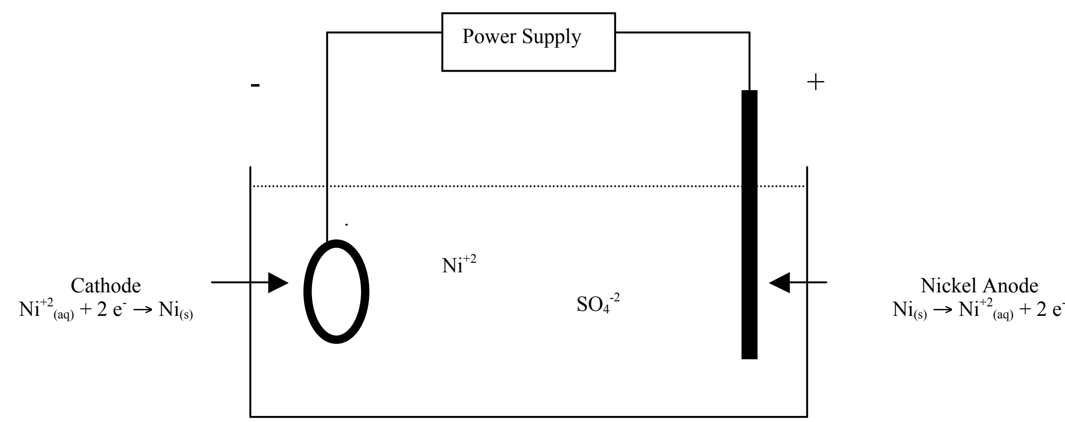
Nickel Electroplating

In a galvanic or voltaic electrochemical cell, the spontaneous reaction occurs and electrons flow from the **anode (oxidation)** to the **cathode (reduction).** In an electrolytic cell, a non-spontaneous reaction occurs using energy supplied by an external source. Electrons are forced from the external circuit at cathode to produce the reduction. The **cathode** is the **negative electrode** and is the **black** connection on the power supply. Electrons are drawn into the external circuit at the anode to produce the oxidation. The **anode** is the **positive electrode** and is the **red connection** on the power supply. Any electrochemical cell can be galvanic or electrolytic depending on the direction in which the reaction is proceeding. For instance, a car battery is a galvanic cell when it is discharging and providing energy and is an electrolytic cell when it is recharging and using energy. Electrolytic cells are commonly used in industry to produce aluminum, silver-plate dinnerware, gold-plate jewelry, and chrome-plate car bumpers.

In an electrolytic cell used for electroplating, the object to be plated is used as the cathode. Metal cations are reduced at the cathode and a layer of metal is deposited on the surface of the object. The anode is usually made of the same metal as the one being plated at the cathode. A concentrated solution of a very water-soluble ionic compound of metal being plated is used in the cell compartment as the electrolyte. This makes the solution very conductive and allows the plating to occur relatively quickly. The rate of electroplating and the amount of metal plated depend on the electrical current, in amps, and the time. A cell for plating copper is shown below.

In this experiment, you will nickel plate a piece of copper to make a pin.



Questions:

1. Which electrode should be the copper (anode or cathode)?
2. To which color wire should the copper be attached? (red or black)?

If a nickel electrode is used as the anode, the nickel is oxidized to Ni^{+2} ion, which goes into solution.

$$Ni_{(s)} \rightarrow Ni^{+2}_{(aq)} + 2\ e^-$$

If a stainless steel is used as the anode, the water is oxidized to O_2 gas which escapes. The H^{+1} ions produced go into solution.

$$2\ H_2O_{(l)} \rightarrow 4\ H^{+1}_{(aq)} + O_{2\,(g)} + 4\ e^-$$

Questions:
1. What happens to the mass of a nickel anode as the electroplating process occurs?
2. What happens to the mass of a stainless steel anode as the electroplating process occurs?
3. What happens to the mass of the cathode as the electroplating process occurs?
4. If a nickel anode is used, what happens to the $[Ni^{+2}]$ in the solution as the electroplating process occurs?
5. If a stainless steel anode is used, what happens to the $[Ni^{+2}]$ in the solution as the electroplating process occurs?
6. Write the anode and cathode reactions if a zinc anode and $ZnSO_4$ solution are used.

In order to get good adhesion between the object being plated and the metal being deposited on it, the surface of the object must be very clean and free of any metal oxide coating and other materials such as grease or oil. The grease is removed by dipping the object briefly in a basic NaOH solution followed by rinsing with water. The metal oxide coating is removed by dipping the object briefly in an acidic HCl solution followed by rinsing with water. **After cleaning, the object should not be handled with your fingers since the oil on your fingers may interfere with the plating process.**

Procedure:

Safety glasses must be worn during this experiment.

Part 1: Cleaning the pin

1. Place about 80mL of the NaOH cleaning solution into a 100mL beaker. Heat the solution on a hotplate to about 60-65^{o}C. <u>This solution is very basic and must be handled carefully. Clean up any spills immediately</u>

2. Hold the pin with a pair of tweezers and move the pin back and forth in the NaOH solution for about two minutes.

3. Holding a piece of paper towel under the pin to catch any solution that may drip, remove the pin from the NaOH solution.

4. Use the tweezers to hold the pin a 250mL beaker of water. Move the pin back and forth in the solution for about one minute to remove the NaOH.

5. Place about 80mL of 3M HCl into a 100mL beaker. <u>This solution is very acidic and must be handled carefully. Clean up any spills immediately</u>

6. Hold the pin with a pair of tweezers and move the pin back and forth in the HCl solution for about two minutes.

7. Holding a piece of paper towel under the pin to catch any solution that may drip, remove the pin from the HCl solution.

