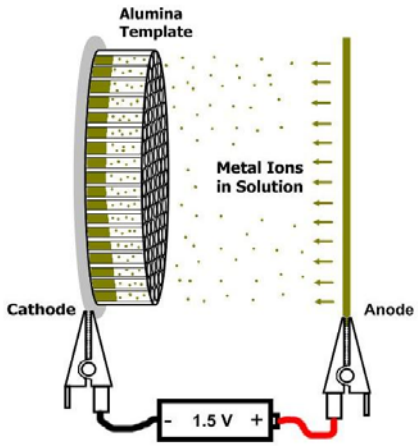
Name_____ Date_____ Class_____

Synthesis of Nickel Nanowires
Student Worksheet

Introduction

Figure 1: Model for synthesis of nickel nanowires. Figure developed by Paul Longwell (2009 NNIN-RET at Penn State)



Nanotechnology is the next big thing, and everyone is talking about it. Biosensors, nanomotors, and medical delivery systems are only a few of the many potential applications that scientists are currently working on. Nanowires are another area a research that may have applications to the electronics and medical fields. In a research lab, nanowires can be produced by sputter coating (where an object is coated with a thin layer of metal) or evaporating metals such as silver or gold onto the back of alumina membranes, which will make contact with the negative electrode in an electrolytic cell allowing various metals to form in the pores. This exact process can not be reproduced in the average high school, but by replacing the contact with a substance which can be applied easily; it may be possible to produce nanowires. In the following procedure you will use a liquid metal (Gallium Indium Eutectic) for contact and a AA battery to force the plating of nickel into the pores.

Purpose

1. To construct and study an electrolytic cell capable of plating metal ions (nickel) into alumina pores with a diameter of 20 nanometers.
2. To demonstrate how simple techniques can replicate the work done in a research level lab.
3. To consider possible applications of this process, and how further experiments could be performed using this procedure as framework.

Question

1. What applications can you think of for nanowires? What may also be needed for your potential application to become a reality?

Hypothesis Develop a hypothesis to answer one or both of the questions listed above.

Key Terms

Oxidation:

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Reduction:

Redox Reaction:

Electroplating:

Voltage:

Conductor:

Anodisc:

Scanning Electron Microscope (SEM):

Materials per student group

- Safety glasses
- Nitrile gloves
- Distilled water bottle
- Three 50 mL beakers per group
- One glass slide
- One small vial
- One thin stem pipette
- One pair of tweezers
- One strong magnet
- One AA battery with holder and +/- leads
- Two wires with alligator clips
- One Volt Meter (shared among groups)
- Electrical tape
- One cotton applicator (Q-tip)
- One piece of emory cloth
- One 3 x 10 copper strip
- One nickel wire
- One Whatman Anodisc (0.02 µm 25 mm) Cat. No. 6809-6022
- Gallium-Indium eutectic, 99.99+% (sigma-aldrich) (shared among groups)
- 50 mL of Watts nickel pure (nickel plating solution)
- 6 M Nitric acid (handled by teacher, NOT students)
- 5 mL of 3 M NaOH
- 50 mL of Acetone

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Procedure

This procedure is adapted from one developed by A. K. Bentley, M. Farhoud, A. B. Ellis, G. C. Lisensky, Ann-Marie Nickel, and G. Lisensky; *Template Synthesis and Magnetic Manipulation of Nickel Nanowires*; J. Chem. Ed. 82, 765-768 (2005).

A. Preparing the disc

Figure 2: GaIn coating of anodisc

1) Obtain a 0.02 micrometer Anodisc filter. The disc is quite brittle and is supported by a polymer ring. When handling, be sure that you are using tweezers to hold the support ring. **WITHOUT** turning the disc over, place on a glass slide and use a small piece of electrical tape to keep it in place.

QuickTime™ and a decompressor are needed to see this picture.

2) With gloves on, use a cotton applicator (Q-tip) to paint liquid Gallium Indium (GaIn) on the surface of the disc. Only dip the applicator into the metal once as it spreads very thin and addition GaIn will not be needed. Check for gaps in the coating by holding the glass slide up to the light. If there are any holes, continue to cover the surface. When you are finished it should look like the disc in figure 2.

