Nickel Plating (Nickel Sulfamate Plating Process)

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.

Revised: September 20, 2012

Warning: Nickel sulfamate is known to cause cancer if inhaled or ingested. For this reason we do NOT mix our own Nickel plating solution, but purchase it already mixed to reduce any chance of inhalation of nickel sulfamate powder.

Nickel sulfamate, nickel bromide, and boric acid solution cause slight irritation to the nasal and respiratory passages, minor skin irritation ("Nickel Itch") and burns to the eyes. If swallowed, will cause irritation to the mouth and throat, nausea, vomiting, and diarrhea.

1. Material Requirements:

1.1 Equipment: Two 1000 mL glass beakers, 2 4L rectangular glass tanks, , stainless steel tweezers, plating bath (polypropylene), 10" by 10" hotplate, FloKing filtration pumping system, small probe thermometer, DC power supply, two 16" long banana to alligator clip wires, two binder clips, threaded Teflon rod, 6" long

1.2 Chemicals: "Technic Nickel Sulfamate Semi-bright Ready to Use" contains nickel sulfamate (20-35%), nickel bromide (0.5-1.5%), and boric acid (1-3%).

1.2.1 Hazards associated with chemicals (nickel sulfamate, nickel bromide, boric acid):

1.2.1.1 Individuals sensitive to nickel may develop allergic dermatitis ("Nickel Itch"), and asthma, bronchitis, or wheezing.

1.2.1.2 Nickel compounds are known to cause cancer.

1.3 Engineering Controls: Store bottles of chemicals (sealed tightly) in cabinets (acid cabinet) with secondary containment. Work area should contain an eye wash and safety shower. All chemical processing should occur in the chemistry fume hood.

1.4 Personal Protective Equipment: Trionic gloves on top of nitrile gloves, and goggles.

2.0 Procedure:

Complete all processes in the fume hood

2.1 Seed Layer Deposition

2.1.1 Deposit (using sputtering or evaporation) a "seed layer" consisting of 20 nm of Ti and 100 nm of Cu onto your wafer. Other metals and thickness will also work as seed layers. Cr/Au 50 nm/300 nm has been tested and also works fine. Some differences in results may be observed for different seed layers.

2.1.1.1 The nickel will plate only onto the seed layer, so if you pattern the seed layer before plating you will end up with patterned thick nickel.

2.1.1.2 **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 seed layer on your wafer they will plate much more slowly than the rest of the wafer.

2.2 Prepare the Technic "Ready to Use" nickel sulfamate semi-bright plating solution (Note: this is truly "ready to use" … it has all the necessary components, and has already been balanced by the manufacturer.)

2.2.1 It is likely that there is already a 4L rectangular tank with Nickel plating solution in it, in the acid hood, labeled as Nickel Plating Solution. Use this if it is already there.

2.2.1.1 Do NOT fill the tank to the fill line yet. The tank should be filled to the fill line only <u>AFTER</u> the filtration pump is in place (this will be discussed below).

2.2.2 If a new tank is needed, fill the tank with "Ready to Use" Nickel Sulfamate (semi-bright, mechanical agitation) from Technic.

2.3 Heat the Solution

2.3.1 For Nickel plating, 50 ^{o}C solution temperature is recommended. Place the plating tank on the 10" x 10" hotplate (carefully!).

2.3.2 Remove the aluminum foil and Parafilm cover.

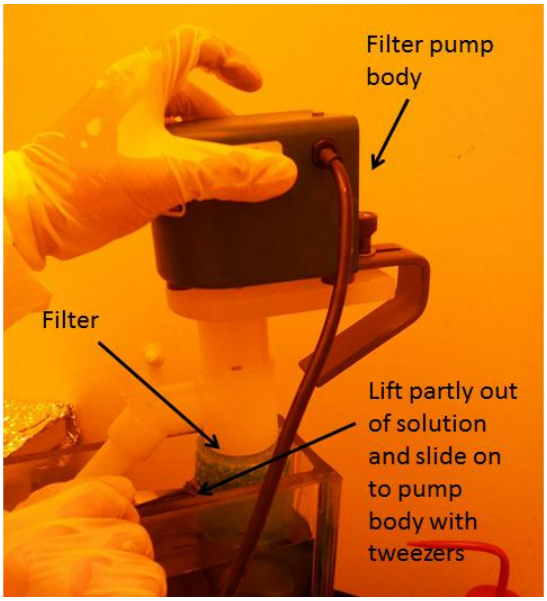
2.3.3 Put the plastic cover on with the angled hole, and put the small battery powered probe thermometer through the hole so that the probe pokes into the solution.

