Effect of Aspect Ratio and Temperature on Magnetic Properties of Permalloy Nanowires

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Arrays of permalloy nanowires (NWs) were fabricated into the pores of self-engineered Anodic Aluminium Oxide (AAO) templates by a simple electrodeposition technique (EDT) with a diameter \( \sim 200 \) nm. By varying the length of the nanowires from 1.5 to 7.5 \( \mu \)m, got corresponding changes in aspect ratio from 7.5 to 37.5. We studied the growth and structural properties of permalloy nanowires using scanning electron microscopy, X-ray diffraction spectra and transmission electron microscopy. We performed the magnetic measurements of permalloy nanowires using vibrating sample magnetometer. The coercivity \( (H_c) \) and anisotropy field \( (H_{an}) \) observed considerably higher when the applied magnetic field was perpendicular to the nanowires axial direction (In-Plane) compare to the applied magnetic field was parallel to the nanowire axial direction (Out-of-plane). Reverse trend was observed for the maximum remanent ratio \( (M_r/M_s) \). So we concluded that the magnetic easy axis of the permalloy nanowires are along the axial direction of the nanowires and magnetic hardness of the nanowires higher in transverse direction compare to the axial direction of nanowires. For the first time temperature (80 K \( \leq T \leq 300 \) K) and aspect ratio dependence magnetic properties of the permalloy nanowires have been studied in detail.

Keywords: Nanowires, Electrodeposition, Magnetic Anisotropies, Magnetic Easy Axis.

1. INTRODUCTION

Magnetic nanowires (NWs) have recently attracted tremendous attention because of their potential applications in various fields, particularly in high density perpendicular magnetic storage.1–4 Highly ordered arrays of such nanowires show novel and interesting magnetic properties different from those of bulk and thin films and sensitively depend on their sizes, shapes, and the interactions among them.5–7 Among the various methods for preparing nanowires, electrodeposition is widely used because of its simplicity and low cost. In this method, various magnetic materials such as Fe, Co, Ni, and their alloys were embedded into the nanopores of silica, alumina, or polycarbonate membranes by the electrodeposition technique.8–10 Magnetic properties of a NW are governed by various anisotropy energies such as shape, magnetocrystalline and surface anisotropies.6–11 However the magnetic behavior of the arrays of nanowires is determined by the magnetic nature of individual nanowires as well as by the interactions among them. The direction of magnetic easy axis of a NW is determined by the direction of effective anisotropy field in it. Due to the strong shape anisotropy in a NW, magnetic easy axis is usually parallel to its length. But in an array of closely arranged NWs, strong magneto-static interaction is developed because of close proximity of neighbouring NWs, resulting the direction of easy axis perpendicular to its length which is undesirable in most of the applications.12,13 Since last decade many magnetic properties studies have been done on the permalloy nanostructures (nanowire, nanotube, nanoparticle etc.).14–21 Still aspect ratio and temperature dependent magnetic properties of permalloy NWs has not done yet. It is still not studied that how magnetic easy axis of permalloy nanowires depends upon the temperature and the aspect ratio.

In this back drop, here we study the structural and magnetic properties of the arrays of permalloy NWs with average NW diameter of \( \sim 200 \) nm were prepared in AAO membrane by the electrodeposition method. The aspect ratio of the nanowires was varied from 7.5 to 37.5 by varying the deposition timing and keeping the diameter...
Figure 1. Illustration of the mechanism for the formation of permalloy nanowires.

constant $\sim 200$ nm. Magnetic properties of the samples such as hysteresis loop, coercivity ($H_C$), and squareness ($M_r/M_s$) ratio were studied at various temperatures between 80 and 300 K with above mentioned different aspect ratio. The experimental result confirms the direction of magnetic easy axis of the NWs along the axis of the NWs.

