

Electrospinning of Fe, Co, and Ni Nanofibers: Synthesis, Assembly, and Magnetic Properties

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Received January 30, 2007. Revised Manuscript Received April 10, 2007

A general synthetic method has been developed to fabricate and assemble ferromagnetic transition metal nanofibers. By employing the novel electrospinning technique followed by subsequent heat treatment, we have successfully prepared uniform nanofibers of Fe, Co, and Ni with diameters of ~ 25 nm and lengths longer than $100\ \mu\text{m}$. Using a specially designed fiber collector, we can conveniently assemble magnetic nanofibers into aligned arrays. The electrospun magnetic metal nanofibers have unique magnetic properties, with much enhanced coercivities relative to bulk materials. The outstanding features of this approach to get one-dimensional magnetic nanostructure are its simplicity, effectiveness, and ease of assembly. Therefore, electrospun magnetic nanofibers can potentially be used in fabrication of high-density magnetic recording, magnetic sensors, flexible magnets, and spintronic devices.

1. Introduction

In the past decade, more and more attention has been drawn to the fabrication of one-dimensional (1D) nanomaterials, including nanotubes, nanowires, nanofibers, and nanobelts because they show some distinctive properties compared with bulk materials.¹ Particularly, 1D magnetic nanomaterials are expected to have interesting properties, as the geometrical dimensions of the material become comparable to key magnetic length scales, such as the exchange length or the domain wall width.² Magnetic nanowires have been extensively exploited for their utilization in high-density magnetic recording, spintronic devices, magnetic sensors and magnetic composites.^{3–6} For example, nanoscale magnetic logic junctions have recently been fabricated with ferromagnetic nanowires as building blocks;⁴ magneto-optical switches have been prepared using suspensions of ferromagnetic nanowires.⁵ Magnetic nanowires are also scientifically interesting because they can be considered as model systems to study interaction processes and magnetic

reversal in low-dimensional magnetic structures.^{6,7} The key synthetic methodologies to get 1D magnetic nanostructures developed so far include template assisted electrodeposition,⁸ metallization of DNA,⁹ nanolithography,¹⁰ organometallic precursors decomposed in solution,¹¹ catalyzed high-temperature growth via the vapor–liquid–solid (VLS) mechanism,¹² solvothermal synthesis¹³ and direct electrochemical precipitation.¹⁴ However, it remains a big challenge to prepare uniform magnetic nanowires in high yield, and it is also difficult to assemble free-standing nanowires into functional devices. Recently, we have developed a low-cost and high-efficiency method to synthesize and assemble ferromagnetic metal nanofibers with diameters of about 20 nm and lengths longer than $100\ \mu\text{m}$ by employing the novel electrospinning technique.

Electrospinning is a simple and effective method for fabricating ultrathin nanofibers, either oriented or laid in a random fashion as a fibrous mat.^{15–19} In a typical electro-

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spinning setup, high voltage is applied to a droplet of polymer solution that rests on a sharp conducting tip. As a result of molecular ionization and charge redistribution, a Taylor cone is formed and a jet of the solution is extracted. The formed jet is then accelerated by the electric field and collected on a grounded substrate. When a volatile solvent is used, in-flight solvent evaporation occurs, hardening the fibers composed of the dissolved material, which are deposited on the substrate. With this technique, fibers have been electrospun from a wide variety of polymers.¹⁶ In addition to polymer nanofibers, ceramic, composite, and carbon nanofibers can be fabricated using this simple technique. For example, oxide nanofibers such as ZnO, TiO₂, NiO, and CuO have been prepared by calcinations of electrospun nanofibers containing polymer and inorganic precursor,¹⁷ and carbon nanofibers can be prepared using electrospun polyacrylonitrile nanofibers as a precursor.¹⁸ Electrospinning of nanofibers with magnetic properties has also met with some success. Some outstanding work has been performed to prepare oxide-based magnetic nanofibers of nickel ferrite (NiFe₂O₄).¹⁹ In this study, we report the first successful synthesis of ferromagnetic metal nanofibers by electrospinning. The process requires neither catalysts nor templates to yield continuous Fe, Co, and Ni nanofibers with diameters of about 20 nm. The nanofibers have novel magnetic properties. The magnetic nanofibers can also be easily assembled into well-aligned arrays. The outstanding features of this new method to get 1D magnetic nanostructure are its simplicity, convenience, and effectiveness.