2.3.4 Set the hotplate front temperature to <u>200 ^{o}C and wait for 1.5 hours</u>.

2.3.5 <u>Reduce hotplate temperature to 160 ^{o}C.</u> The solution temperature should stabilize at 50 ^{o}C. Adjust the hotplate by small amounts if necessary to achieve 50 ^{o}C solution temperature

2.3.6 **AT <u>NO TIME</u> SET THE HOTPLATE TO <u>HIGHER THAN 200^{o}C.</u> IF THE HOTPLATE IS SET TOO HIGH THE GLASS TANK MAY CRACK AND THE SOLUTION ALL COME SPILLING OUT. THIS HAS HAPPENED.**

2.4 Setting up the Filtration Pump



2.4.1 Remove the FloKing filtration pump body from the water soak where it is stored. It should not yet have a filter on it.

2.4.2 You should see the filter cylinder floating in the Nickel plating tank (it is stored in there so it is always soaked with nickel plating solution). Lift the filter gently part way up and at an angle with your tweezers, so that you can slide the filter pump down into the center of the filter. Note that one side of the filter is closed with a plug, only one side will be open, make sure it is the right way around.

2.4.3 Hang the filter pump body on the back of the tank using the clamp.

2.4.4 Once the filter is in place, check the level of the nickel plating solution. It should come up to the line marked on the side of the tank, so it is below the motor portion of the pump, but above the branch for the Y shaped tube. If necessary, add some nickel plating solution from the "ready to use" bottle to bring the solution level up to the marked fill line.

2.4.5 Plug in the filter. You should see gentle recirculation of the solution. It is being mixed and actively filtered. This is critical to achieve a smooth and particle- free film.

2.5 Nickel Plating

Perform these steps in the Chemistry Fume Hood.

2.5.1 Fill two water rinse beakers which will fit your samples (1000 mL beakers). **Do this first**. If something goes wrong, you want the water available to quench the reaction. Fill the 1000 mL beakers with deionized water.

2.5.2 A strip of pure nickel sheet or foil will be used as an anode. It is recommended to use an anode that is approximately the size of the total sample size to maintain a more uniform current density. For a 4" diameter wafer, use a piece approximately 3" wide and 5" long. Make sure this is clean… do not use an old one if it looks tarnished.

2.5.3 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.

