# Electroplating Standard Operating Procedure

## Purpose

1. To describe the startup, operation, and conclusion processes required to use the electroplating process.
2. The process is designed to be used by researchers in the metal coating and other innovative and under study research for metal bonding.

## Scope

1. This SOP is intended for students, staff, faculty and non-university individuals.

## Prerequisites

1. Training requires prior approval from the lab manager

## Responsibilities

1. The equipment coordinator (Dr. Asiabanpour) and his appointees are responsible for conducting the training.

**Emergency Contact:**

* Call 911
* Call EHS & Risk Management at 512-245-3616
* Call Head Lab Technician, Dr. Ray Cook (office 512-245-2050)
* Call Dr. Asiabanpour (office 512-245-3059)

**Before using this machine:**

* You must have permission from Dr. Asiabanpour.
* You must have received formal training from process coordinator, technician or, trained research student (designated by Dr. Asiabanpour) related to process safety and operation.
* You must read and understand **SOP.**
* You must use this process under direct supervision of Dr. Asiabanpour or, Dr. Cook or, trained research student (designated by Dr. Asiabanpour).

1. ***Material***

|  |  |  |
| --- | --- | --- |
| Chemical Name | Chemical Formula | Wt.% |
| Nickel Sulfate | NiSO4 | 70-80 |
| Nickel Chloride | NiCl2 | 10-15 |
| Boric Acid | H3BO3 | 5-10 |

Potential Acute Health Effects: Very hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation. Hazardous in case of skin contact (corrosive, permeator), of eye contact (corrosive). Slightly hazardous in case of skin contact (sensitizer). The amount of tissue damage depends on length of contact.

Physical state and appearance: Solid

Odor: None

APPEARANCE: Green crystal

SOLUBILITY IN WATER: 62

SPECIFIC GRAVITY: 2.070

TOXICOLOGICAL INFORMATION

ACUTE

ORAL LD50: 275 mg/kg (rat)

CARCINOGENICITY

IARC: Suspect cancer hazard.

DISPOSAL METHOD: Comply with all local, state and federal laws.

TRANSPORT INFORMATION

DOT (DEPARTMENT OF TRANSPORTATION)

PROPER SHIPPING NAME: Environmentally Hazardous Substances, Solids, N.O.S.

TECHNICAL NAME: Nickel Sulfate

PRIMARY HAZARD CLASS/DIVISION: 9

UN/NA NUMBER: UN3077

PACKING GROUP: III

REGULATORY INFORMATION

UNITED STATES

DOT LABEL SYMBOL AND HAZARD CLASSIFICATION

 Class 9

|  |  |
| --- | --- |
| Skull and crossbones | C:\Users\nh1166\Desktop\Environment.PNG  Environment |
| Danger | Danger |
| H301: Toxic if swallowed.  H332: Harmful if inhaled.  H317: May cause an allergic skin reaction. 9242258Q: Causes damage to lungs through prolonged or repeated inhalation exposure.  H334: May cause allergy or asthma symptoms or breathing difficulties if inhaled. | H410: Very toxic to aquatic life with long lasting effects. |
| Health hazard |  |
| Danger |  |
| H360: May damage fertility or the unborn child.  H350: May cause cancer. |  |

**POTENTIAL HEALTH EFFECTS**

**EYES:** Causes eye irritation.

**SKIN:** Long term exposure to skin may cause skin irritation and may cause sensitization or allergic

reactions resulting in chronic eczema or "nickel itch"

**INGESTION:** Harmful if swallowed.

**INHALATION:** Dust and processing vapors may cause respiratory tract irritation.

**SIGNS AND SYMPTOMS OF OVEREXPOSURE**

**CHRONIC EFFECTS:** Frequent or prolonged contact may irritate the skin and cause a skin rash

(dermatitis).