2. Experimental Section

To synthesize Fe, Co, and Ni nanofibers, we first prepared Fe₂O₃, CoO, and NiO nanofibers by the combination of electrospinning and sol-gel process. To electrospin precursor fibers, we prepared an aqueous solution containing poly(vinyl acetate) (PVA) and metal nitrate. In a typical procedure, 0.25 g of PVA (Aldrich, weight-average molecular weight $M_w = 8000$) was mixed with 2.25 g of deionized water in a small bottle, followed by magnetic stirring for 1 h to ensure the dissolution of PVA. Metal nitrate (0.25 g; metal = Fe, Co, and Ni) was then added to the solution and stirred for 10 h. Using a peristaltic pump, the precursor solution was delivered to a needle made of stainless steel at a constant flow rate of 1.0 mL/hour. The needle was connected to a high-voltage power supply and positioned vertically on a clamp, with a piece of flat aluminum foil placed 20 cm from the tip of the needle to collect the nanofibers. Upon applying a high voltage of 20 kV, a fluid jet was ejected from the needle tip. The solvent evaporated and a charged fiber was deposited on to the collector. The collected fibers were calcined at 500 °C for 4 h in air followed by annealing at 400 °C for 1 h in a hydrogen atmosphere to obtain Fe, Co, and Ni nanofibers.

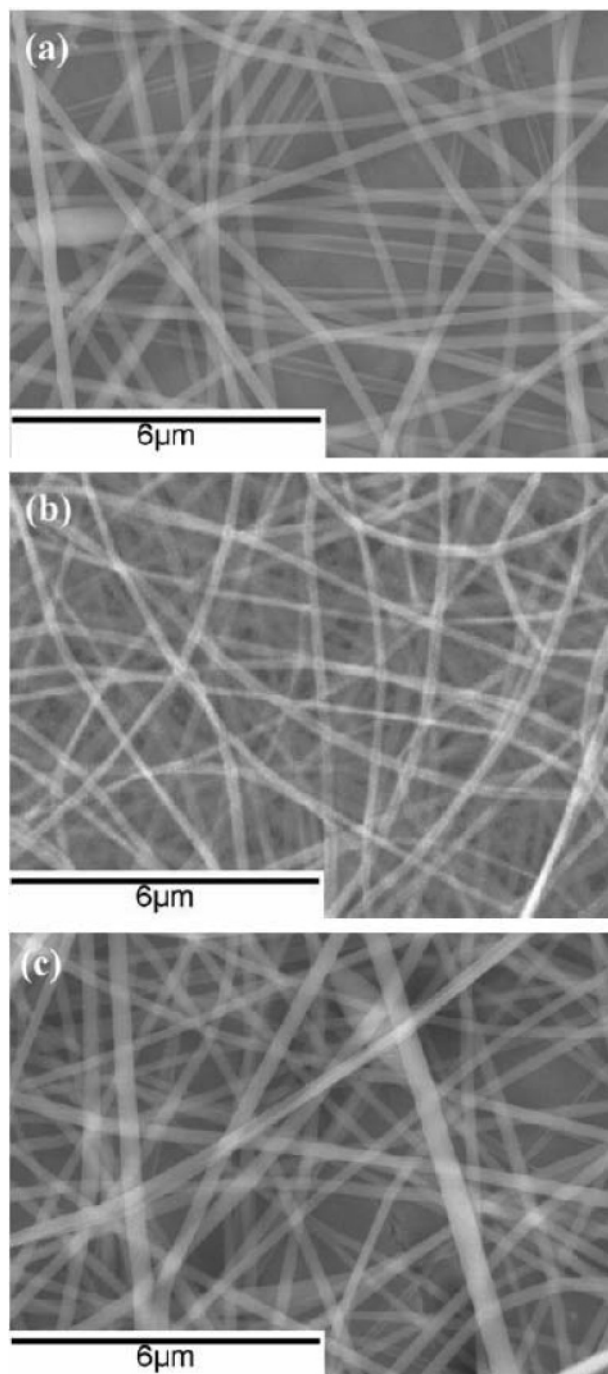


Figure 1. SEM images of precursor nanofibers: (a) Fe(NO₃)₃/PVA nanofibers, (b) Co(NO₃)₂/PVA nanofibers, and (c) Ni(NO₃)₂/PVA composite nanofibers.