3) Obtain a copper strip and sand down both sides using an emory cloth. Then rinse it off with distilled water and dry with a paper towel.

4) Remove the disc from the glass plate and flip it upside down so that the GaIn side is making contact with the **copper** strip that you have just cleaned.

5) Use electrical tape to fasten the disc to the copper electrode. Tape all sides of the disc so that the solution can not leak behind the disc, and tape the back of the copper strip. When in solution none of the copper should be exposed. When you are done your electrode should look similar to figure 3, except for the color of the disc.

QuickTime™ and a decompressor are needed to see this picture.

Figure 3: A completed copper electrode

B. Plating the nanowires

1) Use a voltmeter to test the voltage of a AA battery in a holder. The voltage should be at least 1.3 V. If less, set the battery aside and obtain a new one.

2) Fill the 50 mL beaker with the nickel plating solution. Insert your copper electrode so that the anode disc is completely submerged. If necessary use tape to fasten it to the side of the beaker. Then fasten a nickel wire to the opposite side of the beaker with tape.

3) Connect the **negative** lead of the battery to the **copper** electrode, and the **positive** lead to the **nickel** wire. Make note of the time that you started in your lab notebook. Disconnect the battery after 10 minutes have passed.
4) Remove the copper electrode from solution. Record your observations in your lab notebook.

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5) The nickel solution can be reused for this experiment, so pour it back into the small storage bottle provided by your teacher. **DO NOT** pour it back into the stock solution. Then answer the following question in your lab notebook.
    a. What happened to the nickel ions that were in solution?

    b. If you believe they were depleted, then how can the solution be reused?

C. Harvesting the nanowires

1) Rinse the copper electrode with distilled water.

2) Fill a 50 mL beaker with acetone and soak the electrode for up to 15 minutes. This will remove the adhesive from the tape. When the tape begins to curl, gently peal it off using tweezers.

3) Once the tape is removed, using tweezers remove the anode disc and flip it over so that the shiny GaIn side is facing up. Then place it on a glass slide and tape it down.

4) Take the slide to your teacher who will use nitric acid on a cotton swab to remove the GaIn in a fume hood. The teacher will soak the cotton swab in water before disposal, and rinse off both sides of the disc with distilled water.

QuickTime™ and a decompressor are needed to see this picture.

5) Pour 5 mL of NaOH in a clean 50 mL beaker, and place the disc inside. The ceramic material will begin to dissolve after 5-10 minutes. When the polymer support ring has separated, use a stirring rod to detach the remaining nickel from the inside, and then discard the ring in a solid waste container.

Figure 4: Removal of GaIn with nitric acid

6) Place the beaker on a strong magnet and move it from side to side. The nickel nanowires should follow the magnet.

7) Use the magnet to pull the nanowires to one side of the beaker. Then use a pipette on the opposite side to suck up and remove the NaOH. Add distilled water to the nanowires, and repeat the last step using the magnet and pipette several times to clean the wires.

8) When the wires have been cleaned make sure they are suspended in distilled water, and transfer them to a small vial. Label the vial with the names of your group members, and the contents. Answer the following questions in your lab notebook.
    a. What kind of microscope would allow you to see the nanowires in greater detail, an optical microscope or a scanning electron microscope?

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b. Could you create copper or zinc nanowires? If so, how would you do it?

c. Could you produce nanowires consisting of two different metals? If so, how could you do this? Would the wire be uniform (ex. Copper and nickel throughout) or would it have two distinct ends (ex. One copper and one nickel)?

Cleanup

Plating solutions are replenished through the metal wire or strips connected to the positive end of the battery, so they can be used indefinitely. Collect the solutions from the students in separate bottles or containers, just incase there is a contamination. This will prevent the loss of the stock solution if a procedural error was performed resulting in depletion of ions in solution. The metal strips can be reused. Emory cloth can be used to clean the surface and provide better contact. Small quantities of NaOH can be poured down the drain with excess water, and the nitric acid from the cotton swab can be diluted in water of the course of a few hours.

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