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TITLE:

Iron Nanowire Fabrication by Nano-Porous Anodized Aluminum and its Characterization

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KEYWORDS:

nanotechnology, nanofabrication, iron nanowires, anodization, electrodeposition, membranes

SUMMARY:

In this work, we describe a complete protocol to fabricate iron nanowires, including the formation of the porous alumina membrane that is used as the template, electrodeposition into templates using electrolyte solution, and the release of the nanowires into the solution.

ABSTRACT:

Magnetic nanowires possess unique properties that have attracted the interest of different fields of research, including basic physics, biomedicine, and data storage. We demonstrate a reproducible fabrication method for iron (Fe) nanowires via one-step anodization of aluminum (Al) discs. Using anodic alumina oxide (AAO) templates, cylindrical nanowires are synthesized in a bottom-up fashion by an electrochemical deposition method. The pore length and diameter are controlled by changing the anodizing conditions. The average diameter achieved for nanowires is around 120 nm using oxalic acid as the electrolyte. Nanowires are released by dissolving the alumina using a selective chemical etchant. The nanowire dimensions and structural variations are observed using a scanning electron microscope (SEM) to confirm that nanowires are high quality and specific properties are obtained.

INTRODUCTION:

Cylindrical magnetic nanowires have attracted an enormous amount of interest in the last decade for a variety of promising applications. Nanowires are novel materials that possess unique properties, mainly due to a high aspect ratio and shape anisotropy[1]. Because of these properties, nanowires are considered unique systems and excellent model objects for a number of practical

applications: flow sensors[2], magnetic separation[3], bio-inspired tactile sensors[4], energy harvesting[5], cancer treatments[2,6], drug delivery[7,8], and MRI contrast agents[3,9]. Nanowires are also considered ideal for other applications: magnetic force microscopy[10], giant magnetoresistance[11], spin transfer torque[12,13], and data storage devices[14,15].

In order to exploit these nanowires to their full advantage, a reproducible fabrication method that yields nanowires of high quality and specific properties is required. The anodization of aluminum produces self-organized, highly ordered cylindrical pores with controllable pore diameters. Because of this, AAO templates are preferred in nanotechnology applications over expensive lithographic techniques. Using these membranes as scaffolds, nanowires can be created by direct current (DC), alternating current (AC), or pulsed DC electrodeposition. Controlling the fabrication process of the membrane and the deposition of the nanowires, a wide range of magnetic nanowires can be created for particular applications[1]. Here, we report the fabrication of Fe nanowires, including the formation of the porous alumina membrane that is used as the template, electrodeposition into templates using electrolyte solution, and the release of the nanowires into the solution.

PROTOCOL:

CAUTION: Please consult all relevant material safety data sheets (MSDS) before use. Several of the chemicals used in these fabrications are acutely toxic and carcinogenic. Nanomaterials may pose additional hazards compared to their bulk counterparts. Please use all appropriate safety practices when performing a nanocrystal reaction, including the use of engineering controls (fume hood) and personal protective equipment (safety glasses, gloves, lab coat, full length pants, closed-toe shoes).

1. Preparations of aluminum templates

1.1. Cleaning the aluminum discs

1.1.1. Wash the Al discs in a beaker with deionized (DI) water. Repeat 3 times.

1.1.2. Hold the Al disc with tweezers and wash with acetone followed by isopropyl alcohol (IPA) and DI water.

1.1.3. Place the Al discs in a beaker with acetone and sonicate for 10 min.

1.2. Electropolishing of aluminum discs

1.2.1. Prepare the electropolishing solution, 3 M perchloric acid in ethanol. Cool the electropolishing solution in a fridge at 4 °C before use.

1.2.2. Wash the Al discs in a beaker with DI water. Repeat 3 times.

1.2.3. Grip the cleaned Al template with the dressing forceps and immerse it inside the beaker filled with electropolishing solution along with the platinum (Pt) mesh electrode. Keep the forceps out of the solution as much as possible.

1.2.4. Stir the solution at 400 rpm.

1.2.5. Connect the Al disc to the positive terminal and Pt to the negative terminal of the power supply. Apply a voltage of 20 V while the current is limited to 2 A.