For fabricated assembled nanofibers as uniaxially aligned arrays, a modified electrospinning setup was employed.^{15a} It is essentially the same as the conventional configuration except for the use of a collector containing a gap with a width of 1 mm in its middle. Such a collector was simply fabricated by putting two silver plates in a side-by-side parallel arrangement. Aligned arrays of the electrospun nanofibers can be collected on the gap; the fibers were then transferred onto a silicon wafer for further heat treatment. Single nanofibers of Fe, Co, and Ni suspending across the small gap can also be obtained with a short collection time of about 10 s.

The products were characterized by scanning electron microscopy (SEM, JEOL JSM-6301 F), transmission electron microscopy (TEM, JEOL JEM-2010), and X-ray diffraction (XRD, Regaku

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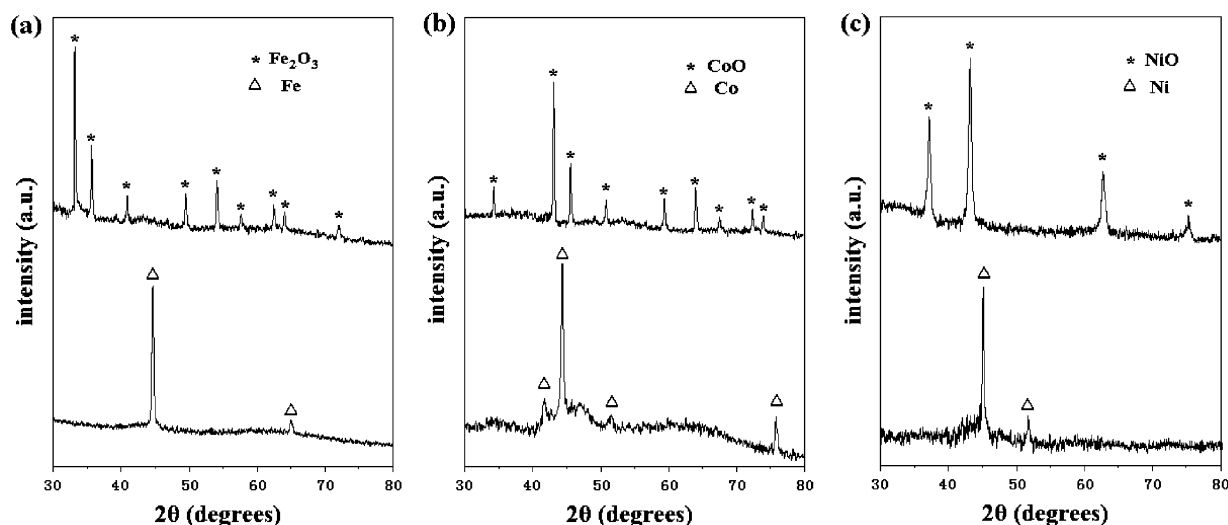


Figure 2. XRD patterns for (a) Fe_2O_3 and Fe nanofibers, (b) CoO and Co nanofibers, and (c) NiO and Ni nanofibers.