**CARCINOGENICITY:** Cancer hazard.

**REASON FOR ISSUE:** Add GHS Info

**PREPARED BY:** L CASWELL

**REVISION SUMMARY:** New MSDS

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## Procedure

## Protection Equipment:

* **Fume Hood:** Working with any Nickel Chrystal (Nickel Sulfate+ Nickel Chloride+ Boric Acid) requires proper ventilation. The hood sash must be in front of the face at all times, and the sample should be at minimum 6 inches back from the opening of the hood.
* **Face**: The eyes must be covered at all times, since Nickel Chrystal can be absorbed through the eyes - one should never work with the material without proper covering. It is highly recommended to wear full face shield (at the very minimum eye covering), and if necessary, respiratory protection which has been fitted and certified by EHS – the user must be properly trained on it as well (<https://www.ehs.ucla.edu/ep/ih/resp>). Respiratory protection must be used during spill clean ups, so it is essential to have a properly fitted mask at all times in the laboratory.
* **Clothing**: Protective coverings of all exposed skin must be worn. Handlers must wear an impermeable lab coat, pants that cover the legs entirely and closed toed shoes. Lab coat must be buttoned up to the top while uncontaminated gloves are on.
* **Additional External Protection**: Full wrist covering thick nitrile/rubber gloves must be worn along with a rubber apron worn across the chest. Additionally, coverings of the shoes can be used as well. Wear nitrile gloves underneath the thick handling gloves, so that when the outer ones are removed there will be a second tier of protection.
* **Never work alone**. Handling acutely toxic chemicals bears certain precautions and trained personnel must be standing by to act immediately in the event of anything going awry during the plating process. Spills are the greatest concern with this chemical.

## Spill Procedure

Controlling spills should be done immediately. Small and large spills should be addressed in a serious manner. Have an evacuating bystander contact Texas State EHS (**512-245-3616**) for any chemical spills.

1. Assess the situation; assist anyone who has been injured. Clear the lab space and if the spill has been over 1 liter, evacuate the lab.
2. Avoid breathing vapors, do not approach the spill site without protective gear and proper training.
3. **Protective gear includes**:
   1. Lab coat
   2. Nitrile gloves under wrist covering thick rubber/poly gloves
   3. Respiratory protection certified by EHS. This includes completing a pulmonary function test, getting fit tested and completing the respiratory protection training.
   4. Feet coverings with booties and no open toed shoes/exposed skin.
   5. Eye coverings with full facial shield (if using a half face respirator lacks attached eye protection)
   6. Legs covered (no shorts in any lab space anyways)
4. Confine the spill (if possible) by sprinkling a non-reactive absorbent spread over the site liberally and utilizing an absorbent cloth wipe up. The spread should have a jelly-like texture when fully saturated. Place into a plastic bag, which should then be placed into a secondary waste storage that has a screw on top to contain fumes. All waste should be at a controlled pH above 11.
5. If the spill is over 1 liter, it’s considered a large spill. Prompt evacuation and closure of the entire lab space should be of utmost importance. Someone besides the electroplater should be in charge of this, as the electroplater must address the spill almost immediately.
6. After placing absorbent material into plastic bag and properly storing it in the sealable waste container – spray all surfaces that could have come in contact with the fluid with a high PH solution. This should be stored directly to the right of the electroplating apparatus. Wipe all surfaces.
7. Once area has been neutralized and spill has been contained, contact EHS to report the spill and ensure that nothing has escaped into any drains.
8. **Never dump any Nickel Chrystal or Nickel Chrystal waste in the sink.**

## Donning of Safety Equipment (Pre-Plate):

1. The contamination area should never be accessed by someone who doesn’t have proper protective equipment: gloves and eye protection at a minimum. When the plating solution is not exposed to the open air and is properly stored, there is little risk of exposure.
2. **Ensure that warning signs are displayed at all entrances of the lab**, and everyone working in the lab is notified the chemical will be out and used.
3. Before entering the contamination area, the electroplater must have his lab coat, facial protection and rubber garb on.
4. Enter garb area with nitrile gloves on. Put the lab coat on and ensure it’s buttoned to the top.
5. Place secondary **clean** thick gloves on. The gloves must go over the sleeves of the coat.
6. Place eye protection and cover mouth with N95 respirator (filter mask).
7. Remove rubber apron from its hanging point, establish which side is the front and fasten it securely behind your back. Avoid touching the front side if you can.
8. Place full facial shield on.
9. Place foot coverings (this should always be the last step)
10. At this point, consider your gloves contaminated –physical contact should not be made outside the contamination workspace.
11. When working with the Nickel Chrystal, other lab workers must retain extreme caution and stay out of the contamination area at all times. Check the state of the absorbent mat, and make sure the neutralizing spray/additional absorbent material are in the fume hood and within reach.
12. Before touching anything inside the fume hood, open the wafer containers and have them within reach. Do not touch the containers once you’ve started handling the plating solution.
13. Turn power supply on, make sure switch under hood is off. If it is on, turn power supply on first before touching the switch.
14. **Commence plating process. Once you’ve gone under the fume hood and started working, do not veer from the work site or touch anything outside the hood without decontamination your gloves.**

