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# Purpose:

The purpose of this procedure is to describe the process used to plate ceramic crystals with electroless nickel and gold plating.

# Responsibilities:

Crystals Department Engineering is responsible for maintaining this procedure.

Crystals Department Management and Technicians are responsible for carrying out this procedure.

## Affected Department / Product Group / Support Group:

Crystals Department

## Associated Documents:

ISO 9001, QAM, QSM, AS9100

# Procedural Notes

## General Description and Practice

This procedure defines the processes used to apply electroless nickel and gold plating onto ceramic pieces. The plating is used to collect the charge produced by the crystals in their application. Electroless gold plating is applied to protect the nickel plating from oxidizing. The gold replaces the nickel on the outermost surface, so it will not plate onto bare ceramic surfaces. Both the platings are applied by through a chemical process.

Cleanliness is particularly important in any plating process. Any contamination on the utensils or parts being plated may prevent the plating from adhering to the ceramic surface. Therefore, it is very important that once the ceramic pieces have been cleaned prior to plating, the operator should only handle the pieces when wearing latex gloves or finger cots. Likewise, fixtures, beakers, etc., should be handled in the same manner.

## Safety Precautions

NOTE: The Transene Bright Electroless Gold solution contains potassium gold cyanide, a poison. This material must be stored and handled in a fume hood. Handle with extreme caution, avoiding contact with clothing, skin and eyes. Avoid breathing vapors when plating crystals because the plating process generates hydrogen cyanide gas.

The operator may come into contact with the following materials and equipment that requires caution:

* Acid solutions
* Base solutions
* Warm to hot drying ovens (80° – 150°C)
* Boiling water baths
* Volatile solvents

Common sense and good laboratory practice should be used at all times.

IN THE CASE OF ANY INJURY, INFORM YOUR SUPERVISOR IMMEDIATELY.

### Handling Acids and Bases

In any operation that calls for the use of strong acids or bases, use rubber gloves and safety goggles. Avoid all contact, especially the eyes, with these strong solutions and avoid breathing the vapors. Always prepare and use the solutions in the fume hood. Avoid splashing and spills of any of the acid or base solutions. If for any reason some of the acid comes into contact with the skin, flush immediately with water and neutralize with soda from the acid spill kit. An Emergency Eyewash station is available in the lab and should be used if necessary. In the case of any accidents, inform your supervisor. Where there is a major spill not in contact with the skin, call your supervisor and then neutralize an acid with soda or dilute a base with water and then clean as with any spill. When mixing an acid solution, always add the acid to the water.

### Handling Volatiles

With volatile solvents such as methanol, toluene, or acetone, the vapors should not be inhaled. These solvents should be used in a fume hood whenever possible. In addition, most of these solvents are usually extremely flammable and those flames may not be visible.

### Use of Ovens

When inserting into or removing anything from an oven that is warmer than 60°C (140°F), use a set of gloves or mitts and the appropriate utensils that allow the operator to perform the operation without risk of burns.

## Approximate Lot Sizes

This plating process will deposit a thin layer of gold and nickel over the entire surface of the pieces being plated. In order to maintain consistency, use the following lot sizes as a guideline for each plating run:

|  |  |
| --- | --- |
| Part Number | Lot Size |
| 5342-01 and similar | 250 |
| 22446-02 and similar | 125 |
| 22446-01 and similar | 60 |
| 7056-01 and similar | 25 |
| All plate shear strips | 50 |

## Equipment

1000ml flask labeled “Sensitizing Solution”

1000ml flask labeled “Nickel Seeding Solution”

1000ml Pyrex beaker labeled “Nickel Plating Solution A”

1000ml Pyrex beaker labeled “Nickel Plating Solution B”

150mm x 75mm Pyrex crystallizing dish labeled “Nickel Plating Solution”

150mm x 75mm Pyrex crystallizing dish labeled “Etching Solution”

150mm x 75mm Pyrex crystallizing dish labeled “Nitric Acid Solution”

150mm x 75mm Pyrex crystallizing dish labeled “Sensitizing Solution”