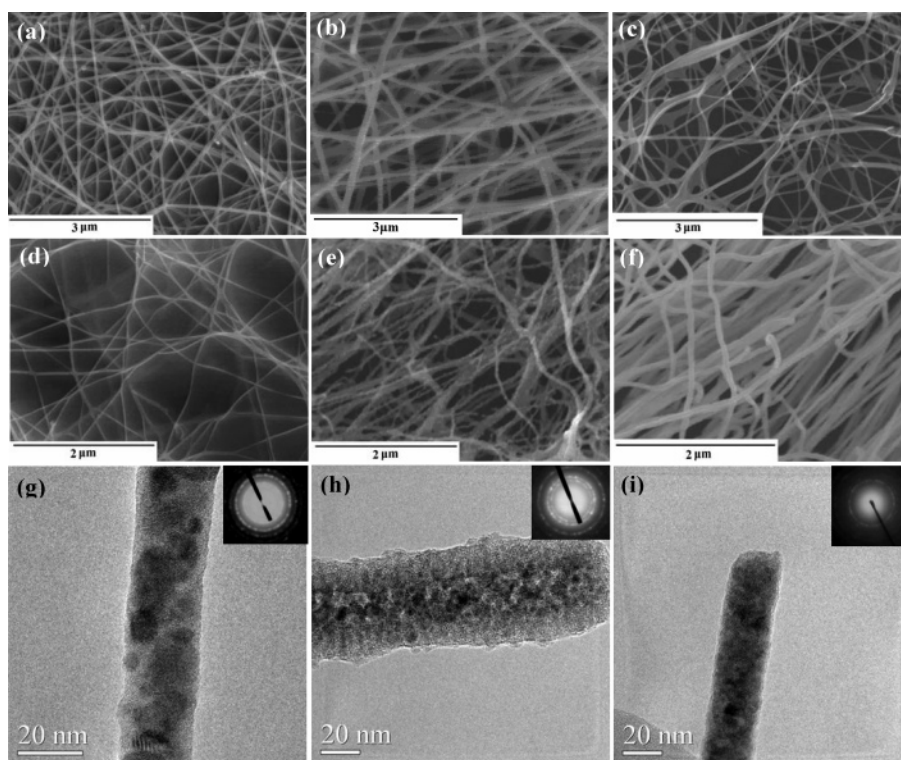


Figure 3. (a–c) SEM images of the prepared oxide nanofibers: (a) Fe_2O_3 nanofibers, (b) CoO nanofibers, (c) NiO nanofibers. (d–f): SEM images of synthesized Fe, Co, and Ni nanofibers: (d) Fe nanofibers, (e) Co nanofibers, (f) Ni nanofibers. (g–i): TEM images of the synthesized Fe, Co, and Ni nanofibers: (g) Fe, (h) Co, (i) Ni. The insets show the SAED patterns of the corresponding nanofiber.

D/max-R diffractometer using nickel-filtered $\text{CuK}\alpha$ radiation). Magnetic measurements at both room temperature (300 K) and low temperature (5 K) were carried out using a Lake Shore 7307 vibrating sample magnetometer (VSM). The measurements were performed on calcined electrospun nanofibers, which were collected on aluminum foil. Magnetization was measured as a function of the external field being swept up and down, with the applied magnetic field parallel (in-plane) to the nanofiber film surface.

3. Results and Discussion

From SEM images of the precursor nanofibers (Figure 1), it can be seen that the prepared $\text{Fe}(\text{NO}_3)_3/\text{PVA}$, $\text{Co}(\text{NO}_3)_2/\text{PVA}$, and $\text{Ni}(\text{NO}_3)_2/\text{PVA}$ composite fibers have a uniform

and smooth surface, with diameters of about 210, 180, and 220 nm, respectively. The precursor fibers were then heated to 500 °C under a heating rate of 10 °C/min and kept there for 4 h in an air atmosphere. As the PVA was selectively removed during the heating, the nanofibers remained as continuous structures, with reduced diameters of about 40, 54, and 50 nm (see Figure 3 for SEM images). The size reduction is due to the loss of PVA from the precursor fibers and the crystallization of the oxides. X-ray diffraction patterns (Figure 2a–c) show that the calcined nanofibers were polycrystalline Fe_2O_3 , CoO, and NiO, respectively.

Fe, Co, and Ni nanofibers could be obtained by very carefully deoxygenizing Fe_2O_3 , CoO, and NiO nanofibers

