Student Activity Guide

Exploring Factors Effecting Deposition, Morphology, and Thickness of Thin Metallic Layer of Copper

Purpose: You will create thin layers of copper and nickel in an electrochemical reaction to understand how an electric current passed through an ionic solution will result in a chemical reaction which will separate materials. In addition you will also explore the various parameters effecting deposition, morphology, and thickness of the film to be deposited.

Nanotechnology is the science of the very small – atoms and molecules. Scientists and engineers are creating new materials and devices by using unique properties of nanoscale materials. Thin film layers are particularly important in nanotechnology and may have many applications including solar cells, fuel cells, and even DNA identification. You will be experimenting with variables that may or may not affect the depostion of thin films.

Make a Prediction

What variables do you think will have the most effect on the thickness and morphology of the copper film deposited in this experiment?

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Materials

- Balance
- Metal shears
- Drying oven or heat lamo
- AA batteries
- Ruler
- Acetone
- Copper wires
- Alligator clips
- (2) 500mL beakers
- 1 glass stirring rod
- Magnetic stir bar and stirrer
- Nickel foil
- Forceps
- 100 mL graduated cylinder
- Copper II Sulfate (anyhydrous)
- 10% Nitric Acid
- Cu foil (2)
- Nail Polish
- Connecting electrical wires
- 1 large watch glass
- 16oz soft drink bottle
- Microscope
- Spatula
- Voltmeter
- Sulfuric acid
- Toothpick
- Hammer

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Procedure

Cu Foil and Electrode Preparation

1. Obtain two pieces of copper foil and 1 piece of nickel foil from the designated area. Measure the length and width of the copper foil in centimeters and record in the table provided.
2. Determine the density of copper using your desired research method and record in the designated table.
3. Smooth out the foils with a hammer as demonstrated by the instructor.
4. Using forceps, you will clean the copper foil using a solution of nitric acid. Set solution aside for later use.
5. Pipette the nitric acid solution into a large watch glass 1 inch from the outer boundary.
6. Holding the foil firmly with the forceps, submerge the foil into the nitric acid and gently agitate the foil in the solution until it appears to be lustrous.
7. Clean each of the foil pieces in this manner.
8. Using a distilled water squirt bottle, hold the foil over your water rinse beaker and liberally rinse the foil completely.
9. Set aside the foil on clean paper towel, and blot dry. The foil must not be touched with bare hands once cleaning is completed
10. Using toothpick and nail polish, label one copper foil piece "CE1"(counter electrode 1) and the other, "CE2" (counter electrode 2) respectively.
11. Record the mass of each of the foil pieces and label in the appropriate table.
12. Obtain 3 lengths of copper wire that will serve as electrode post and clean with the nitric acid in the watch glass.
13. Hang the copper foil on the copper wire post by carefully bending the outer long edge boundary of the foil along the electrode post. Try to expose as much of the foil surface as possible.
14. Check to be sure that the foil is firmly contacted to the electrode post to ensure proper connection. Ask instructor if unsure of connection between the copper foil and post.
15. Next, take the remaining nickel foil and bend the short edge around the remaining electrode post so that as much of foil is exposed as possible.

Electrolysis Cell

1. Obtain the electrode chamber and rinse thoroughly with distilled water. Dry thoroughly with a paper towel.
2. Place the ends of the foil electrode posts through the appropriate holes in the electrode chamber in a way that the foil surface area exposed is maximized.
3. Check to be certain the setup is stable. See Figure 1.

Figure 1: Electro deposition Cell

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4. Next, transfer approximately 130mL of copper (II) sulfate solution and carefully pour into the electrode chamber. Fill to just below the holes punched.
5. It is imperative that the electrode post remain dry.

Battery, Connections, Exploration

Figure 2. Battery

1. Obtain a AA battery and two insulated wires. With electrical tape, secure one wire to the positive terminal, and the other wire to the negative terminal. Set aside for later use. See Figure 2.
2. Using a wire with alligator clips secure it to one end of the working electrode post (center post) and connect the other end to the negative terminal wire on the battery.
3. Decide what parameters the group as whole will test and perform the necessary steps to implement the test. All groups should have five strips on nickel foil on which either voltage, acidity, or time will be tested. One strip will serve as the control.
4. Label the strips with nail polish according to the parameter tested.
5. Due to the limited availability of nickel strips and time constraints students should only conduct five test with the five nickel strips which they have.