2.5.4 Attach the second binder clip to the nickel anode.

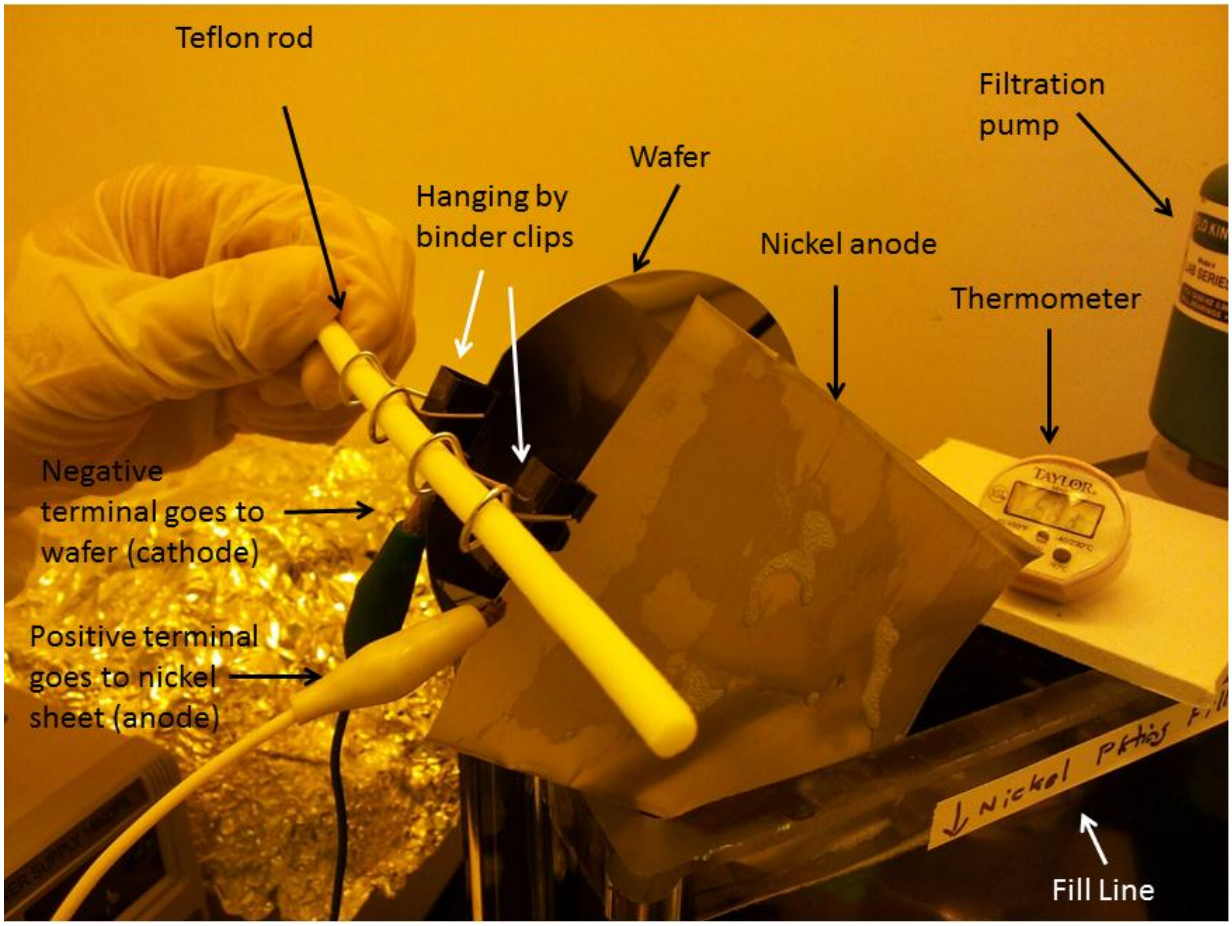
2.5.5 Hang the wafer and the nickel anode from the Teflon threaded rod, with the surface to be plated facing the Nickel anode. Hang the two so they are approximately 1 inch apart, and parallel.

2.5.6 Immerse the cathode and anode in the solution. The top edge of the wafer and top edge of the nickel should poke out above the surface a little bit.

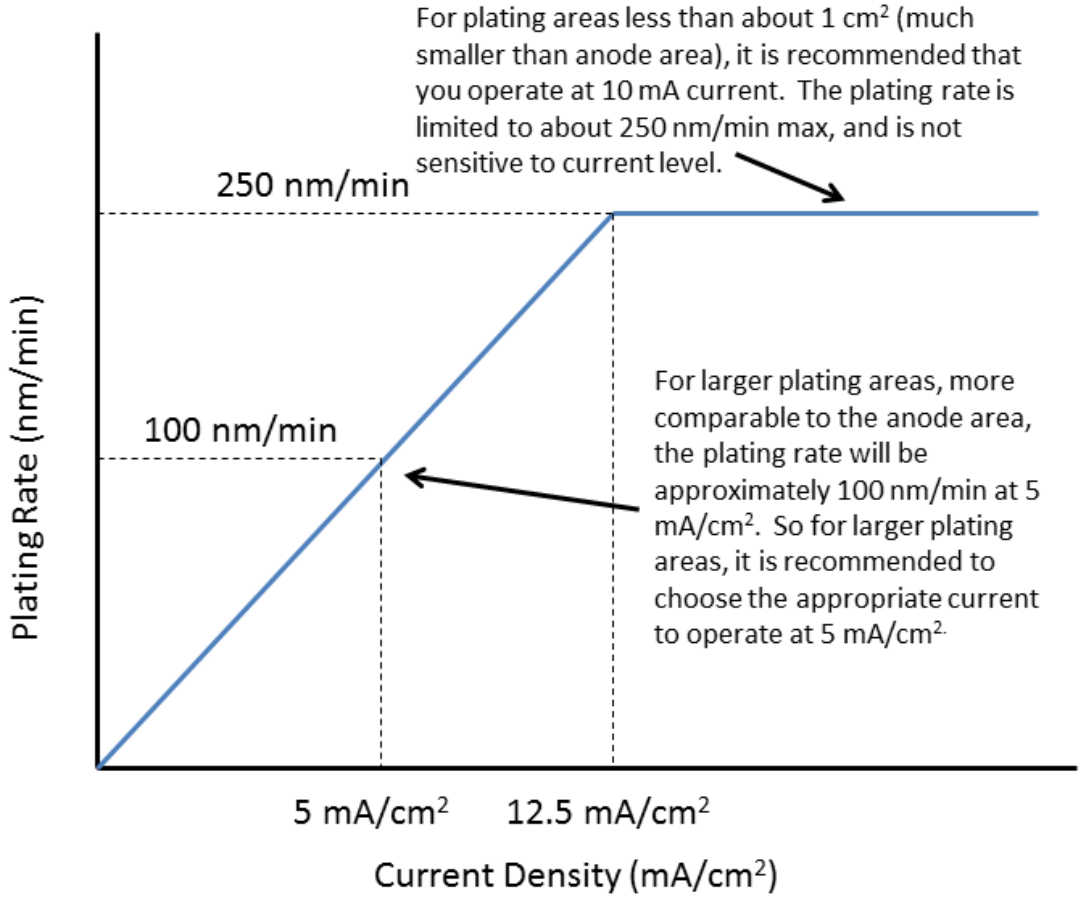
2.5.7 Attach the alligator clips to the anode and cathode. Note that it is critical that there is a place for the alligator clip to attach to the wafer that is not coated with photoresist! You need a good contact region to make connection to the seed layer. This should be designed in as part of your mask, otherwise you may been to try to use acetone on a swab to open up a little region at the edge of the wafer to make contact.

