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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 DEPARTMENTS / PRODUCT GROUPS / SUPPORT GROUPS

Crystals Department

ASSOCIATED DOCUMENTS

ISO 9001, QAM, QSM, AS9100

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 nitrile 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 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, yellow aprons and sleeves 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 and shower 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 VOLATILE SOLVENTS**

* With volatile solvents such as methanol, or acetone, the vapors should not be inhaled. These solvents should be used in a fume hood whenever possible.
* Most of these solvents are usually extremely flammable and those flames may not be visible.
* These solvents should not be used in the same fume hood as or come into contact with any acids.

**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 |

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.*

**SENSITIZING SOLUTION**

1. Using the 500mL graduated cylinder, measure 500mL of DI water and pour it into a 1000mL flask labeled “Sensitizing Solution.”
2. Using the 100mL graduated cylinder, measure 9mL of Tin Tetrafluoroborate and add it to the 1000mL flask.
3. Stopper the top of the flask.

**SEEDING SOLUTION**

1. Using the 500mL 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 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 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 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 B.”

**PREPARING THE NICKEL BATH**

1. Check that the Nickel plating water bath, 190mm x 100mm crystallizing dish with alumina grinding media covering the bottom, is ¾ full with DI water.
2. Place the dish onto a hot plate and set the heat dial to 185°C.
3. Measure out 200ml of Nickel plating solution part A and 200ml of Nickel plating solution part B and add them to the 150mm x 75mm dish marked “Nickel Plating Solution.”
4. Place the dish of nickel-plating solution into the water bath to form a “double boiler”.
5. Measure the temperature of the mixture using the digital thermometer; it should be 75–80°C.

**PREPARING THE GOLD BATH**

1. Check that the Gold plating water bath, 190mm x 100mm crystallizing dish with alumina grinding media covering the bottom, is 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.
4. Place the 150mm dish containing gold solution into the water-filled crystallizing dish to form a “double boiler”.
5. 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.*

*NOTE: 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. It is acceptable to use the acetone that the parts came in.
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. Repeat steps 3 and 4 until the acetone is clear after washing.
6. Clean the parts for 30 minutes in Micro 90 Cleaning Solution, using ultrasonics.
7. 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 DI sink drain.

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

1. If holding parts before plating continue to Step 11 below. Normally continue with **Cleaning of Etched Grains** to plate parts right away.
2. Clean the parts for 30 minutes in Methanol, using ultrasonics.
3. Pour off the used methanol in the appropriate waste container.
4. Place the dish in the Crystals drying oven until dry.

CLEANING OF ETCHED GRAINS

*NOTE: Chemical cleaning/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 to the crystalizing dish labeled “50/50”.
2. Add enough 50/50 Nitric Acid solution to the dish to cover the parts.
3. Soak the parts in the solution for 1 minute.
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. Use a Separate rubber policemen 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, and rinse the parts using overflowing DI water for 3 minutes.
3. Pour off the water from the dish and drain the excess water from the parts.

*NOTE: After pouring off the water, the remainder of the plating process must be performed in IMMEDIATE succession, until the parts are nickel plated. DO NOT LET THE PARTS DRY AT ALL BETWEEN OPERATIONS.*

SENSITIZING

1. Carefully transfer the parts to the crystalizing dish labeled “Ni Sens”.
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 3 minutes.
5. Gently stir the solution every 20 seconds using a policeman.