1.2.6. Polish the discs for 3 min and wash the discs with DI water.

2. Hard anodization

2.1. Preparing the cells

2.1.1. Wash the cell parts (copper plate, PDMS/rubber O rings, cell, Pt mesh cap) with DI water.

2.1.2. Take the electropolished Al discs out of the DI water and place it on the cell holes with O-rings. Check carefully that there are no leaks.

2.2. Anodization

2.2.1. Fill the assembled cell with 0.3 M oxalic acid and place it on the cold plate at 4 °C.

2.2.2. Once the oxalic acid is between 2-5 °C, apply 40 V for 20 min (mild anodization). Then, increase the voltage in steps of 0.1 V/s up to 140 V.

2.2.3. Keep this voltage constant for 45 min. The anodized template will be a bright golden color.

2.2.4. Open the cell and wash the Al disc with DI water and dry with nitrogen (N_2).

3. Preparation for deposition

3.1. Removal of Al back

3.1.1. Prepare a copper solution with 0.1 M of $CuCl_2 \cdot 2H_2O$ and 6 M of HCl.

3.1.2. Place the anodized template in a cell (with a 10 mm hole diameter) with the back side facing upwards.

3.1.3. Pour the copper solution and a magnetic stirrer into the cell and agitate at 300 rpm.

3.1.4. After around 15 min, the solution becomes transparent. Replace it with fresh solution and agitate for 5 min more.

3.1.5. Wash the discs with DI water and dry with N_2.

3.2. **Opening the pores**

3.2.1. Place the sample (back side facing upwards) in a Petri dish on a pH strip.

3.2.2. Deposit 10 wt% phosphoric acid to completely cover the membrane. Add more phosphoric acid every hour to avoid dryness.

3.2.3. After 6.5 h, wash with DI water, and dry with N_2.

3.3. **Gold sputtering**

3.3.1. Prepare the sputtering machine. Open the inert gas valve and vent the chamber.

3.3.2. Tape the Al disc onto the sputter stage with the back side facing up.

3.3.3. Adjust parameters to deposit 200 nm and run the profile.

4. Deposition of nanowires

4.1. Prepare a solution of 0.2 M of iron (II) sulfate, 0.16 M of boric acid and 0.05 M of L-ascorbic acid.

4.2. Mount the Al membrane into the cell (15 mm diameter hole)

4.3. Pour the solution into the cell and connect the source meter with the positive contact attached to the copper plate and the negative contact to the platinum mesh.

4.4. Apply a constant current of 2.5 mA to start electrodeposition. The length of the nanowire is directly proportional to the electrodeposition time.

5. Membrane removal and washing of nanowires

5.1. **Gold etching**

5.1.1. Break the membrane using a tweezer. Select small pieces (around 1 or 2 mm^2).

5.1.2. Prepare one or more small pieces for dry etching using reactive ion etching (RIE) equipment. Glue the pieces to a dummy wafer using lubricant, keeping the gold face up.

5.1.3. Etch the gold in the RIE equipment for 2 min using the following parameters: T = 25 °C, P = 150 W and argon flow rate = 25 cm^3/min. Repeat in shorter cycles if some gold is still present.

5.2. **Nanowire release**

5.2.1. Prepare the chrome solution using 0.2 M of CrO_3 and 0.5 M of H_3PO_4.

5.2.2. Fill a 1.5 mL microtube tube with 1 mL of the chrome solution and the small pieces of membrane containing nanowires.

5.2.3. Leave the solution working for 24 h at 40 °C.

5.2.4. When the nanowires are completely released, no black particles should be observed with the naked eye.

5.2.5. Wash the nanowires by placing the microtube in a magnetic rack and replacing the chrome solution with 1 mL of ethanol.

5.2.6. Repeat the washing process at least 10 times.

REPRESENTATIVE RESULTS:

After electropolishing, the Al disks reflect light well, as seen in **Figure 1**. If any small scratches or dots are observed, discard the disk. The plot of the applied current during the anodization process should be smooth and follow the three steps of anodization. In case of contaminated solution, excessive defects on the disk surface, incorrect preparation of the cell (see **Figure 2**), or the solution being too warm, the applied current plot curves will show peaks and irregularities. Two actual anodization curves are shown in **Figure 3**, including pictures of the samples. Anodization takes place on one side of the Al disk (top side). After removing the Al back, the membrane should be clearly visible from both sides. The pore opening can be checked using scanning electron microscopy (SEM) on the bottom side. **Figure 4** shows a sample in which the pores were not completely opened. The deposition rate of Fe nanowires for membranes of this size is around 300 nm/min. As an example, Fe nanowire of around 1 µm is shown in **Figure 5**. Note that this image was taken after breaking the membrane.