Voltage Parameter

1. In this test, the experimenter will cyclically alter the voltage exposure duration of the nickel substrate to examine the affects of the copper deposition.
2. As an example, the experimenter might choose start with a five second voltage exposure with a one second pause, or the experimenter might start with a one second voltage exposure or a five second pause.
3. It is up to the individual group to decide in what way the parameter will be tested. The only stipulation is that the voltage must be tested in such a way that leads to a clear relationship between cyclic duration and copper film deposit thickness (height).

Acid Concentration

1. In this test, the experimenter will alter the concentration of sulfuric acid in the $CuSO_4$ solution.
2. Again, it is up to the individual group to decide the manner in which the acid concentration will be altered to gain a clear picture of its effect on the deposition of copper.

Time Parameter

1. In this test the experimenter will alter the time the electro-deposition is allowed to take place.
2. Again, it is up to the group to decide the appropriate times to test.

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The Reaction

1. Once the parameters to be tested are known, secure a split wire and connect the split wires to both electrode posts on opposite sides of the foil post. Connect it to the positive end of the battery.
2. Record the time that the reaction began, and allow to run for 2 minutes (except those groups testing time)

Copper Foil Processing

1. After the reaction has run for 2 minutes, disconnect the battery terminal wires to stop the reaction.
2. Carefully remove the nickel foil from the electrode chamber.
3. Remove the foil from the electrode post by sliding the copper wire away from the foil.
4. Rinse the nickel foil gently with distilled water.
5. Place the nickel foil in a clean beaker and dry for 10 minutes under a heat lamp or in a drying oven.
6. Once the foil is completely dry obtain the mass of the foil sample and record in the appropriate table.
7. Repeat this process with the four remaining strips.

Microscopic observations

1. Describe the overall appearance of the deposited film, and make detailed notes of your observations.
2. Compare and contrast the surface of the plated copper to the other parameters tested.
3. Save a picture of your deposited layer using the computer connection to the microscope if (available).
4. How might an electron microscope allow one to study the sample more in depth.

Cleanup:

1. Any discarded nitric acid should be placed in the designated acid disposal container.
2. Acetone rinse should not be placed in the sink, but in a bottle designated for organic waste disposal.
3. Excess copper sulfate solution should be placed into a container designated for inorganic waste.
4. Copper wire electrodes not used up in the reaction should be cleaned in nitric acid, rinsed with distilled water, and placed in a beaker to dry.

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Data

I. Nickel Foil

Length(cm)	Width(cm)	Height cm (before)	Height cm (after)	Volume(cm^3)	Mass before	Mass after	Density (g/cm3)

	Mass C1	Mass C2
Cu Foil		
Cu Foil		

mass$_{before}$

mass$_{after}$

II. Voltage Parameter

Voltage Strength	Height of Deposit

III. Voltage Interval

Application Time	Height of Deposit

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IV. Exposure Time

Time of Exposure	Height of Deposit

V. Acidity of Solution

H_2SO_4 Concentration	Height of Deposit

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IV. Determining Thin Film Thickness

1. **The height of the film will be equal to the difference in thickness of the copper foil before and after the reaction.**
2. **The volume and density equations will be used in tandem to determine the height of the foil post reaction.**

Calculations

Height Before Reaction __.0046cm_ **mass nickel before = __.62g____**

V_{foil}= l * w * h **D=m/v**

l= 5 cm w = 3 cm

V_{foil}= mass/$Denisty_{copper}$

h= V_{foil}/l*w **mass=.62g $Density_{copper}$= 8.956 g/cm^3**

h= .06292cm3/5cm*3cm **V_{foil} = .62g/8.956 g/cm^3 = .0692 cm3**

h= .0046cm

Height After Reaction .0067cm **mass nickel after= ___.9g____**

V_{foil}= l * w * h **D=m/v**

l= 5 cm w = 3 cm

V_{foil}= mass/$Denisty_{nickel}$

h= V_{foil}/l*w **mass=.9g $Density_{nickel}$= 8.956 g/cm^3**

h= .1005cm3/5cm*3cm **V_{foil} = .9g/8.956 g/cm^3 = .1005 cm3**

h= .0067cm

Height of Thin Film= Height After Rxn – Height Before Rxn

= .0067 cm - .0046 cm

= .0021 cm, 21micrometers

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Analyze the Results

1. Did you observe what you predicted?

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If not, how did your observation differ from your prediction?

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2. What technologies do thin films stand to revolutionize?

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3. Which electrode served as the cathode in the electrolysis chamber?

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4. Which electrode served as the anode in the electrolysis chamber?

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5. Why might intermittent voltage affect deposition thickness and morphology?

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6. Why might solution acidity affect deposition thickness and morphology?

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7. How might the use of a scanning electron microscope (SEM) improve analysis of the deposited copper film?

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