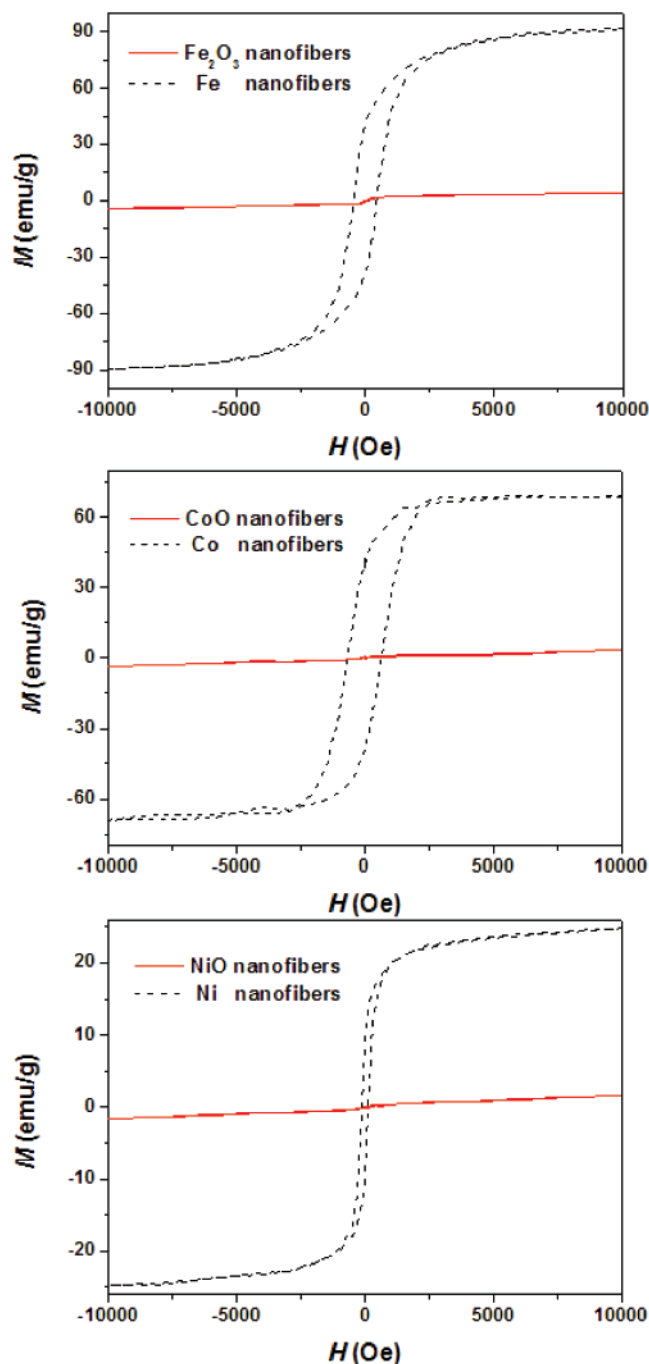


Figure 4. Magnetic properties of the electrospun nanofibers before and after being annealed in a hydrogen atmosphere: (a) magnetic hysteresis loops for Fe_2O_3 nanofibers (solid line) and Fe nanofibers (dashed); (b) CoO (solid) and Co nanofibers (dashed); (c) NiO (solid) and Ni nanofibers (dashed). The measurements were conducted at room temperature, with the applied magnetic field parallel (in-plane) to the nanofiber film surface.

in a hydrogen atmosphere at 400 °C for 1 h. The heating rate was 2 °C/min. XRD patterns clearly indicated the formation of pure Fe, Co, and Ni phases after heat treatment in hydrogen (Figure 2a–c). Figure 3d–f shows the SEM images of the synthesized Fe, Co, and Ni nanofibers. As observed, uniform nanofibers of Fe, Co, and Ni with the average diameters of about 25 nm (20 ± 3 nm for Fe, 30 ± 8 nm for Co, and 23 ± 5 nm for Ni nanofibers) were obtained. The decrease in the average nanofiber diameter (from ~ 50 nm (oxides) to ~ 25 nm (metals)) may be attributed to deoxygenization and shrinkage during heat

