

Synthesis of Nickel Nanowires

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Purpose:

Synthesize nickel nanowires via electrolysis.

Learning objectives:

- 1) Describe electrolysis
- 2) Identify the anode and cathode
- 3) Apply knowledge of electrolysis to a particular reaction to identify which parts are being oxidized and reduced.

Introduction:

Electrochemistry is the study of chemical reactions that occur between a conductor and an electrolyte in solution. This process involves electrolysis. In electrolysis there is a conductive cathode and an anode electrode in solution. Electrolysis involves the passing of an electrical current through the solution where a reaction then occurs at the electrodes. During electrolysis the positively charged cations move towards the cathode and the negatively charge anions move towards the anode. At the cathode a reduction reaction is occurring and at the anode an oxidation reaction is occurring.

In this experiment Nickel nanowires can be synthesized in a membrane. The membrane is coated with GaIn alloy which creates a conductive surface against a copper electrode. A nickel electrode is also added. The synthesis of the nickel nanowires takes place in a nickel plating solution. Below is a schematic of the electrodeposits of nickel into the membrane filter.

Materials:

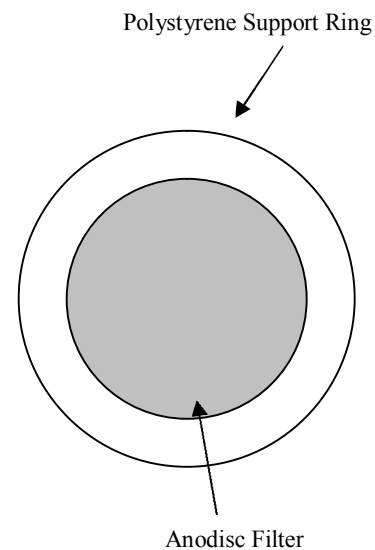
- Whatman Anodisc alumina filters with polypropylene support rings and 0.02 micrometer pores.
- GaIn Eutectic
- Nickel plating solution
"Watts Ni Pure," #130859, \$42.25/gallon
- 10 cm Ni wire
- Copper sheet
- 2 alligator clip wires/ with alligator clips on both ends
- battery holder
- Electrical tape
- 1 AA battery
- 6 M NaOH
- Concentrated nitric acid
- Microscope glass slide

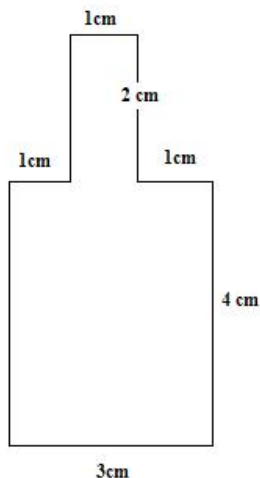
Procedure:

Nickel nanowires can be made using two different methods—called the beaker or the syringe methods. The beaker method uses less equipment but requires some skill in taping the filters down without breaking them. This procedure will use the beaker method because it has the simplest set up that needs to be made.

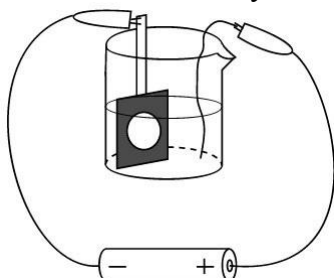
A) Synthesis of Nickel Nanowires

- 1) Obtain a 0.02 micrometer Anodisc filter.
 - a. NOTES
 - i. Remember which side was facing upward in the box
 - ii. Always carry by the support ring and a pair of forceps
 - iii. Discs are very brittle
- 2) Fully coat the upper side of the filter with GaIn alloy.
 - a. Dip cotton applicator in the GaIn only once, and spread it thin on the membrane.
 - b. The support ring does not need to be painted
 - c. Any areas without GaIn will appear blue on the underside and not the opaque white that should be observed
- 3) Obtain a copper slide with these dimensions.