150mm x 75mm Pyrex crystallizing dish labeled “Seeding Solution”

150mm x 75mm Pyrex crystallizing dish labeled “E-Gold”

150mm x 75mm Pyrex crystallizing dish labeled “DI Water”

150mm x 75mm Pyrex crystallizing dish labeled “Methanol”

One gallon bottle marked “Nickel Plating Solution A”

One gallon bottle marked “Nickel Plating Solution B”

Hot Plate and Magnetic Stirrers

190mm x 100mm Pyrex dishes with a bed of alumina beads

Plating Fixtures, part numbers 56303-01 and 56304-01, as required

Balance with resolution ±0.05g

Various graduated cylinders (10mL, 25ml, 100mI, 250ml, 500mI)

Polyethylene stirrers (“policemen”)

Glass cover dishes, 150mm dia or similar

Various other beakers as required

Drying Oven, set to 110°C

Digital Thermometer

Plastic-tipped tweezers

Paper towels

Kimwipes

Lab timers

## Chemicals

Reagent grade Stannous Chloride (PCB p/n 100-8879-20)

Reagent grade Palladium Chloride (PCB p/n 100-11515-10)

Reagent grade Nickel Sulphate (PCB p/n 100-8767-40)

Reagent grade Sodium Citrate (PCB p/n 100-8832-80)

Reagent grade Sodium Acetate (PCB p/n 100-8832-90)

Reagent grade Sodium Hypophosphite (PCB p/n 100-8882-40)

Reagent grade Magnesium Sulfate (PCB p/n 100-8791-30)

Reagent grade 37.3% Hydrochloric Acid (PCB p/n 100-8374-30)

Reagent grade 0.52-0.60% Hydrofluoric Acid (PCB p/n 100-11240-41)

Transene Bright Electroless Gold Solution (PCB p/n 100-8766-00)

50/50 Solution of Reagent grade 69.7% Nitric Acid (PCB p/n 100-8374-20) and DI water

50/50 Solution of Ammonium Hydroxide (PCB p/n 100-8374-40) and DI water

Methanol (PCB p/n 100-8374-70)

DI water

Tap water

# Preparation of Solutions

NOTE: It is acceptable to make different quantities of solution as required, as long as the concentration of the solution is the same as below.

## Etching Solution

NOTE: The etching solution contains strong acids. Hydrofluoric acid is particularly hazardous. Operators must wear gloves, goggles and shop/lab coats when handling these chemicals.

* + 1. Using a 500mL graduated cylinder, measure 900mL of Dilute Hydrofluoric Acid and pour it into the Nalgene container.
    2. Using a 100mL graduated cylinder, measure 50mL of Hydrochloric Acid and add it to the Dilute HF Acid.
    3. Cover the container with the appropriate lid, label the container “Etching Solution” and store it under the fume hood.

## Sensitizing Solution

* + 1. Using the 250mL graduated cylinder, measure 200mL of DI water and pour it into a 1000mL flask labeled “Sensitizing Solution.”
    2. Using the balance, weigh out 2.0g of Stannous Chloride and add it to the 1000mL flask.
    3. Put the flask on the stir plate and stir until the Stannous Chloride is dissolved (30 min to 1 hr).

## Seeding Solution

* + 1. Using the 250mL graduated cylinder, measure 500mL of Dl water and pour it into the 1000ml flask marked “Seeding Solution.”
    2. Using the 10mL graduated cylinder, measure 5mL of Hydrochloric Acid and add it to the 1000ml flask.
    3. Using the balance, weigh out 0.16g of Palladium Chloride and add it to the 1000mL flask.
    4. Put the flask on the stir plate and stir until the Palladium Chloride is dissolved (30 min to 1 hr).

## Nickel Plating Solution A

* + 1. Using the 500mL graduated cylinder, measure 750ml of Dl water and pour it into the 1000mL beaker labeled “Nickel Plating Solution A”.
    2. Using the balance, weigh out 52.8g of Nickel Sulphate and add it to the beaker.
    3. Put the beaker on the stir plate and stir until the Nickel Sulphate is dissolved (30 min to 1 hr).
    4. Pour the entire contents of the beaker into the one gallon bottle labeled “Nickel Plating Solution A.”