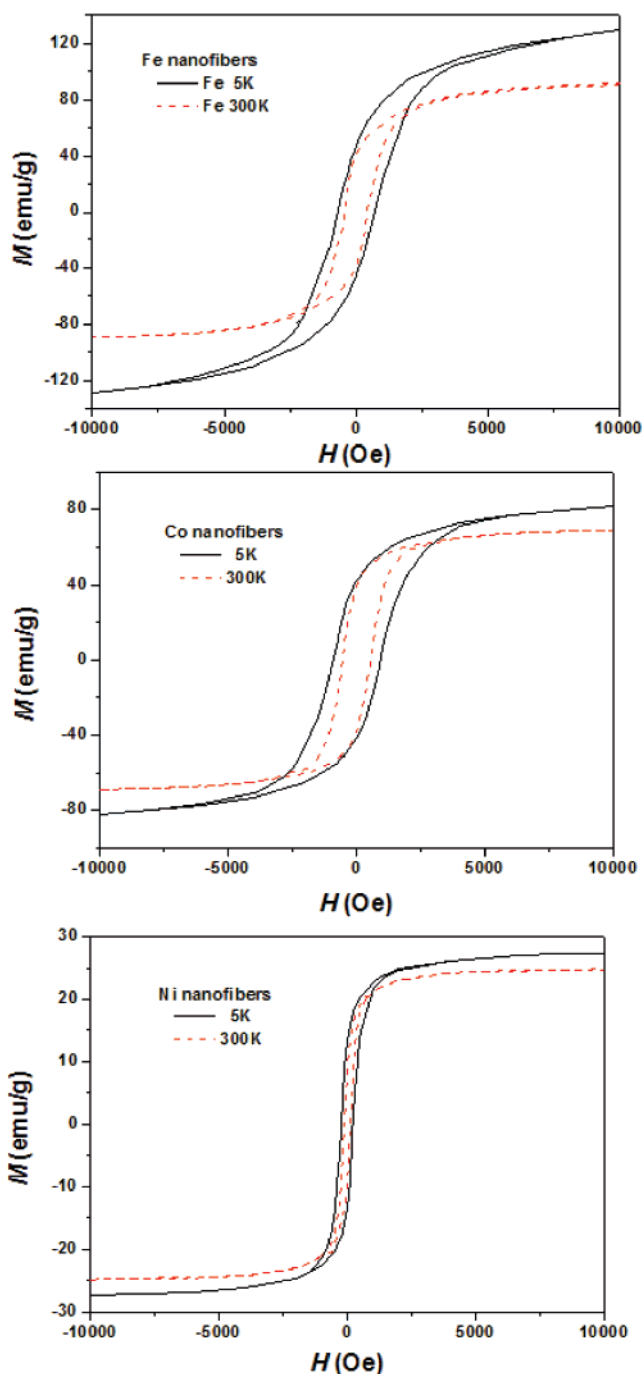


Figure 5. Hysteresis loops at room temperature (300 K) and 5 K. (a) Fe, (b) Co, and (c) Ni nanofibers.

treatment in hydrogen. The microstructures of the nanofibers were further investigated by TEM imaging. Figure 3g–i shows typical TEM images of single Fe, Co, and Ni nanofibers, from which smooth surfaces of the nanofibers were observed. The selected-area electron diffraction (SAED) patterns of the synthesized nanofibers (insets of Figure 3g–i) could be indexed as polycrystal Fe, Co, and Ni.

Magnetic properties of the synthesized nanofibers were characterized using vibrating sample magnetometer (VSM). Figure 4 shows the room-temperature magnetization hysteresis loops of (a) Fe_2O_3 , (b) CoO, and (c) NiO nanofibers and their corresponding metallic nanofibers. It can be seen that the hysteresis curves of Fe, Co, and Ni nanofibers revealed typical ferromagnetic behaviors, whereas the curves