- 4) Place the filter face down (shiny side down) on top of the clean satin finish side of the cut out copper sheet.
- 5) Use electrical tape to fasten the disc to the copper sheet.
 - a. Fasten the disc with tape as shown below.
 - b. Only tape the support ring and not the filter
 - c. Also tape the entire back side of the copper electrode, so that none of the electrolysis solution comes in contact with the copper electrode.
- 6) Place the copper electrode into a 50 ml beaker. Pour in the nickel solution until it covers the disk.
- 7) Make a circuit as shown below with or without an ammeter. The positive wire attaches to a nickel wire placed in the electrolysis solution. The negative wire attaches to the copper electrode.
 - a. Clip one end of two different the alligator wires to the end of the battery holder. Use a 1.5 V(AA) battery for the electrolysis of the nickel.
 - b. Note: make the nickel wire long enough so that the alligator clip can be out of the electrolysis solution.



<http://mrsec.wisc.edu/Edetc/nanolab/nickel/index.html>

- 8) Allow the electrolysis to run for 10-50min.
 - a. The longer it runs the longer the nanowires are.
- 9) After the electrolysis time is finished remove the battery from the system.
- 10) The nickel solution can be reused
- 11) Rinse the electrode with distilled water
- 12) To remove the tape immerse the electrode in a beaker of acetone for about 15 min until all of the adhesive is removed

- 13) Remove the disc and tape it shiny side up onto the end of a microscope glass slide.
 - a. Use concentrated Nitric acid to remove the GaIn from the filter.
 - b. Ensure all of the GaIn is removed before proceeding.
- 14) Rinse completely and carefully with distilled water.
- 15) Place glass slide into a 20 ml beaker and add about 15 ml of 6 M NaOH for 10 minutes. Make sure the NaOH is covering the filter disc on the glass slide.
- 16) With tweezers remove the poly support ring. (The rest will dissolve in the NaOH).
- 17) Place a magnet under the beaker and pour off the NaOH.
- 18) Rinse with water.
 - a. Before pouring, ensure that most of the nanowires have fallen down to the bottom of the beaker.
 - b. Repeat the rinse several times with water to ensure all of the NaOH is out of the solution.
- 19) If finished with the lab at this point transfer the nanowires suspended in water to a 3-20ml vial.

B) Analysis of Nanowires

- 1) With 10 ml of water in the beaker containing the nanowires, take the cow magnet and place it parallel to the beaker at a distance of 5 to 10 cm.
- 2) What is observed?

- 3) Place the magnet perpendicular to the beaker at the same distance. What is observed?

- 4) Depending on vial size available use the magnet on the bottom of the beaker to pour off any excess liquid so that the nanowires can fit into the vial.
- 5) Transfer the final suspension to a 3-20 ml vial for storage
- 6) Move the magnet around the vial to manipulate the nanowires. Write down any observations that can be seen

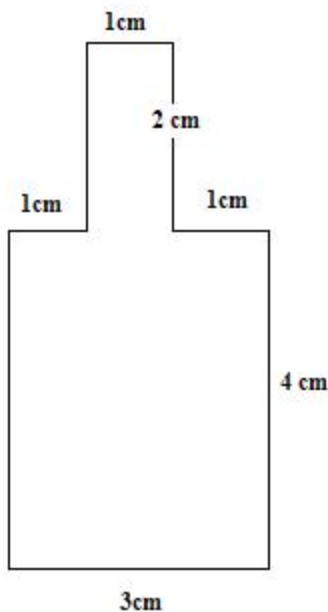
- 7) Mix the vial up so the nanowires are suspended in the water. Take a glass pipette and transfer the nanowires onto a slide. Use a depression slide if available.
- 8) Place a coverslip on top gently so that there is still water under the cover slip.
- 9) Place the slide under a microscope.
- 10) View the slide and bring it into focus.
- 11) Place a magnet on one side of the slide. Then move it around the slide
- 12) Write down any interesting observations about the slide.

Questions:

- 1) Describe electrolysis?
- 2) Identify the anode and cathode in this experiment?
- 3) What is being oxidized and what is being reduced.
- 4) Why doesn't the concentration of the Nickel plating solution change throughout the course of the experiment?