FIGURE AND TABLE LEGENDS:

Figure 1: Aluminum disks. Before polishing (left) and after polishing (right). Marks on top of the polished disk are caused by the forceps.

Figure 2: Anodization cell. (**A**) Components of the cell. (**B**) Detail of the Al disk positioned over the PDMS O-ring. (**C**) Cell assembled. (**D**) Cell located over the cold plate and with the mechanical stirrer.

Figure 3: Applied current versus time during anodization for a successful (left) and unsuccessful (right) anodization. The three steps of anodization can be easily recognized. The stable 40 V (0–20 min); the constant increase up to 140 V (20–36:40 min), shown first as an increase of applied

current and later as a constant current; and third, the stable 145 V until the end of the process. When anodization occurs properly, curves are smooth like the one on the left. When the curves show peaks or chaotic behavior (right) the sample would be burnt. In this case, the Al disk diameter was 25 mm.

Figure 4: SEM image of a membrane from the bottom side. This image shows the morphology of a membrane next to its edge. At any other point of the membrane, the membrane shows open pores like the ones in the picture. If the pores are not open properly, the hexagonal structure that is shown at the edge of the picture would be visible anywhere in the membrane.

Figure 5: Cross section SEM image of iron nanowires inside the membrane. The Fe nanowire is clearly recognizable from the alumina membrane due to its higher electron density.

DISCUSSION:

As in any other nanomaterial production, high-quality solutions and materials are required in this protocol. Electropolishing and electrodepositing solutions can be reused several times. However, the anodization solution should only be used once and be freshly made. After removing the Al back, the membranes are extremely weak and can be broken if not handled carefully. The N_2 should not be directly applied when drying the membranes. All processes prior to anodization are equally important for the self-ordering of pore structures. Surface impurities, pits, and scratches may lead to poorly ordered nanopores.

The thickness of the alumina membrane generated in step 2 is usually around 60 μm, much longer than the nanowire we require. If longer nanowires are needed, this protocol can be adapted to make thicker membranes by increasing the time of anodization. These nanopores can be used as templates for forming arrays of standing nanowires or released by a subsequent chemical removal of alumina structure. Furthermore, different metals can be electrodeposited using the same setup, including multisegmented nanowires[15], by changing the solution and the applied current. Rate deposition would be different for each metal.

The main advantage of the anodization method presented is the high quality of the pores: constant diameter along tenths of micrometers, small diameter distribution, and high pore density. Further, this technique is efficient, economical, and highly reproducible. It can be done safely at ambient conditions in the general laboratory. Nanowires promise a lot in future energy conversion devices (including photovoltaics, thermoelectrics, and betavoltaics[16]) and as biological and medical sensors[17]. All of these applications will require extensive material and device development.

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DISCLOSURES:

The authors have nothing to disclose.

