

Gold Plating

Standard Operating Procedure

Faculty Supervisor: Prof. Robert White, Mechanical Engineering (x72210)

**Safety Office: Peter Nowak x73246 (Just dial this directly on any campus phone.)
(617)627-3246 (From off-campus or from a cell phone)**

Tufts Emergency Medical Services are at x66911.

Green, Todd A. "Gold electrodeposition for microelectronic, optoelectronic and microsystem applications." *Gold Bulletin* 40.2 (2007): 105-114.

Margesin, B., et al. "Stress characterization of electroplated gold layers for low temperature surface micromachining." *Design, Test, Integration and Packaging of MEMS/MOEMS 2003. Symposium on.* IEEE, 2003.

Warning: Elevate Gold 7990 RTU causes eye and skin irritation and burns. Causes respiratory and digestive tract irritations and burns. Dangerous if swallowed.

1. Material Requirements

1.1. Equipment: Three 1000 mL glass beakers, stainless steel tweezers, 10" by 10" hotplate, small probe thermometer, DC power supply, two 16" banana to alligator clip wires, two binder clips, 6" threaded Teflon rod

1.2. Chemicals: Elevate Gold 7990

"Elevate Gold 7990" contains sodium sulfite (4 %) and sodium gold sulfite (1.6 %) and is not considered toxic under Section 313 of Title III/SARA, and 40 CFR, Part 372

1.2.1. Hazards associated with chemicals (Elevate Gold 7990 RTU)

1.2.1.1. Inhalation of vapor and mist will cause irritation of nasal and respiratory passages

1.2.1.2. Contact with the eyes will cause irritation and burning

1.2.1.3. Brief skin contact will cause mild irritation. Prolonged contact may result in dermatitis

1.2.1.4. Ingestion will cause nausea, vomiting and diarrhea. Swallowing large amounts may result in systemic toxicity

1.3. Engineering Controls: Chemicals must be stored in tightly sealed bottles in cool, dry cabinets with secondary containment away from incompatible materials. All chemical processing must be carried out in the fume hood. Work space must be within proximity of an eye-wash station and safety shower.

1.4. Personal Protective Equipment (PPE): Trionic gloves on top of usual nitrile gloves, splash goggles and chemical apron.

2. Procedure:

Complete all processes under the fume hood

2.1. Seed layer deposition

- 2.1.1. Using PVD (sputtering or evaporation), deposit a seed layer. Typically Cr/Au is used but other seed layers may also work.
 - 2.1.1.1. The gold will plate only onto the seed layer, so if you pattern the seed layer before plating you will end up with patterned thick gold.
 - 2.1.1.2. Alternatively, photoresist may be patterned on top of the seed layer and used as a plating mask to pattern the thick gold.
 - 2.1.1.3. *Note that there must be a continuous electrical connection between the seed layer and the cathode during plating, so if you leave any unconnected "islands" of Cr/Au on your wafer they will not plate at the same rate as the rest of the wafer.*