8. Use the tweezers to hold the pin a 250mL beaker of water. Move the pin back and forth in the solution for about one minute to remove the HCl.

9. Handle the pin with tweezers or with paper towels to avoid getting oil on the surface.

Part 2: Zinc Plating the Pin

1. Fill a 50x100mm crystallizing dish with the nickel plating solution to within an 1/8inch from the top (≅210mL). This solution contains hydrochloric acid, is corrosive, and must be handled carefully. Clean up any spills immediately

2. Place the dish on a hotplate and warm the solution to about 60-65^{o}C

3. Bend a piece of stainless sheeting so that it will hang over the edge of the dish and extend into the solution. This will be the anode and does not have to be cleaned.

4. Handle the pin with tweezers or with paper towels to avoid getting oil on the surface.

5. Attach the black alligator clip to the pin. The clip should just hold onto the edge of the pin. Use a point on the pin that will not interfere with the design on the pin. The bottom edge may be the best point. Attach the red alligator clip to the stainless steel anode. **The power should be turned on before the pin is lowered into the solution. The meter should be set at 5 on the dial.**

6. Lower pin into the solution. **Be careful not to let the pin touch the anode at anytime during the plating process.** The pin should be completely under the surface of the solution and held close to the edge of the dish. Plate the pin for about five minutes or until and even coating has been created. This may take less than 5min. Keep the front surface pointed towards the anode during the plating process so the nickel plates mainly on that surface of the pin. Some nickel will plate on the back surface but this will be minimal if the pin is held close to the side of the dish.

7. Remove the pin from the solution and rinse with water. Carefully dry the pin. Attach the pin back. Follow your instructor's directions about coating the pin with a clear acrylic spray.

This experiment is modified from Activity 6.4 Electroplating a Copper Ring from **Art in Chemistry; Chemistry inArt**,by Greenberg and Patterson, Teacher Ideas Press, 1998, pp 165-167.

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Setup Sheet

6 50x100mm glass crystallizing dishes labeled nickel plating
6 Anodizing units
6 Step-up transformers
6 Pairs of wire leads (one black and one red) with alligator clips at both ends
6 Tweezers (metal)
4 100mL beakers labeled NaOH cleaning soln.
4 100mL beakers labeled 3M HCl
4 100mL beakers labeled Rinse water
4 Wash bottles
8 Hotplates

Nickel plating solution (2 liters)
3M HCl (500mL)
6 Stainless steel strips (1/2 x 4 inch) for anodes
Acrylic spray

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Instructor's Notes

1. The copper for the pin was ordered from a jewelry supply house and cut on a metal brake. This avoids sharp and ragged edges on the ring and pin. The edges may need a little filing if they are still sharp. The pin was 1x 1.5inch. The pin was cut from copper plate.

2. The stainless steel for the anodes came from Reactive Metals, the anodizing supplier, and was cut with metal shears. The protective sliding door on computer floppy disk is a high nickel content stainless steel and can be cut into strips for use.

3. The nickel-plating solution came from a commercial plating company and included brighteners that gave a good shiny coating. This solution contains a concentrated solution of nickel chloride and nickel sulfate**. Mark the initial level of the solution on the edge of the evaporating dish. The plating solution is close to being saturated. Water will evaporate from the solution as it is heated during the class period. Add distilled water and stir to keep the volume roughly constant. If you lose too much water, crystals will start to grow in the beaker and on the anode. Crystals growing on the anode may stop the flow of current through the solution.**

4. The HCl solution will turn yellow from the dissolved copper ions from the oxide coating forming a complex ion with chloride ion. This solution should be saved and reused for this lab even if it is very yellow.

5. The NaOH cleaning solution can be reused.

5. It will save considerable time if a class plating system is set up ahead of time. Two beakers of NaOH cleaning solution can be sat on the same hotplate. Have the NaOH solution, rinse water, and the HCl solution at the same station. The transformer and anodizer unit should already be connected. The crystallizing dishes should have the solutions and the anodes in them already and the anode attached to the red leads. The NaOH cleaning solution and the plating solution should already be heated to the correct temperature.

6. The meters are set so the battery chargers produce 1-2V. The voltage output can be checked using a hand-held voltmeter.

7. Better plating is achieved if the power is turned on before the object to be plated in placed in the plating solution. You want a relatively low voltage and current. A high voltage and current will cause rapid but uneven plating with metal whiskers coming off the object. You want relatively low current and an even charge distribution across the object to get even plating. The anode and cathode should be kept as far apart as possible. The object will plate more heavily on the surface towards the anode. This is the front

surface of the pin is kept towards the anode. The object will not plate well were the clip is attached so the clip should be attached in the least obvious place.

8. Watch the plating carefully. If the object is left in the solution too long, you may build up a layer of metal that is thick enough to obscure the surface design.

9. The pin should be protected from surface oxidation with an acrylic spray.

10. Parts of the pin can be left a copper color by placing a resist on the surface. Heavy-duty packing tape or contact paper will work. Asphaltum will melt if the pin is left in the solution for very long. Asphaltum can be applied with toothpicks or thin stirring rods. It can be melted in a jar sat in a dish of warm water. It can be removed with acetone or hexane or mineral spirits.

11. The pin may be held by creating a small cage around the pin with thin copper wire. The wire should make minimal contact with the surface of the pin. The copper wire can be connected to the black lead during the plating process.