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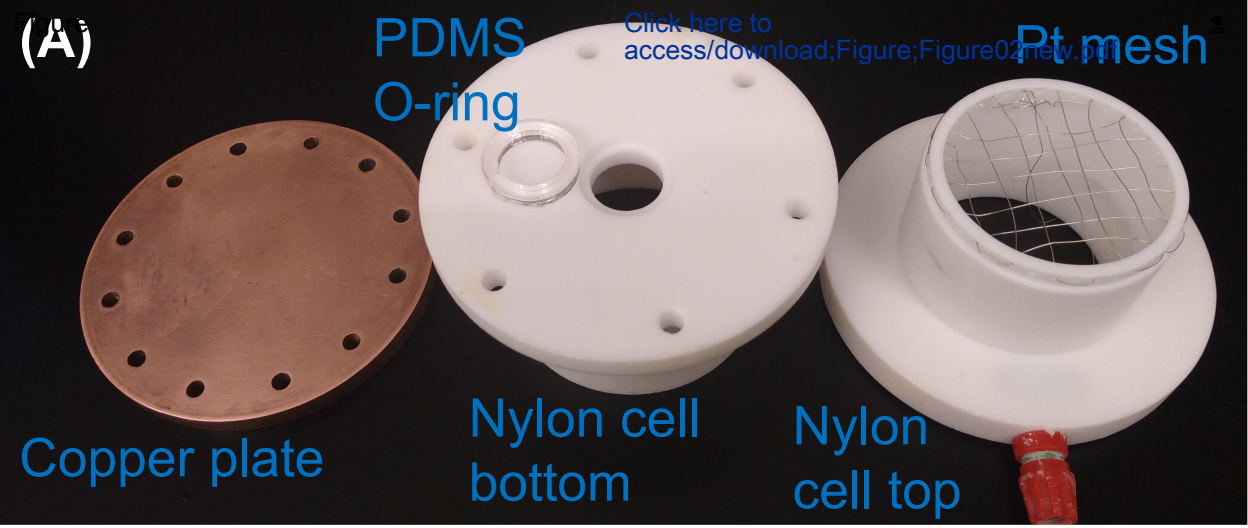
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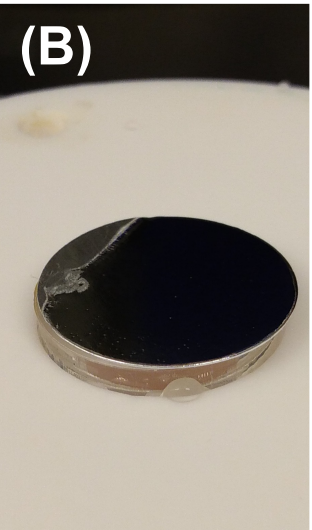
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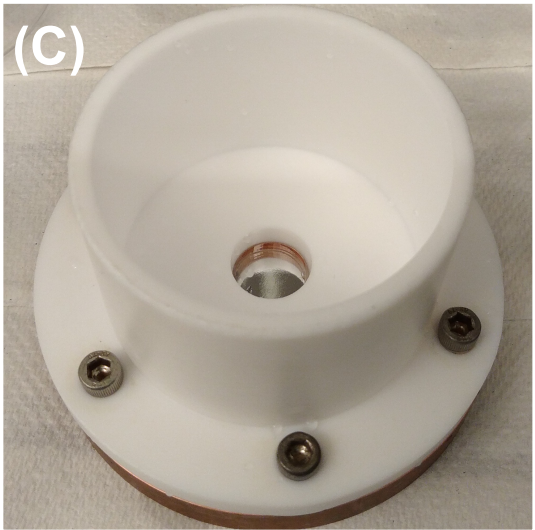