2. EXPERIMENTAL DETAILS

High-density arrays of permalloy NWs have been fabricated by the synthesized template assisted electrochemical route (see Fig. 1). A self-organized nanoporous anodic aluminium oxide (AAO) template fabricated by the controlled two-stage electrochemical anodization of high purity Al foil, as described elsewhere,$^{22-24}$ was used as the host to prepare the permalloy NWs. A through pore AAO template with average pore diameter of $\sim 200$ nm with a thickness of $\sim 50 \mu$m and an average pore density of $\sim 10^9$ cm$^{-2}$ was prepared for this work. A layer of Au sputtered onto one side of the through pore AAO template was used as the working electrode in the electrodeposition process. The software controlled typical conventional three electrode electrochemical cell and a power supply (potentiostat AutoLab-30) was employed for the electrochemical deposition of permalloy NWs by using the gold-coated AAO template as the working electrode (cathode). A high purity Pt wire and an Ag/AgCl calomel electrode were used as the counter and reference electrode, respectively. Permalloy NWs have been electrodeposited in the pores of AAO using an aqueous solution of 100 g L$^{-1} \text{NiSO}_4\cdot7\text{H}_2\text{O}$, 20 g L$^{-1} \text{FeSO}_4\cdot7\text{H}_2\text{O}$ and 45 g L$^{-1} \text{H}_3\text{BO}_3$ as the electrolyte at room temperature (RT). Here, the boric acid plays the role of the buffer. The pH of the electrolyte was...
Effect of Aspect Ratio and Temperature on Magnetic Properties of Permalloy Nanowires
Singh and Mandal

maintained at 3.5 and the electrodeposition was conducted by using a dc voltage of $-1.03\,\text{V}$ following linear sweep voltammetry results. The electrodeposition was carried out for 5, 15, 30 and 45 min in order to prepare permalloy NWs of $\sim1.5, 3.2, 5.4$ and $7.5\,\mu\text{m}$ length respectively. Afterwards, the template containing the permalloy NWs was dipped in a $2\,\text{M NaOH}$ solution at RT for a day to release the permalloy NWs by dissolving the template.

The crystal structure study of the permalloy NWs were analyzed by X-ray diffraction (XRD, Panalytical X’Pert Pro diffractometer). The account of the chemical composition and elemental composition of the NW was investigated by energy dispersive X-ray (EDAX). The morphology and the structure of the NWs were studied by scanning electronmicroscope (SEM, FEIQuanta-200 Mark-2), transmission electron microscope (TEM, FEITECNAI G2 TF20ST) and scanning transmission electron microscopy (STEM). The crystalline structure of the NW was further investigated by the high-resolution TEM (HRTEM). Magnetic measurements of the permalloy NWs have been performed using a vibrating sample magnetometer (VSM, Lakeshore, model 7144) by placing the axis of the arrays of NWs sample along the direction of the applied magnetic field (OP-out of plane, $\mathbf{H}||\text{wire axis}$) and perpendicular to the direction of the applied magnetic field (IP-in plane, $\mathbf{H}\perp\text{wire axis}$) within the temperature range of $80–300\,\text{K}$.

3. RESULTS AND DISCUSSION
3.1. Morphology, Crystallography and Chemical Composition

From SEM micrograph (Fig. 2(a)) we observed that the pores of Anodized alumina oxide (AAO) template obtained after two step anodization technique have uniform pore diameters with average pore diameter of $\sim200\,\text{nm}$ with a thickness of $\sim50\,\mu\text{m}$ and an average pore density of $\sim10^9\,\text{cm}^{-2}$. The crystallographic nature of the arrays of permalloy NWs investigated by XRD (Fig. 2(b)) indicates that the as grown permalloy NWs are crystalline in nature. The diffraction peaks in the XRD pattern appear from the pure FeNi$_3$ face centered cubic (fcc) and the metallic Au layer underneath the NWs. The characteristic peaks of FeNi$_3$ at 44.3 (111) and 51.4 (200) were observed in the XRD patterns after electrodeposition. No iron and nickel

Figure 3. (a) TEM micrograph of permalloy nanowire for AR 37.5. (b) Typical HRTEM image and (inset of (b)) SAED pattern indicating the polycrystalline nature of the NW. (c) Corresponding intensity profile for the line across the lattice fringes. (d) EFTEM micrograph (colour mapping) of the as-prepared permalloy nanowires.
oxides, hydroxides or other impurity phases are detected. Figure 2(c) shows the scanning electron microscopy (SEM) image of as-prepared permalloy NWs grown perpendicular to the supporting Au substrate. It is evident from Figure 2(c) that the length and the diameter of the NWs are uniform in nature as well as the surface of the nanowires is smooth. The local elemental composition of the as-prepared permalloy NWs was studied by EDAX microanalysis at the single NW level, shown in Figure 2(d). It confirms that the permalloy NW are composed of Fe and Ni elements. The elemental composition of Fe:Ni is 1:3 respectively, in atomic and weight percentage.

