of the NWs width from previous reports^{14,15} and overcoming

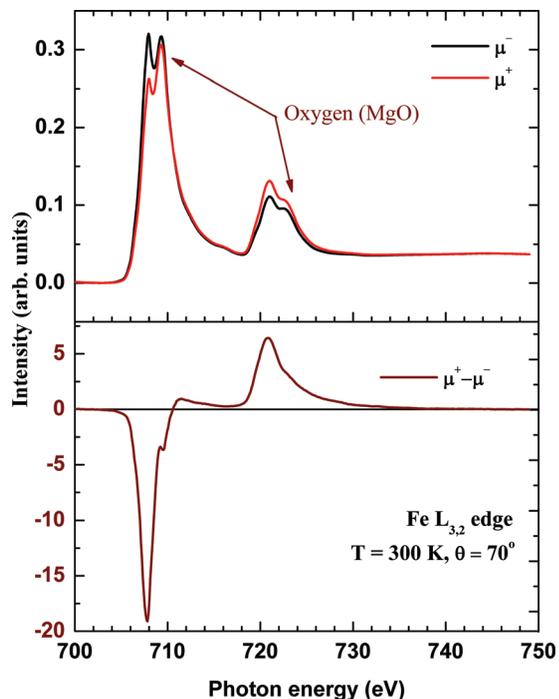


FIG. 3. (Color online) X-ray absorption spectra taken for the $Fe L_{3,2}$ edge for a 40 nm average width Fe -NW array on 120 nm periodicity oxidized silicon template. Spectra were recorded in TEY geometry with an incidence angle of 70 degrees to the surface normal at 300 K for magnetization parallel and antiparallel to the x-ray polarization vector (black and red curves). Strength of the applied magnetic field was 5 Tesla. In the lower panel the MCD curve (marked $\mu^+ - \mu^-$) using the sum rules described in text are shown.

the superparamagnetism associated with the small thickness of the NWs in planar NW arrays, by growing thick NWs on self assembled templates. Another important point to notice is that the width fluctuations along the length of the wire are quite low, owing to the highly periodic nature of the templates used in this investigation. In a previous step decoration study¹² the amount of wire width fluctuation along the wire length was huge (20–200% of wire width) as opposed to our case. Here, we have shown that by selecting an appropriate combination of deposition angle and template periodicity one can grow magnetic NWs of varying width and periodicity.

In summary, planar array of Fe -NWs grown using ATLAS method with wire widths down to 30 nm possess ferromagnetic behavior at room temperature with a preference of easy axis along the length of the wires. H_C of the arrays is greater for H_{\parallel} than H_{\perp} direction owing to the shape anisotropy of the wires. Element specific XMCD studies at $Fe L_{3,2}$ edge confirms the ferromagnetic nature of wires at 300 K. Presence of oxidized interfaces between the Fe -NWs and the cap-layer of MgO owing to $O2p$ - $Fe3d$ hybridization was also noticed.

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