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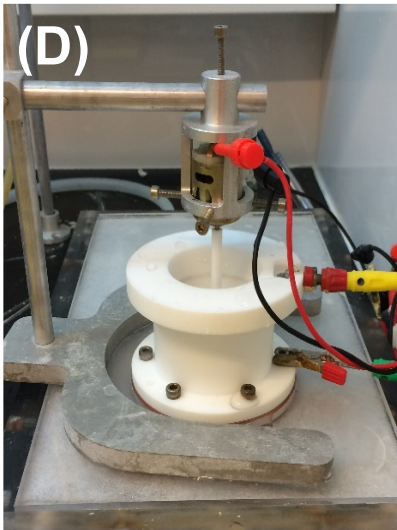
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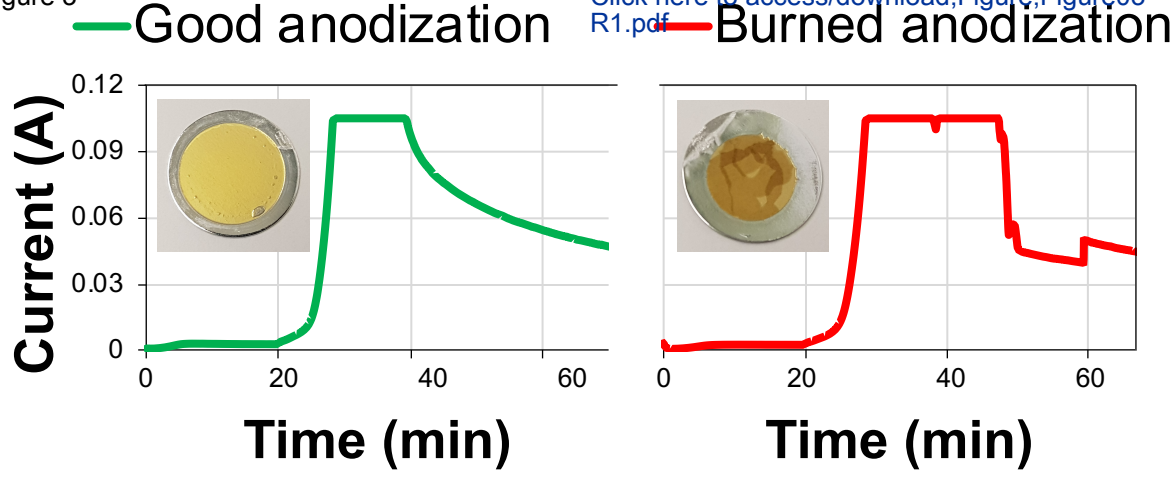
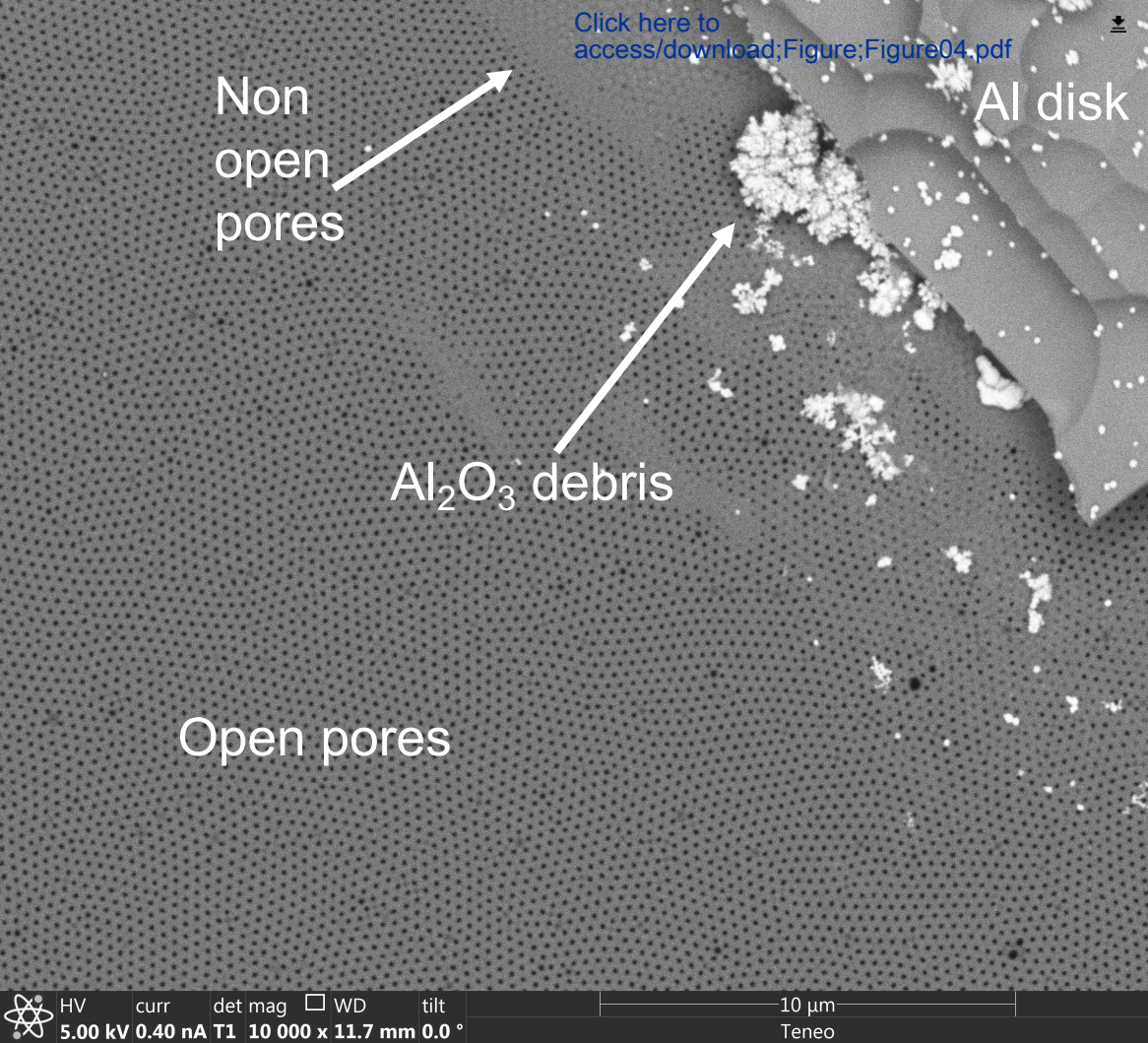
Figure 3