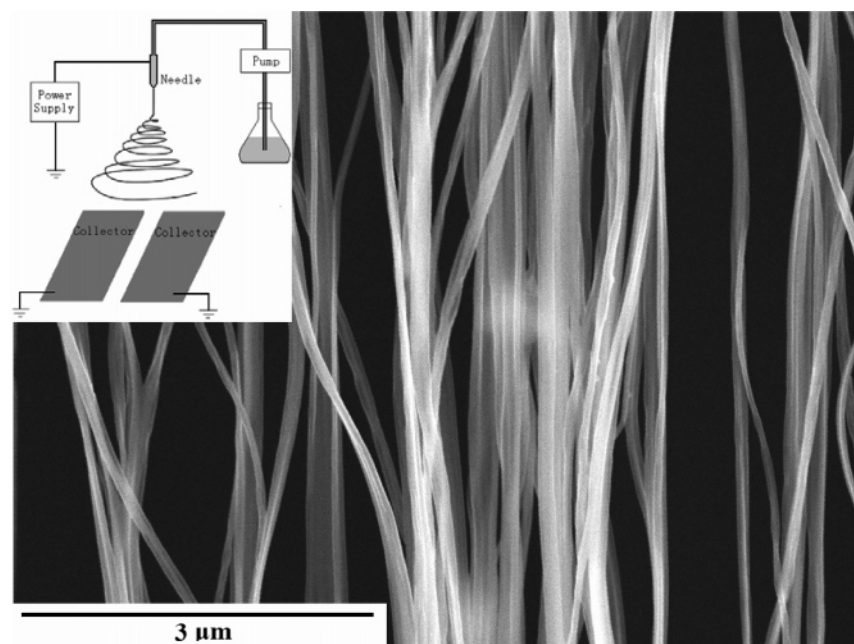


Figure 6. SEM image of a uniaxially aligned array of Ni nanofibers that were prepared using two paralleled electrodes as fiber collector. Inset is the schematic illustration of the setup for electrospinning that we used to generate uniaxially aligned nanofibers. The collector contained two pieces of conductive silicon stripes separated by a gap.

Table 1. Some Magnetic Properties of Synthesized Fe, Co, and Ni Nanofibers. (RT = 300 K)

| | nanofibers | | | | | | bulk materials | | |
|---------------|------------|--------|--------|--------|--------|--------|----------------------|----------------------|---------------------|
| | Fe(RT) | Co(RT) | Ni(RT) | Fe(5K) | Co(5K) | Ni(5K) | Fe(RT) | Co(RT) | Ni(RT) |
| H_c (Oe) | 427 | 651 | 124 | 702 | 907 | 228 | 1 ²² | 10 ²³ | 0.7 ²³ |
| M_s (emu/g) | 91.74 | 69.22 | 24.76 | 124.53 | 81.97 | 27.37 | 221.71 ²⁰ | 162.55 ²⁰ | 58.57 ²⁰ |
| M_r (emu/g) | 44.90 | 51.05 | 20.20 | 45.00 | 50.04 | 18.88 | | | |
| H_s (Oe) | 5000 | 2500 | 1500 | 10000 | 10000 | 5000 | | | |

of Fe₂O₃, CoO, and NiO nanofibers show nearly a flat line with little magnetization. The ferromagnetism of Fe, Co, and Ni nanofibers is clearly shown by coercivity (H_c), saturation magnetizations (M_s), remanent magnetization (M_r), and saturation field (H_s) listed in Table 1. The room-temperature saturation magnetizations of Fe, Co, and Ni nanofibers are 91.74, 69.22, and 24.76 emu/g, respectively, which are about half of the value of corresponding bulk metals (Fe, 221.71 emu/g; Co, 162.55 emu/g; and Ni, 58.57 emu/g,²⁰ in Table 1). A saturation magnetization lower than that for bulk materials is normal for nanomaterials.²¹ Typical reasons for this include the oxidation of the surface of magnetic nanofibers, which may create a magnetically dead layer. The large specific area and the imperfection of the crystalline structure at the surface may also lead to a significant decrease in the nanofiber saturation magnetization.

It is important to note that the synthesized magnetic metallic nanofibers exhibit great enhanced coercivities, which are about 2 orders of magnitude higher than that of bulk materials. The coercive fields obtained in the Fe, Co, and Ni nanofibers was 427, 651, and 124 Oe; whereas that of bulk Fe, Co, and Ni is only 1, 10, and 0.7 Oe, respec-

tively.^{22,23} The enhancement of coercivity may be attributed to the single-domain nature and anisotropic shape of the nanowires.²⁴ A higher coercivity is a key factor for information storage, although real devices also require intensive studies on magnetization interaction, local magnetization reversal characterization, and other important issues. Nevertheless, such novel magnetic nanofibers are of potential interested for high-density information storage application. Additionally, because the magnetic coercivities of the nanofibers are outstanding and the synthetic method is low-cost and highly efficient, bulk amounts of randomly oriented ferromagnetic metal nanofibers could also be used for manufacturing flexible magnets after blending different loads with polymers.