The transmission electron microscope (TEM) and the high resolution transmission electron microscope (HRTEM) micrographs of as-prepared permalloy NWs shown in Figures 3(a) and (b), respectively, clearly shows the formation of permalloy NW ∼ 200 nm diameter. It is also evident from Figures 3(a) and (b) that the formation of NW is uniform in nature. The inset image of the Figure 3(b) shows the selected area electron diffraction (SAED) pattern of the permalloy NW which indicates the polycrystalline nature of the NW. Figure 3(b) shows a HRTEM image at the selected edge of an individual permalloy NW from Figure 3(a). As shown in Figure 3(c), a typical intensity profile covers the line scan (labeled by a line in Fig. 3(b)) across the lattice fringes. The periodic fringe spacing of 0.21 nm agrees well with interplanar spacing between the (111) planes of the permalloy NW. Figure 3(d) shows the typical EFTEM-EDX elemental colour mapping images of an individual permalloy NW. The results confirm the existence of Fe and Ni elements, which are distributed homogeneously over the entire permalloy NW.

### 3.2. Magnetic Properties

Figure 4 shows the OP (Hparallel wire-axis) and IP (Hperpendicular wire axis) hysteresis loops of the four samples at room temperature. For all samples, the change in magnetization with the magnetic field in the IP hysteresis loops is much less than that in the corresponding OP hysteresis loops. Remanence in the IP direction is also much smaller compared to the OP counterpart. Along the OP direction, magnetization saturates at a lower field compared to the IP direction. In the absence of an external magnetic field, the exchange interaction and different anisotropy fields e.g., shape, magneto crystalline, surface anisotropy fields, determine the equilibrium magnetization of a ferromagnetic nanostructure.

At remanence, the arrangement of spins in a ferromagnetic cylinder can give rise to a flower or vortex state depending on the diameter of it. In flower state, the spins are parallel to each other and are oriented along the NW axis. It follows a coherent magnetization reversal and produces square shaped hysteresis loop. Whereas in the vortex state, the magnetic spins are tilted in the circumferential direction and make a certain angle to each other. In this state, curling type of reversal takes place giving rise to a tilted hysteresis loop with much low remanence and high coercivity as observed in the Figure 4. Here, the experimental data pointing towards the remanent state of our NWs is to be the flower state. All these characteristics

![Figure 4](image)

**Figure 4.** In-plane and out-of-plane magnetic hysteresis loops of arrays of nickel nanowires having an aspect ratio of (a) 7.5, (b) 16.0, (c) 27.0 and (d) 37.5.

![Figure 5](image)

**Figure 5.** (a) Temperature dependence of in-plane and out-of-plane coercivity of arrays of permalloy nanowires having an aspect ratio of 7.5, 16.0, 27.0, and 37.5. (b) Variation of in-plane and out-of-plane coercivity of arrays of permalloy nanowires with the aspect ratio at temperatures 80 and 300 K. Error bars for the measurements are shown in the graph.
Effect of Aspect Ratio and Temperature on Magnetic Properties of Permalloy Nanowires

Singh and Mandal

indicate the direction of the domain magnetization parallel to the wire axis or the magnetic field. Which means the magnetic easy axis on the NWs are along the axis of the NWs. Temperature dependence (80–300 K) of OP coercivity ($H_{OP}^{C}$) and IP coercivity ($H_{IP}^{C}$) of the four samples is shown in Figure 5(a). $H_{OP}^{C}$ and $H_{IP}^{C}$ of the four samples decrease with the increase in temperature. One of the possible reasons for this effect may be due to the reduction in magnetocrystalline anisotropy energy with the increase in temperature as observed in most of the ferromagnetic materials. Figure 5(b) shows the variation of $H_{OP}^{C}$ and $H_{IP}^{C}$ with the aspect ratio at 80 and 300 K. At 300 K, $H_{OP}^{C}$ changes from 137 to 99 Oe and $H_{IP}^{C}$ changes from 167 to 90 Oe due the change in the aspect ratio from 7.5 to 37.5. At 80 K, $H_{OP}^{C}$ changes from 466 to 245 Oe and $H_{IP}^{C}$ changes from 610 to 519 Oe due to the same change in the aspect ratio. $H_{OP}^{C}$ is higher than $H_{IP}^{C}$ for all aspect ratios used in the present study.