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Non open pores
Al disk
Al_2O_3 debris
Open pores
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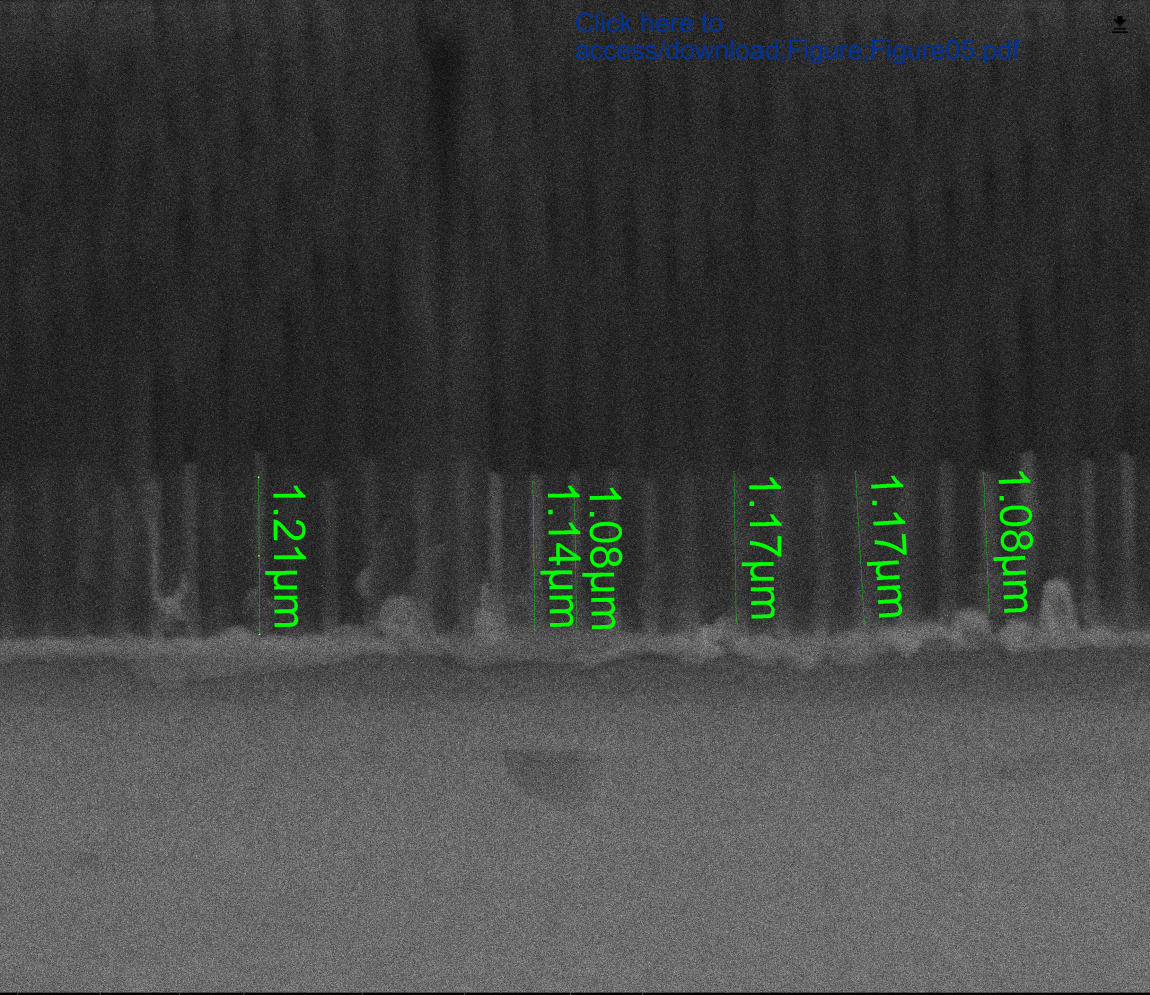
Teneo