## Plating Process (Nickel)

1. C:\Users\nh1166\Desktop\22.tifMake a beaker bar (1 lit) from a piece of 1.27 cm diameter copper pipe. Sit on top of the beaker.
2. Fill the beaker with 0.5 liter distilled water (figure 1).
3. Install the heater and heat the water at approx. 77 C.
4. Add 120.7 gr of Nickel Electroplating Crystals. Stir until most of the crystals are dissolved through magnetic stirring.
5. Add 21 ml of Nickle Brightener
6. (optional) Mark liquid level with permanent marker
7. (optional) Turn off the heater and let solution sit overnight to age
8. Take one nickel anodes (sheets of nickel) and cut a 0.3 cm strip most of the way up the side of each anode (refer to Fig 2). Wrap each anode with a white gauze bandage. Hang these anodes on each side of the tank, so they mostly sit in the plating solution.

Figure 1. Setup for Nickel Electroplating

1. ****Connect the anode with a wire
2. Connect the anode to the positive side of your power source
3. Connect the tank bar to the negative side
4. Hang the part to be plated into the tank with copper wire
5. Install heater and pump , heat to approx. 45 C
6. (Optional) Decontaminate the area with Bleach (NaOCl) but only after you are done and stepping away from fume hood. **Do not spray bleach directly into the plating solutions.**
7. Repeat process for multiple wafers.

Figure 2.

## Decontamination (Post-plate):

1. After the completion of the process – ensure that no spills have occurred. *If a spill has occurred, refer to the spill containment section above.*
2. Decontaminate/neutralize the area utilizing a high pH spray made with Sodium or Calcium hypochlorite. Wipe with absorbing cloth and dispose of the cloth.
3. Spray your apron with water and bleach, dry with clean absorbing cloth.
4. Spray gloves with water and bleach, dry with clean absorbing cloth. Take gloves off from the top and pull outward so that they are inside-out when completely removed.
5. Untie the apron with your nitrile gloves still on, make sure the outer gloves you just removed have been cleaned and the apron has been cleaned. Hang up in its designated spot. Remove your booties *from the back* and place in designated box. **Do not step back onto the mat.**
6. Remove your lab coat and place in its case next to cyanide workspace.
7. Remove nitrile gloves and place into proper waste container.

## Controlling the Power:

**The Power Supply and Power Requirements**.

Unlike plating, anodizing has the peculiarity of becoming an insulator to itself, cutting off power and stopping further growth of the film. The thicker the film, the more insulated the part becomes from the power supply. There comes a point when a Peak Anodic Resistance (PAR) is reached, when the film will grow no more, and if power is kept being applied, it actually erodes away the film. PAR is quite visible on a rectifier, because the amperage needle drops off. It is therefore useful to install some type of ammeter into your system, so you can see when you reach PAR. The optimum current requirement is 4.5 amps per sq foot, or 30 milliamps per sq inch. This can easily be supplied from a 12 volt battery and controlled using light bulbs.