## Nickel Plating Solution B

* + 1. Using the 500mL graduated cylinder, measure 750ml of Dl water and pour it into the 1000mL beaker labeled “Nickel Plating Solution B”.
    2. Using the balance, weigh out 15.0g of Sodium Citrate and add it to the 1000mL beaker.
    3. Using the balance, weigh out 15.0g of Sodium Acetate and add it to the 1000mL beaker.
    4. Using the balance, weigh out 22.3g of Sodium Hypophosphite and add it to the 1000mL beaker.
    5. Using the balance, weigh out 30.0g of Magnesium Sulphate and add it to the 1000mL beaker.
    6. Stir the beaker with a policeman until all of the solids are dissolved.
    7. Pour the entire contents of the beaker into the one gallon bottle marked “Nickel Plating Solution B.”

## Preparing the Nickel Bath

* + 1. Cover the bottom of a 190mm x 100mm crystallizing dish with alumina grinding media, then fill to ¾ full with DI water.
    2. Place the dish onto a hot plate and set the heat dial to 185°C.
    3. Measure equal parts of Plating Solution A and Plating Solution B using the 100ml graduated cylinder and add them to the 150mm x 75mm dish marked “Nickel Plating Solution.”
    4. For 5342-01 and similar parts, and all shear plate strips, measure 100ml of Part A and 100ml of Part B.
    5. For 22446-01 and 7056-01 and similar parts, use 200ml of each solution.
    6. Place the beaker of nickel plating solution into the water-filled crystallizing dish to form a “double boiler”.
    7. Measure the temperature of the mixture using the digital thermometer; it should be 75–80°C.

## Preparing the Gold Bath

* + 1. Cover the bottom of a 190mm x 100mm crystallizing dish with alumina grinding media, then fill to ¾ full with DI water.
    2. Place the dish onto a hot plate and set the heat to 175°C.
    3. Pour enough of the Transene solution into the 150mm x 75mm dish labeled “E-Gold” to cover the parts.

NOTE: When plating shear plate strips, place the appropriate fixture into the bath so it heats up along with the solution.

* + 1. Place the 150mm dish containing gold solution into the water-filled crystallizing dish to form a “double boiler”.
    2. Measure the temperature of the mixture using the digital thermometer; it should be 75–80ºC.

# Plating Procedure

NOTE: Thoroughly drain parts when transferring from one solution to another, or from rinsing into solutions, to avoid transferring or diluting any solution. Do not dry in an oven unless instructed to do so.

## Cleaning Parts Received from Machining

NOTE: Due to the many variables involved in cleaning the machined parts, it may be necessary to deviate from the times specified in the following section or repeat portions of it. The times and sequence stated below should be used as a guideline for the cleaning process. If problems cleaning parts persist, contact your Supervisor, Engineer, or Lead Technician.

* + 1. Clean the parts for 30 minutes in Acetone, using ultrasonics
    2. Pour off the used Acetone in the appropriate waste container.
    3. Replenish the dish with fresh Acetone and clean for another 30 minutes, using ultrasonics.
    4. Pour off the used Acetone in the appropriate waste container.
    5. Clean the parts for 30 minutes in Micro 90 Cleaning Solution, using ultrasonics.
    6. Discard the cleaning solution in the appropriate waste container.

NOTE: DO NOT POUR MICRO 90 CLEANING SOLUTION INTO THE DI WATER SYSTEM.

* + 1. Fill the dish with DI water and clean for 30 minutes, using ultrasonics.
    2. Discard the rinse water into the appropriate waste container.

NOTE: DO NOT POUR MICRO 90 CLEANING SOLUTION INTO THE DI WATER SYSTEM.

* + 1. Clean the parts for 30 minutes in Methanol, using ultrasonics.
    2. Pour off the used methanol in the appropriate waste container.
    3. Place the dish in the Crystals drying oven until dry.