The low-temperature magnetic properties of Fe, Co, and Ni nanofibers were also examined. Figure 5 show the hysteresis curves of Fe, Co, and Ni nanofibers at 5 K, from which increased H_c values of 702, 907, and 228 Oe were observed (Table 1). Similar results were obtained from ferromagnetic metal nanowires prepared by the template-assisted electrodeposition method.^{8,13,25} The larger coercivity

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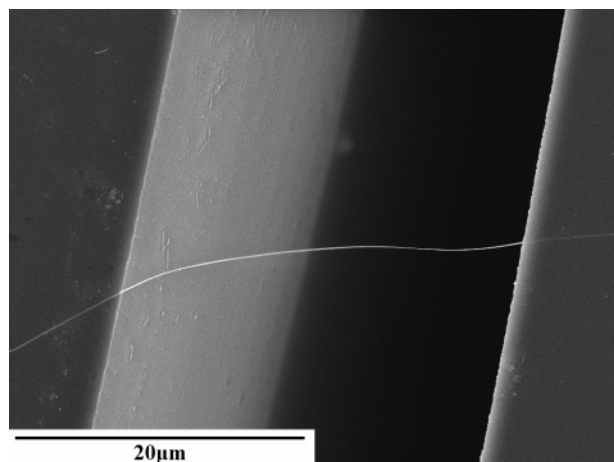


Figure 7. SEM image of a single Ni nanofiber suspended across a 25 μm gap. The fiber was collected within a short-collecting time of about 10 s during the electrospinning process.

at low temperature for the nanofibers may be explained by considering the reduced influence of thermal fluctuation on the rotation of magnetic dipoles. As a result, an increased amount of energy (or higher magnetic field strength) was required to change the magnetization direction of these aligned dipoles, and a larger coercivity of the nanofibers can be observed. In addition, the saturation magnetization (M_s) increases a lot as temperature goes down for Fe, Co, and Ni nanofibers. The higher M_s may be attributed to a strong increase in the local surface anisotropy at 5 K with surface spins frozen along the easy axis. When the applied fields are high enough, the surface anisotropy is overcome and the surface spins tend to align along the field direction, which results in a higher M_s value.

The preparation of aligned 1D magnetic nanostructures, rather than randomly oriented configurations, is of great interest for functional devices in nanoscale, especially for high-density information storage devices. Oriented ferromagnetic metal nanofibers can be prepared by employing a specially designed electrode as the fiber collector. The inset

of Figure 1a illustrates the schematic setup used for fabricating aligned nanofibers. It is essentially the same as the conventional configuration except for the use of a collector containing a gap in its middle. Such a collector was fabricated by putting two stripes of conductive silicon in a side-by-side parallel arrangement. The stripes were grounded using thin copper wire. During the electrospinning process, the electrostatic force drives the positively charged fibers aligning between the two counter electrodes.^{15a} Figure 6 shows a SEM image of an oriented Ni nanofiber array, which was collected within 10 min. The density of the fiber array depends on the collecting time. Within a long collecting time of several minutes, it is easy to obtain a dense fiber array, whereas within a very short collecting time of several seconds, only a single fiber with controlled orientation can be collected. Figure 7 shows a SEM image of a single Ni nanofiber bridging two silicon wafers with a 25 μm gap, the fiber was collected in a short time of about 10 s. Aligned fiber arrays or single nanofiber of Fe and Co can also be prepared using same method. These oriented magnetic nanofibers might be useful in assembling ordered magnetic circuits instead of using expensive electron-beam lithography and have potential applications in data storage.⁶

4. Conclusion

In conclusion, we have developed a new strategy for the facile synthesis of ferromagnetic transition metal nanofibers. Uniform nanofibers of Fe, Co, and Ni were prepared by electrospinning and subsequent heat treatment. The synthesized nanofibers show ferromagnetic properties with enhanced coercivities. Aligned arrays of the metallic nanofibers, as well as oriented single nanofibers, can also be prepared using a modified fiber collector. By adjusting a number of fabrication parameters, the diameter of nanofibers may be further varied in a controllable fashion, and the magnetic properties of the nanofibers may be further controlled and fine-tuned using these means. In addition, this synthetic route can be extended to nanofibers of various types of FeCoNi alloy as well. These novel magnetic nanofibers can potentially be used in fabrication of high-density magnetic recording, magnetic sensors, flexible magnets, and spintronic devices.

Acknowledgment. This study was supported by the National Natural Science Foundation of China (Grant 50572042).

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