See the FLASH VIDEO on [www.caswellplating.com](http://www.caswellplating.com/) concerning controlling the power with light bulbs

**Battery Chargers**

Battery chargers do a good job on anodizing, but the current still needs to be controlled. Using light bulbs will do this economically. Set the charger to the 12 volt position.

|  |  |  |
| --- | --- | --- |
| At 0 A | (no load) | V=13.4 V |
| At 3.66 A | (3 Ω) | V=11 V |
| At 5.35 A | (2 Ω) | V=10.7 V |

There are some major misconceptions about using battery chargers as power sources. Battery chargers are rated for driving a partially discharged battery, not a grounded load like anodizing or plating. As an example, the voltage was measured and current of a charger under load. This unit was rated for 12V at 10A, when loaded the results were as follows:

A 10 amp load wasn’t tested as it would overheat the charger and open its thermal circuit breaker if operated for any realistic length of time. If this unit were rated as a transformer isolated unregulated power supply, using a full wave rectifier (which is what it is) the rating would be 10.8 V at 5 A. Larger or smaller chargers will scale accordingly.

Besides not putting out the voltage and current that you think you are getting, battery chargers also have no effective means to reduce the voltage and current provided to the load. You can compensate for the first problem by de-rating the battery charger as discussed above, and there is something you can do about the control problem. A perfectly simple way to solve the control problem is to use an ordinary 600 W lamp dimmer to control the input to the charger. This is shown in Figure 6. A charger is a transformer load, not a motor load. The dimmer can power a transformer as easily as it can power a light bulb. The resolution you can actually get isn’t great, but it is better than you can get using any reasonable number of power resistors or light bulbs to control the current.

1. Batteries

Batteries are not the best power source for anodizing. You are better off using a battery charger controlled with a light dimmer switch. Monitor the current using a multi-meter and a shunt 0.1ohm resistor, as per fig 6.

## Rectifiers

Rectifiers are the ultimate in anodizing. Variable controls, voltage and amperes dials, allow you to fine tune your anodizing efforts. This can be especially useful when dying, as the minor variations can effect pore size of the anodize, which may interfere with the acceptance of the dye.

## The Water Break Test:

Also recognized as ASTM-F-22

This test is probably one of the most important procedures in any plating or anodizing operation. Make sure you carry out this test after doing all the preparation work, including degreasing and etching in pickles

**To pass the test water will sheet off the part rather than bead off.**

1. Take a cleaned and dried part and set it in a vertical position. Use a spray bottle containing distilled water.

2. Spray the part two to three times from at least 15.24 cm away.

If the part is clean and free of oily residue, the water spray should sheet off (Figure 3).

1. If some oily residue remains, the water will tend to bead on the part Repeat the cleaning process until the part passes the test (Figure 4).

Alternatively, apply several drops of distilled water to the cleaned surfaces. If the surface is inadequately cleaned, the spherical form of the drop is largely retained, and the surface must be cleaned once more. If the water runs on the treated surface, then wetting has been satisfactory and the part is ready for plating.



Figure 3. Oil/dirt film makes water bead up



Figure 4. No oil/dirt film allows water to cover part

## Procedures for Nickel Electroplating:

Using a 5.678 lit **Nickel Electroplating Kit**, which contains two tanks (plating and Degreasing),

The steps to the setup the kit are:

For 11 lit kit, multiply by 2

**Degreasing Tank**

1. Fill the 1 lit beaker with 0.5 lit of Distilled water.
2. Add 80 gr of the SP Degreaser powder

Heat and Mix Well. Degreaser works best at hot temperatures

|  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **PROCEDURE** | **SETUP** | | **OPERATING PARAMETERS** | | | | **EQUIPMENT** | | | **SAFETY** | |
| 1. SURFACE PREPARATION | Buff & Polish for a mirror finish. Bead Blast for a ‘flat’ finish. Nylon Abrasive wheel buff for a ‘scratched brush’ look.  DO NOT ATTEMPT TO PLATE POT METAL DIRECTLY WITH THIS KIT  (Prime first with FLASH COPPER ) | | | | | | | | | | |
| 2. DEGREASING |  | | | | 60- 93 C  No agitation  5 mins immersion  80 gr SP Degreaser 0.5 lit Distilled water | | | 1 x Tank  1 x tank lid  1 x heater  1 x 80 gr SP Degreaser | | |  |
| 3. RINSE IN DISTILLED WATER SPRAY  4 .Water BREAK TEST  5. CALCUCAULATE TOTAL SURFACE AREA AND AMPERAGE REQUIRED (0.07 AMPS per square inch ) | | | | | | | | | | | |
| 6.TANK  MAKEUP |  | | | 45 C  Agitation  1 amp per 16 sq’’ for nickel  Ph= 3.5- 4.5 | | 1 x Heater  1 x Plasstic Tank  1 x tank lid  2 x Nickel Anodes  1 x Air pump  Crystals and brighteners  Distilled Water | | | Wear rubber gloves and goggles | | |
| Plating cycle must not be INTERRUPTED  Or Delamination of subsequent plating may occur | | | | | | | | | | | |
| 7. Plating times | | Immersion time depends on plating thickness  Time Application Plate thickness  15/30 mins Indoor items, decorative. Etc 0.00025’’  30/60 mins Hand tools, guns, nuts & bolts, brackets etc 0.0005’’  60/90 mins Marine, motorcycle , car or outdoor fittings 0.001’’ | | | | | | | | | |
| 1. ADD LOST WATER | | After plating, top up the tank with DISTILLED water to the original waterline. | | | | | | | | | |
| 1. BUFF AND POLISH | | Buff and polish to enhance the finish, using white buffing compound or Blue Be gone Polish | | | | | | | | | |
| 1. WAX | | If the nickel is your finished product, apply a coat of Collinite Metal Wax | | | | | | | | | |

## General Notes and Specifications:

• DC Power supply

• Adjustable current and voltage

• Voltage between 0 and 18 volts

• Current between 0 and 3 amps

• Digital operation

\*\*You can switch the display between volts and amps with the switch to the right of the digital display

## Operation:

1. Wear gloves, safety glass, and observe all clean room policies

2. Prepare stand with materials to be electroplated

3. Fill with electrolyte solution to desired level (Above the sensors)

4. Insert prepared stand into solution between heating coils and tank wall

5. Connect positive terminal (red) to anode (i.e. nickel)

6. Connect negative terminal (black) to cathode (i.e. copper)

7. Slide top into place

8. Connect positive terminal to power supply (+)

9. Connect negative terminal to power supply (-)

10. Turn on heating supply located directly to the right of the right hood

11. Adjust heater to desired temperature and wait for temperature to rise to desired level (max. 70°C)

12. Determine if constant voltage or constant current is desired

13. Check that power supply is plugged in (cord is in back)

14. Press power button on power supply

15. Adjust current or voltage as desired first with the coarse adjustment knob, then with the fine adjustment knob (current is on the left, voltage on the right)

16. The C.C. light will be lit if the device is operating in constant current mode

17. The C.V. light will be lit if the device is operating in constant voltage mode

18. If the C.C. light is not lit, increase the voltage until C.C. is lit and vice versa for the C.V. light

19. At end of electroplating, simply switch off the power

20. Disconnect tank from power supply

21. Remove top

22. Disconnect the cathode and anode and remove stand from tank

23. Return electrolyte solution to its original container and wash tank with DI water

24. Clean up all electrolyte solution

\*\*It is difficult to pour accurately from the tank, so it is best to first pour into a large flask, then into the bottle.

## Precautions:

• Operate between 0 and 40 deg C and less than 80% humidity

• Store between -10 and 70 deg C and less than 70% humidity

• Keep alligator clips and wires out of electrolyte solution

• Wear gloves and eye protection (face protection shield is optional)

• Consult manual if you need to do series or parallel operation of multiple power supplies • If constant voltage operation is desired, you should set the current limit before connecting the power supply to the electroplating tank

## Setting the Current Limit:

1. Determine max safe current for electroplating process

2. Temporarily short the (+) and (-) terminals of the power supply together with a test lead

3. Rotate the coarse voltage control away from zero sufficiently for the C.C. indicator to light

4. Adjust the current control for the desired current limit and read the current value on the Ammeter

5. The current limit (overload protection) has now been preset. Do not change the current control setting after this step

6. Remove the test lead

1. References

<http://www.caswellplating.com/movies.html>

<http://support.caswellplating.com/index.php?/Knowledgebase/List/Index/45>

## Definitions

1. N/A