## Chemical Etch

NOTE: Chemical etching is done on the ceramic pieces to improve nickel adhesion. Operators must wear gloves, goggles and shop/lab coats when handling these chemicals. Avoid splashing the solution when handling.

* + 1. Carefully transfer the parts into the crystalizing dish labeled “Etching Solution”.
    2. Add enough Etching Solution to the dish to cover the parts.
    3. Soak the parts in the solution for 1-2 minutes.
    4. Gently stir the solution as the parts soak.
    5. Pour off the solution into a 1000mL plastic beaker for temporary storage during the process.
    6. Fill the dish with DI water, agitate lightly to rinse, then pour off the water.
    7. Fill the dish with DI water, clean in the ultrasonic cleaner for 1 minute, then pour off the water.
    8. Fill the dish with DI water, clean in the ultrasonic cleaner for 1 minute, then pour off the water.

NOTE: It is permissible to repeat the DI water rinsing process until the rinse water is clean.

NOTE: It is permissible to place the crystals in the drying oven, dry and hold them at this point until the plating process can be completed.

## Cleaning

* + 1. Carefully transfer the parts to the crystalizing dish labeled “50/50 Nitric Acid Solution”.
    2. Add enough 50/50 Nitric Acid solution to the dish to cover the parts.
    3. Soak the parts in the solution for 1-2 minutes.
    4. Gently stir the solution as the parts soak.

NOTE: From this point on, use only clean tweezers and policemen to touch the parts to ensure proper plating. Rinse the tweezers with DI water after each step. Separate rubber policemen are to be used for each step.

* + 1. Pour off the solution into a 1000mL plastic beaker for temporary storage during the process.
    2. Fill the dish with DI water, agitate lightly to rinse, then pour off the water.
    3. Fill the dish with DI water, clean in the ultrasonic cleaner for 1 minute, then pour off the water.
    4. Fill the dish with DI water, clean in the ultrasonic cleaner for 1 minute, then pour off the water.

NOTE: After pouring off the water, the remainder of the plating process must be performed in immediate succession, until the parts are nickel plated.

NOTE: Due to the many variables involved in plating, it may be necessary to deviate from the times specified in the following sections. The times stated in this procedure should be used for the first plating run. If the parts do not plate or the nickeling solution turns black, contact the leadperson or engineer. Adjustments will first be made to the rinse times to address problems. If the problems persist, adjustments to the sensitizing and seeding times will be made.

## Sensitizing

* + 1. Carefully transfer the parts to the crystalizing dish labeled “Sensitizing Solution”.
    2. Lightly agitate the storage flask marked “Sensitizing Solution” to mix the solution.
    3. Add enough Sensitizing Solution to the dish to cover the parts.
    4. Soak the parts in the solution for no more than 3 minutes.
    5. Gently stir the solution every 20 seconds using a policeman.

NOTE: Shear plate strips need to be flipped over during this process.

NOTE: While the parts are sensitizing, make sure the dish marked “DI Rinse” is rinsed clean.

* + 1. Pour off the solution into a 1000mL plastic beaker for temporary storage during the process.

NOTE: The following steps must be done in immediate succession until the parts are immersed in the seeding solution.

* + 1. Carefully transfer the parts to the dish labeled “DI Rinse”.
    2. Rinse the parts using overflowing DI water for 1–5 minutes.
    3. Pour off the water from the dish and drain the excess water from the parts.

## Seeding

* + 1. Carefully transfer the parts to the crystalizing dish labeled “Seeding Solution”.
    2. Add enough Nickel Seeding Solution to the dish to cover the parts.
    3. Soak the parts in the solution for no more than 3 minutes.
    4. Gently stir the solution every 20 seconds using a policeman.

NOTE: Shear plate strips need to be flipped over during this process.

* + 1. Pour off the solution into a 1000mL plastic beaker for temporary storage during the process.

NOTE: The following steps must be done in immediate succession until the pieces are immersed in the nickeling bath.

* + 1. Carefully transfer the parts to the dish labeled “DI Rinse”.
    2. Rinse the parts using overflowing DI water for 1–5 minutes.
    3. Pour off the water from the dish and drain the excess water from the parts.