In-plane squareness ratios, ($M_r/M_s)^{OP}$, of all the samples are very small (Fig. 6(a)) and remain almost constant with temperature. On the other hand, out-of-plane squareness ratios, ($M_r/M_s)^{IP}$, are remain almost constant below room temperature and shows significant sudden decrease with the increase in temperature from 80 to 300 K. The variation of squareness on the aspect ratio is shown in Figure 6(b) at 80 and 300 K. ($M_r/M_s)^{OP}$ increases with increase of aspect ratio for 80 K and vice versa observed for 300 K. ($M_r/M_s)^{IP}$ have an initial increase at lower values of the aspect ratio then started decreasing for 80 K and vice versa observed for 300 K.

Temperature dependence (80–300 K) of OP anisotropy field ($H_{OP}^{an}$) and IP coercivity ($H_{IP}^{an}$) of the four samples is shown in Figure 7(a), we calculated from their hysteresis loops. $H_{OP}^{an}$ of the four samples shows variation increase and decrease with the increase in temperature from 80 to 300 K. $H_{IP}^{an}$ of the four samples follow the reverse trend of $H_{OP}^{an}$ with the increase in temperature from 80 to 300 K. Figure 7(b) shows the variation of $H_{OP}^{an}$ and $H_{IP}^{an}$ with the aspect ratio at 80 and 300 K. At 300 K, $H_{OP}^{an}$ changes from 4761 to 6694 Oe and $H_{IP}^{an}$ changes from 8239 to 7298 Oe due the change in the aspect ratio from 7.5 to 37.5. Which indicates that $H_{OP}^{an}$ increases with the increase of aspect ratio and $H_{IP}^{an}$ shows the reverse trend. At 80 K, $H_{OP}^{an}$ changes from 4802 to 6216 Oe and $H_{IP}^{an}$ changes from 8770 to 7220 Oe due to the same change in the aspect ratio. $H_{IP}^{an}$ is higher than $H_{OP}^{an}$ for all aspect ratios used in the present study. Above studies clearly indicate that the

![Figure 6](image6.png)  (a) Temperature dependence of in-plane and out-of-plane squareness ratio ($M_r/M_s)^{OP}$ of arrays of permalloy nanowires having an aspect ratio of 7.5, 16.0, 27.0, and 37.5. (b) Variation of in-plane and out-of-plane squareness ratio ($M_r/M_s)^{IP}$ of arrays of permalloy nanowires with an aspect ratio at 80 and 300 K. Error bars for the measurements are shown in the graph.

![Figure 7](image7.png)  (a) Temperature dependence of in-plane and out-of-plane anisotropy field ($H_k$) of arrays of permalloy nanowires having an aspect ratio of 7.5, 16.0, 27.0, and 37.5. (b) Variation of in-plane and out-of-plane anisotropy field ($H_k$) of arrays of permalloy nanowires with an aspect ratio at 80 and 300 K. Error bars for the measurements are shown in the graph.
magnetic easy axis direction of the permalloy NWs are in the axial direction of the NWs.

$H_{an}^{aw}$ and $H_{an}^{OP}$ includes all the anisotropies that are present in the NWs in that direction. Anisotropies arise from geometrical shape, crystallinity of the NWs and magnetostatic interaction among the NWs are believed to be the most prominent contributions of $H_{an}^{OP}$. Shape anisotropy of a NW originates from the self-demagnetization field of the NW. Numerical calculations of demagnetization fields, of the NWs used in our study show that, the shape anisotropy ($H_{sh}$) always tries to make the OP as easy direction of magnetization. From the above discussion it is clear that, $H_{sh}$ is much lesser in 200 nm NWs as they consist of textured fcc as well as polycrystalline phase. From the above discussions it is clear that, all the NWs we studied and the resultant of these two tries to make the OP as the easy direction of magnetization.

4. CONCLUSION

In summary, permalloy nanowires with an average diameter of ~ 200 nm have been successfully fabricated in the pores of the self-engineered anodized aluminium oxide (AAO) templates by electrodeposition technique. By varying the length from 1.5 to 7.5 μm, got corresponding changes in aspect ratio from 7.5 to 37.5. Structural and magnetic studies of these nanowires have been done in details. Temperature and aspect ratio dependent in-plane and out-of-plane magnetic measurements of the permalloy nanowires indicate the magnetic easy axis of the nanowires along their axial direction, which is very desirable in magnetic recording device applications.

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References and Notes