2.5.8 **Make sure the electrical connection to the power supply is the right way around!!! Negative terminal (cathode): wafer, Positive terminal (anode): nickel plate**.

2.5.9 The completed set up should look similar to the picture below.



2.5.10 Decide what current you want to operate at. It is recommended that you operate at 5 mA/cm^2 of plating current density at the cathode in order to produce a smooth film. However, in many cases, you may find that your plating area is so small (less than 1 cm^2) and so 5 mA/cm^2 is a very small current (less than 10 mA). In these cases, operate at 10 mA of current. At the time of writing, the following current density to plating rate curve has been approximately observed (YOU BETTER CHECK THIS! IT COULD BE DEPENDENT ON A NUMBER OF OTHER PARAMETERS! DON'T TRUST ME TOO MUCH… DO SOME TEST JOBS AND MEASURE YOUR OWN PLATING RATE).



2.5.11 Set up the power supply. You want to operate in constant current mode (current limited mode) to get a constant deposition rate. In order to accomplish this:

2.5.12 First turn the voltage and current all the way down on the power supply.

2.5.13 Unclip both of the alligator clips (open circuit).

2.5.14 Turn the voltage up, to about 3 V. You will notice you are in constant voltage (CV) mode. You should not have to operate at voltage above 3 V. You have set the maximum voltage to 3 V.

2.5.15 Now, make sure the current is turned all the way down.

2.5.16 Clip the clips together (short circuit).

2.5.17 Now you will be in constant current (CC) mode. Slowly turn up the current until you get to the plating current you want to operate at (see chart above to choose desired current).

2.5.18 Now unclip the clips and turn off the power supply. Clip them back on to the wafer and nickel. Remember: **Make sure the electrical connection to the power supply is the right way around!!! Negative terminal (cathode): wafer, Positive terminal (anode): nickel plate**.

2.5.19 Now turn the power supply back on. If you have a good connection to the nickel and the wafer, you should see that you are in constant current mode at the current you set, and the voltage should be somewhere around 1.5V. If the voltage is much higher than this, you may have a bad connection somewhere… either the clips are not well placed, the clips are corroded, or there is a dielectric barrier (like photoresist) between the clip and your seed layer. Adjust accordingly. Clips will need to be replaced periodically when they get too corroded.

2.5.20 If the voltage and current look good, you are plating! Start your timer.

2.5.20.1 You should see the voltage drop slowly while the current remains constant.

2.5.20.2 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.

2.5.20.3 If the voltage rises very suddenly, the problem is most likely a connection coming loose. It could also be anode polarization (see above).

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

2.6 DI Water rinse: 10 min

Perform these steps in the Chemistry Fume Hood.

2.6.1 Disconnect the wire from the sample.

2.6.2 Transfer the sample carefully to the first DI water rinse beaker.

2.6.3 If you used tweezers to move the sample, make sure you rinse them as well.

2.6.4 Let the sample and tools soak in DI water for 3mins.

2.6.5 Transfer the sample to the second DI rinse beaker, and rinse for another 3 mins.

2.6.6 Do the same for the nickel anode. As long as it has not corroded too extensively, it may be reused.

2.7 Sample dry:

2.7.1 After the water rinse is finished, remove your samples and the anode and blow them dry with the air gun

2.7.2 Inspect wafer for the thickness of the nickel. If more plating time is required, place wafer back into the plating tank with the solution. Repeat plating, rinse and drying procedure.