Teacher materials and preparations:

- Whatman Anodisc alumina filters with polypropylene support rings and 0.02 micrometer pores (not available directly from Whatman)
 - Fisher Scientific, #09-926-34, pack of 50 is \$97.60
 - VWR International, #28138-067, pack of 50 is \$94.80
- GaIn Eutectic
 - Aldrich, 49542-5, \$38.30 for 5g
 - To prevent waste of the mater possible prepare onto the alumina filters prior to lab.
- Nickel plating solution
 - "Watts Ni Pure," #130859, \$42.25/gallon
 - Technic, Inc., 1 Spectacle St, Cranston, Rhode Island 02910
 - Tel: 401-781-6100, Fax: 401-781-2890, Email: info@technic.com
- Ni wire, cut into 10 cm strips.
 - VWR International, #AA41361-G6, Alfa Aesar 1 mm diameter x 10 m long, 99.5%, \$37.90
 - CAUTION: Avoid physical contact (especially inhalation) with nickel and nickel solutions as nickel is an irritant and [carcinogen](#).
- Copper sheet
 - 0.032 inch thick satin finish copper sheet, 12" x 12", McMaster-Carr, #9801K11, <http://www.mcmaster.com>, \$30 –
 - Cut out a copper slide with these dimensions prior to lab.



- 2 alligator clip wires/ with alligator clips on both ends
- battery holder
- 1 AA battery

References:

- A. K. Bentley, M. Farhoud, A. B. Ellis, G. C. Lisensky, Anne-Marie Nickel, and W.C. Crone, "Template Synthesis and Magnetic Manipulation of Nickel Nanowires," *Journal of Chemical Education*, **82**, 765-768 (2005). Retrived June 4, 2008 from <http://jchemed.chem.wisc.edu/Journal/Issues/2005/May/PlusSub/V82N05/p765.pdf>
- Electrolysis . (2008). Electrolysis . In *Sci-Tech Encyclopedia* [Web]. Answers Corporation. Retrieved June 4, 2008, from <http://www.answers.com/topic/electrolysis>
- Lisensky, G. (2007, May, 14). Synthesis of nickel nanowires. *Interdisiplinary Education Group*, Retrieved June 4, 2008, from <http://mrsec.wisc.edu/Edetc/nanolab/nickel/index.html>

Answers:

Questions:

1) Describe electrolysis?

Electrolysis is a chemical change, produced by an electric current in an electrolyte. There are anodes and cathode electrodes that oxidize and reduce components of the reaction. Oxidation occurs at the anode, and a reduction reaction occurs at the cathode.

2) Identify the anode and cathode in this experiment?

The anode in this experiment is the nickel wire, and the cathode in this experiment is the copper plate.

3) What is being oxidized and what is being reduced.

Nickel solid from the wire is being oxidized, while Nickel ions in the electrolyte are being reduced into the membrane filter by the cathode.

4) Why doesn't the concentration of the Nickel plating solution change throughout the course of the experiment?

The nickel ions are replaced by those that are oxidized from the nickel wire.

Suggestions for application into class room

The idea of electrolysis is initially covered in undergraduate general chemistry classes, and the skill level required to complete this lab is on par with what could be done in a general chemistry lab. However, the other subject matter in this lab (ie. circuits and magnetism) is normally covered in an undergraduate physics course, so this lab would probably fit best in a General Physics lab. It should be noted that if there is a SEM available then the lengths of the nanowires can be measured for upper level chemistry students to do a rate study on the lengths of the nanowires.

Supplies for 10 students

10 of the Whatman Anodisc alumina filters

GaIn alloy

0.500 L of Nickel plating solution

100 cm of Ni wire

1 sq ft sheet of copper

20 alligator clips wires/ with alligator clips on both ends

10 battery holders

Electrical tape

10 AA batteries

0.150 L of 6 M NaOH

10 Glass slides and coverslips

50.0 ml concentrated Nitric acid

Access to microscopes