## Nickel Plating

* + 1. Carefully transfer the parts to the heated bath of nickel solution prepared earlier.
    2. Refer to the router for plating time and set the timer accordingly.

NOTE: Plating will proceed with the evolution of hydrogen gas. The parts should be agitated constantly to keep hydrogen bubbles from clinging to the surface and preventing plating in those areas.

NOTE: As the plating process proceeds, the pieces will turn black or dark gray from their original color, then they will slowly turn silver-gray.

NOTE: If the solution bubbles excessively and turns black, it is over-plating and adjustments need to be made as stated earlier. Strip the nickel and replate.

* + 1. Pour off the used plating solution into a container labeled “Waste Nickel” and allow it to cool.
    2. Rinse the parts using overflowing tap water for 1-5 minutes, then pour off the water.

NOTE: The first rinse is done using tap water to deactivate any iron contaminants.

* + 1. Fill the dish with tap water, clean in the ultrasonic cleaner for 1 minute, then pour off the water.
    2. Half fill the dish with DI water and add a couple of squirts of 50/50 ammonia.
    3. Agitate the parts slowly, then clean in the ultrasonic bath for 1 minute, then pour off the water.
    4. Fill the dish with DI water, clean in the ultrasonic cleaner for 1 minute, then pour off the water.
    5. Add enough clean DI water to the dish to cover the parts.

NOTE: As parts are transferred from their original dish, make sure to transfer the job number to the new dish.

NOTE: At this point, the nickel plated crystals can sit for no longer than 1 hour in clean DI water before being gold plated.

## Gold Plating

NOTE: From this point on, use only clean plastic tweezers and policemen to touch the parts to ensure proper plating. Rinse the tweezers with DI water after each step.

* + 1. Carefully transfer the parts to the heated bath of gold solution prepared earlier.

NOTE: When plating shear strips, transfer the parts directly to the plating fixture.

NOTE: When plating all other parts, spread them out so their edges are not touching.

* + 1. Refer to the router for the plating time and set the timer accordingly.
    2. Gently agitate the solution by using the plastic stirrer.

NOTE: The parts should begin to turn gold in the first few minutes.

* + 1. Fill the dish labeled “DI water” with DI water, if it is not already.
    2. After plating for the designated time, remove the parts from the solution.

NOTE: When plating small parts, pour off the gold solution into another dish, then carefully transfer the parts to the DI water, then replace the gold solution in the bath.

NOTE: When plating larger parts, remove the parts using platics tweezers and place them directly into the DI water.

NOTE: When plating shear plate strips, remove the strips only from the plating fixture using plastic tweezers and place the strips in a dish containing a plating fixture and D.I. water.

* + 1. Gently agitate the parts to rinse, and then remove the parts from the water in a similar manner.
    2. Fill the dish labeled “Methanol” with Methanol, if it is not already.
    3. Carefully transfer the parts to the dish, gently rinse the parts, then pour off the methanol into another dish.
    4. Place the parts onto a clean glass dish and dry in the oven for at least 10 minutes.

NOTE: The glass tray needs to be labeled with the job number and time into the oven using a Sharpie.

## Cleanup

* Clean and rinse all dishes and trays, and store appropriately.
* Pour the etching solution back into its nalgene container and return it to storage underneath the fume hood.
* Return the 50/50 Nitric Acid Solution to its storage container.
* Pour the sensitizing solution back into the flask marked “Sensitizing Solution.”
* Pour the seeding solution back into the bottle marked “Nickel Seeding Solution.”
* Once cool, pour the spent nickeling solution into the waste nickel solution container.
* Dispose of any waste paper towels contaminated with the gold solution by wrapping them up in rubber gloves prior to discarding in the trash.
* Pour the methanol into the waste solvent drum after the plating run is entirely complete.
* Store the used gold solution in an appropriately labeled plastic bottle for future use.

NOTE: The Gold solution can be reused many times until depletion occurs. Depleted solution should be stored in an appropriately labeled plastic bottle for disposal.