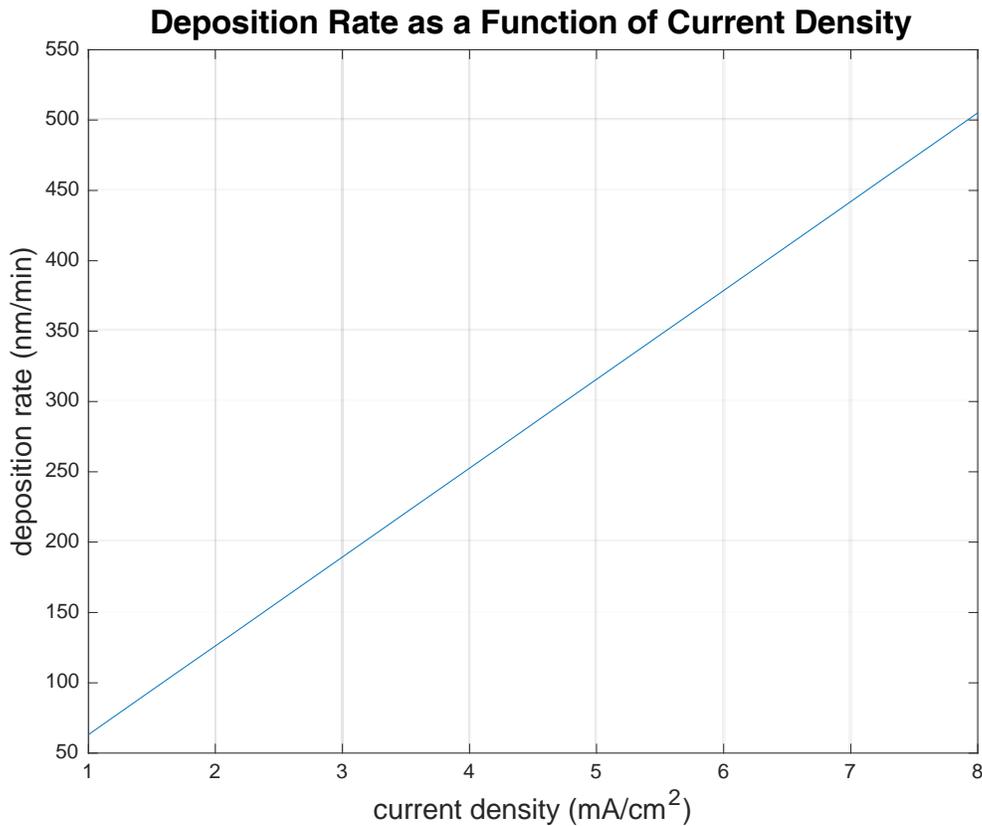
2.2. Solution preparation

- 2.2.1. There may already be a 1000 mL beaker with the Elevate Gold plating solution in the acid hood labeled as gold sulfite plating solution. Use this if it is already there.
- 2.2.2. If a new batch of the solution is needed, fill a 1000 mL beaker with enough Elevate Gold 7990 RTU so that your sample can be completely submerged.
 - 2.2.2.1. Make a clear entry in the log book indicating that the plating solution was changed and the date.
 - 2.2.2.2. Also please be sure to enter into the log book your plating current density and time so users can know how much the plating solution has been used.
- 2.2.3. Place the beaker and solution on the hotplate and set it to the desired temperature. The manufacturer recommends a temperature range between 45°C and 65°C. Monitor the solution temperature with a probe thermometer. **Note that the solution temperature will be considerably below the hotplate front temperature. Monitor the temperature carefully with the probe thermometer. There is a probe thermometer specifically for gold sulfite plating in the gold electroplating bin on the shelves.**
 - 2.2.3.1. *Mergasin, et al (see citation at top of SOP) found the optimal process conditions for low stress to be 3.5 mA/cm² and 58 °C. Stress was found to vary strongly with both parameters. You may need to experiment with the process parameters to achieve best results.*

2.3. Gold plating

- 2.3.1. Get two water rinse beakers that will fit your samples (1000 mL beakers for a 4" diameter wafer). **Do this first.** If something goes wrong, you want the water available to quench the reaction. Fill the 1000 mL beakers with deionized water such that the water level will cover the entire sample.

- 2.3.2. Use the platinized titanium as the anode. This will be stored in the gold electroplating bin on the shelves.
- 2.3.3. A binder clip works well to hold the wafer. Attach the binder clip to the wafer so that the wafer hangs down from the binder clip. Make sure that the binder clip makes good electrical contact to the seed layer!
- 2.3.4. Immerse the cathode and anode in the solution. The top edge of the wafer and top edge of the gold should poke out above the surface a little bit.
- 2.3.5. Hang the wafer from the Teflon threaded rod, and place the anode in the beaker with the surface to be plated facing the gold anode. Hang the two so they are approximately parallel.
- 2.3.6. Attach the alligator clips to the anode and cathode.
- 2.3.7. **Make sure the electrical connection to the power supply is the right way around!!! Negative terminal (cathode): wafer, Positive terminal (anode): platinized titanium.**
- 2.3.8. Determine your current based on the area of your seed layer. The manufacturer recommends a current density between $1\text{mA}/\text{cm}^2$ and $8\text{mA}/\text{cm}^2$. Higher currents will result in higher deposition rates but may increase surface roughness and other variables such as film stress.
 - 2.3.8.1. *Mergasin, et al (see citation at top of SOP)* found the optimal process conditions for low stress to be **$3.5\text{mA}/\text{cm}^2$ and $58\text{ }^\circ\text{C}$** . Stress was found to vary strongly with both parameters. You may need to experiment with the process parameters to achieve best results.
- 2.3.9. It is a good idea to set up this current value on the power supply beforehand, so that the correct current value will be attained immediately after turning on the power supply. Turn on the power supply and adjust to the correct current value if necessary.
 - 2.3.9.1. You should see the voltage drop slowly while the current remains constant.
 - 2.3.9.2. If the voltage starts to rise, it is most likely due to corrosion around the electrical connection to the wafer. Turn off the power supply, rotate the binder clip connection to another location on the edge of the wafer and then restart the power supply.
 - 2.3.9.3. If the voltage rises very suddenly, the problem is most likely a connection coming loose.



2.3.10. Using the graph above, calculate the approximate plating time for your process. At 3.5 mA/cm², expected deposition rate is 220 nm/min. The curve is determined using information provided by the manufacturer under ideal conditions. Actual rates may vary from the approximation so make sure to do your own tests to find an accurate rate and time!