2.8 Clean-up:

Perform these steps in the Chemistry Fume Hood.

2.8.1 Turn off the hotplate.

2.8.2 Unplug the filter pump.

2.8.3 With your tweezers, carefully slide the filter off the filter pump body so it stays in the nickel tank and continues to soak.

2.8.3.1 If the filter becomes too dirty, leave it on the pump and pump water through it for 5 minutes. Then leave it to drip dry into the water rinse tank (4L rectangular tank labeled as water rinse). When it is dry discard it in the acid/base solid waste trash can.

2.8.3.2 If you discard an old filter, then get a new one and leave it to soak in the nickel plating solution for at least 2 hours before being used.

2.8.3.3 If you replace the filter **note it in the log book.**

2.8.4 Move the filter pump body over to the water rinse tank (without the filter on it, unless the filter is being discarded as described above). Hang it on the side just like you did in the nickel plating tank and plug it in. Let it recirculate rinse water for 5 minutes, then unplug it and leave it hanging in the water rinse tank.

2.8.5 The nickel plating solution may be used many 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. The solution should be discarded after 80 Amp-Hours of plating, or 6 months, whichever comes first (These criteria are arbitrary as of the writing of this SOP, and may need to be refined based on experience).

2.8.5.1 In most cases, the nickel solution will be re-used. Cover the tank carefully with parafilm and the glass cover, then with two layers of aluminum foil. We don't want evaporation. Move it off the hotplate and back into the corner of the hood. Make sure it is clearly labeled.

2.8.5.2 If the solution is too old and needs to be replaced, pump it (using the peristaltic waste pump) into the nickel plating waste bottle (HDPE bottle) which is labeled "nickel sulfamate, nickel bromide, boric acid" with the red hazardous waste tag. Keep the bottle in the satellite accumulation area (under the hood). If a waste bottle

already exists, use that one, otherwise start a new one. You may need two waste bottles for a full 4L tank.

2.8.5.3 If you change the nickel solution **note it in the log book.**

2.8.6 Dump the first DI rinse beaker and into the nickel plating waste bottle (HDPE bottle).

2.8.7 Dump the second DI rinse beaker into the 5 gallon HDPE "Dilute Acid Waste" container.

2.8.8 Rinse both containers a second time with DI water. This time, dump them into the 5 gallon HDPE "Dilute Acid Waste" container.

2.8.9 Return all lab ware to its proper location.

2.8.10 Wipe up any drips in the area with chemical wipes and dispose in the acid trash.

3.0 Storage:

3.1 Chemical one: Technic Nickel Sulfamate Semi-bright Ready to Use (Nickel sulfamate/nickel bromide/boric acid)

3.1.1 Store in a tightly closed container. Store in the acid cabinet. Store in a cool, dry, well-ventilated area away from incompatible substances.

4.0 Waste Disposal:

4.1 Technic Nickel Sulfamate Semi-bright Ready to Use (Nickel sulfamate/nickel bromide/boric acid)

4.1.1 Solid waste for chemicals and corroded anodes should go in the acid waste bin.

4.1.2 Liquid waste for chemicals should go in the nickel plating waste bottle. Label "nickel sulfamate, nickel bromide, boric acid".

5.0 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 Technic Nickel Sulfamate Semi-bright Ready to Use (Nickel sulfamate/nickel bromide/boric acid)

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

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

5.1.1.3 Ingestion: If patient is conscious, rinse mouth and drink at least two large glasses of water. DO NOT induce vomiting. Never give anything by mouth to an unconscious person. Obtain immediate medical attention.

5.1.1.4 Inhalation: Remove patient to fresh air. Support breathing if required. Obtain medical treatment for dizziness, unconsciousness or irritation or difficulty in breathing.

5.2 Spill:

5.2.1 If a small, contained spill occurs, such as inside the hood, wipe it up with chemical wipes and dispose of in the appropriate trash container.

5.2.2 If a large spill occurs that you are not comfortable cleaning up:

5.2.2.1 Notify the Tufts emergency services (x66911) immediately. Also notify the faculty advisor.

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.