curr	mode	det	mag	WD	HV	dwell	3 µm
22 pA	SE	ETD	28 830 x	5.0 mm	15.0 kV	2 µs	Nova Nano

Name of Material/ Equipment	Company	Catalog Number	Comments/Description
2-PROPANOL	BASF	50489068	
Acetone	Sigma Aldrich	CAS 67-64-1	
Aluminium Discs 99.999%	GoodFellow	AL000957	Thickness: 0.50mm +/- 10%, Diameter 25.0mm +/- 0.5m
Big Beaker			1000 mL
Boric acid	Sigma Aldrich	101942058	99%
Cables			
Chromium (VI) oxide	fisher chemical	A98-212	
Cold plate	Thermo Scientific	Accel 500 LC	
Computer			Used with LabView to control the Sourcemeter
Copper (II) chloride			
Copper plate			Custom made
DC Power Source	Agilent	E3646A	
DI Water			
Dressing Forceps	fisher scientific	12-460-164	30.5 cm length, serrated tips
Ethanol	VWR International Ltd. (US)	20823.327	
Fume hood	Flores valles		
Hydrochloric acid	VWR International Ltd. (US)	20255.290	
Iron (II) sulfate	Merck	1.03965.1000	
L-Ascorbic acid	MP biomedicals	100769	
Magnetic rack	life technologies	DynaMag 2	
Magnetic stirrer and hot plate	IKA	RCT basic	
Mechanical stirrer	Aslong	JGB37-520	
Mixer and heater	Eppendorf	ThermoMixer F1.5	
Nylon cell			Custum made
Oxalic Acid	VWR International Ltd. (US)	20063.365-5L	

PDMS O-ring			Custom made
Perchloric acid	VWR International Ltd. (US)	20583.327	70-72 %
Petri dish			Or any other container
pH strip			Any pH strip
Phosphoric acid	acros organics	201140010	85%wt
Platinum	Goodfellow	PT005115	Diameter 0.05mm, 99.9% purity
Platinum wire	Goodfellow	PT05120	Diameter: 0.2 mm, Purity: 99.95%
Power Supply	Rhode & Scharz	NGPX 35/10	
Retort stand (x2)			
Screws			
Small beaker			50 mL
Source meter	Keithley	2400-C	
Sputter	Quorum	Q300T D	
Tape			Any temperature resistant tape
Teflon propeller			
Ultrasonic cleaner			

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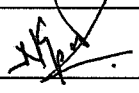
Signature:	
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Dear Editor,

We thank you for your comments. We have taken then into account as explained below.

1. Please take this opportunity to thoroughly proofread the manuscript to ensure that there are no spelling or grammar issues.

Typos corrected:
Line 51, produce → produces
Line 237, anodizations → anodization

2. Figure 1 was not cited in the manuscript.

It was originally cited in the Protocol section, now it is cited in Representative Results. As now it is cited after former Figure 2, we have changed their numeration. Added text in Line 210: *"…incorrect preparation of the cell (see Figure 2)…"*

3. Please sort the items in alphabetical order according to the name of material/equipment.

List of items has been sorted alphabetically. Also, item name "*2 retort stands*" has been changed to *"Retort stand (x2)".*

4. There are additional comments marked in the manuscript. Please check and revise the manuscript accordingly.

Both typos have been corrected:

[1] → ^{1}
m → µm

5. There are still a few sections of the manuscript that show significant overlap with previously published work. Please rewrite lines 53-57.

This part of the text has been modified:

"Using these membranes as scaffold, nanowires can be created by direct current (DC), alternating current (AC) or pulsed DC Electrodeposition. Controlling the fabrication process of the membrane and the deposition of the nanowires, a wide range of magnetic nanowires can be created for particular applications."