2.3.11. After the plating time is complete, turn down the current and turn off the power supply.

2.4. DI water rinses

Perform these steps in the fume hood

- 2.4.1. Disconnect the wires from the sample.
- 2.4.2. Transfer the sample carefully to the first DI water rinse beaker.
- 2.4.3. If you used tweezers to move the sample, make sure you rinse them as well.
- 2.4.4. Let the sample and tools soak in the first DI water for 3 mins.
- 2.4.5. Transfer the sample to the second DI rinse beaker, and rinse for another 3 mins.
- 2.4.6. Do the same for the Titanium anode.

2.5. Sample dry

- 2.5.1. After the water rinse is finished, remove your samples and the anode and blow them dry with the air gun
- 2.5.2. Inspect wafer for the thickness of the gold. If more plating time is required, place wafer back into the plating tank with the solution. Repeat plating, rinsing and drying procedure

2.6. Clean-up

Perform these steps in the fume hood

- 2.6.1. The plating solution may be used several times before it needs to be replaced. Make sure to enter your deposition information in the log-book so we know how old the solution is. As of the writing of this SOP we do not have data on the effective lifetime of the solution.
 - 2.6.1.1. In most cases, the gold plating solution will be re-used. Cover the beaker carefully with parafilm. Make sure it is well sealed, we don't want any evaporation. Move it off the hotplate and back into the corner of the hood and ensure that it is clearly labeled with "Gold Sulfite Plating Solution" and the date.
 - 2.6.1.2. The DI rinse water should also be disposed of in the electroplating waste stream. This should be in an HDPE or glass bottle. The solution can be mixed with the copper and nickel plating wastes – sulfuric acid, nickel sulfamate, boric acid, and copper sulfate. If there is not an existing bottle in the satellite accumulation area, start a new stream in an HDPE bottle and label it appropriately.
 - 2.6.1.3. If the plating solution needs to be disposed of due to age, it should also be added to this same waste stream.
 - 2.6.1.3.1. **Make sure to leave a note in the logbook if you change the solution so that we know the total Amp-Hours of plating it has been used for.**
- 2.6.2. Rinse both DI water rinse beakers a second time and pour the waste into the electroplating waste bottle.
- 2.6.3. If the plating solution was disposed of, rinse the process beaker twice with water and pour the waste into the electroplating waste bottle.
- 2.6.4. Return all lab ware to its proper location.
- 2.6.5. Wipe up any drips in the area with chemical wipes and dispose in the acid trash.

3. Storage:

3.1. Elevate Gold 7990 RTU

- 3.1.1. Store Elevate Gold 7990 RTU in a bottle in a cool, dry and well place. Ensure that the bottle is tightly sealed and kept away from any incompatible substances.

4. Waste disposal:

- 4.1. Solid waste should be disposed of in the acid/base trash.

4.2. Liquid waste from this process should be disposed of in an HDPE or glass bottle. The solution can be mixed with the copper and nickel plating wastes – sulfuric acid, nickel sulfamate, boric acid, and copper sulfate.

5. **Accident Procedures:**

5.1. Contact: Read MSDS prior to working with any chemical to familiarize yourself with the symptoms of exposure and recommendations for treatment

5.1.1. Elevate Gold 7990 RTU

5.1.1.1. Eye contact: Flush with water, including under lids, for fifteen minutes. Obtain immediate medical attention.

5.1.1.2. Skin contact: Remove contaminated clothing and wash affected area thoroughly with soap and water. Launder clothing before re-wearing. Seek medical attention for prolonged skin irritation.

5.1.1.3. Inhaled: Remove from exposure and move to fresh air. Obtain medical treatment for dizziness, unconsciousness, irritation or any difficulties in breathing.

5.1.1.4. Swallowed: Do not induce vomiting. If patient is conscious, rinse mouth and drink at least two large glasses of water. If patient is unconscious, do not attempt to give anything by mouth. Obtain immediate medical attention.

5.2. Spills

5.2.1. For a small spill, collect spilled material by absorption using a lab wipe and dispose of it in the proper solid waste barrel.

5.2.2. If a large spill occurs and you are not comfortable cleaning it, Notify the Tufts emergency services (x66911) immediately. Also notify the faculty advisor.

5.2.3. Do not allow any of the chemicals to enter a storm or sanitary sewers or any other waterways.

If at any time you feel a situation is dangerous, do not hesitate to call the safety office (x73246, Peter Nowak) or the faculty supervisor (x72210, Robert White).

Report all accidents (injuries, major spills, fires) to the safety office at x73246 (Peter Nowak) and the faculty supervisor at x72210 (Robert White). For emergencies, call Tufts Emergency Services at x66911.