*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 3 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 “Ni Seed”.
2. Add enough Nickel Seeding Solution to the dish to cover the parts.
3. Soak the parts in the solution for 3 minutes.
4. Gently stir the solution every 20 seconds using a policeman.
5. 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 3 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. The plating solution will quickly go from a light green to a dark green to black if you are not agitating enough.*

*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.*

******

Figure 22446-01 Showing Good Nickel Plating

*NOTE: If the solution bubbles excessively and turns black, it is over-plating. Strip the nickel and re-plate.*

1. Pour off the used plating solution into a container labeled “Waste Nickel” and allow it to cool.

*NOTE: Nickel plating solution will be used only one time and then discarded. This means that it may need to be made up several times to plate a single job.*

1. Rinse the parts using overflowing DI water for 3 minutes, then pour off the water.
2. Fill the dish with DI water, clean in the ultrasonic cleaner for 1 minute, and then pour off the water.
3. 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.*

*NOTE: Parts must stay submerged until transferred (wet) into the gold plating solution.*

GOLD PLATING

*NOTE: Only use clean plastic tweezers and policemen to touch the parts to ensure proper plating. Rinse the tweezers with DI water after each step.*

1. Check that the gold plating bath is between 75°C and 80°C, adjust the hotplate if necessary.
2. Carefully scoop the parts directly from the DI water into the heated bath of gold solution prepared earlier. Some extra DI water will not negatively affect the gold plating process.

*NOTE: Spread all parts out so their edges are not touching.*

*NOTE: Small parts with blind cavities, such as 17744-01, should be placed on their edge for plating.*

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, inspect the parts.
	1. If the plating looks complete, uniform and yellow, remove the parts from the solution.
	2. If the plating looks thin, grayish, allow the parts to plate up to five minutes longer and then replace the gold plating solution before plating more parts.
	3. If the parts still have not plated after the additional five minutes, parts are to be stripped and re-plated.

*NOTE: It is normal for the solution to turn blue when parts are added and to remain blue after parts are removed.*

*NOTE: When plating parts 22446-01 sized parts and smaller, pour off the gold solution into another dish, then carefully transfer the parts to the DI water, and then replace the gold solution in the bath.*

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

1. If the plating solution in the dish gets too low to cover parts, it can be replenished with some additional plating solution. Check that the solution is back at temperature before continuing with plating.
2. Tape test an AQL sample, per Table 1, of finished parts per each plating run. If the parts fail the tape test, strip the plating off the parts and then re-plate the parts starting at **Cleaning of Etched Grains**.

**Table 1: AQL 0.065 Level I Sampling Plan for Major Defects**

|  |  |
| --- | --- |
|  | **0.065 Single Normal** |
| **LOT SIZE** | **SAMPLE SIZE Ac Re** |
| 1. to 8
 | 2 0 1 |
| 1. to 15
 | 2 0 1 |
| 1. to 25
 | 3 0 1 |
| 1. to 50
 | 5 0 1 |
| 1. to 90
 | 5 0 1 |
| 1. to 150
 | 8 0 1 |
| 1. to 280
 | 13 0 1 |
| 1. to 500
 | 20 0 1  |
| 1. to 1200
 | 32 0 1 |
| 1. to 3200
 | 50 0 1 |
| 1. to 10000
 | 80 0 1 |
| 10001 to 35000 | 125 0 1 |



Figure Good gold-plated 22446-01s. Plating looks complete, uniform and yellow.

CLEANUP

1. Clean and rinse all dishes and trays, and store appropriately.
2. Return the 50/50 Nitric Acid Solution to its storage container.
3. Pour the sensitizing solution back into another flask marked “Used Sensitizing Solution” for future use. Once this solution becomes spent, pour into the waste nickel solution container.
4. Pour the seeding solution back into another bottle marked “Used Nickel Seeding Solution” for future use. Once this solution becomes spent, pour into the waste nickel solution container.
5. Once cool, pour the spent nickeling solution into the waste nickel solution container.
6. Dispose of any waste paper towels contaminated with the gold solution by wrapping them up in rubber gloves prior to discarding in the trash.
7. Pour the methanol into the waste solvent drum after the plating run is entirely complete.
8. 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.*

APPENDIX: EQUIPMENT AND CHEMICALS

|  |  |
| --- | --- |
| **EQUIPMENT** | **CHEMICALS** |
| 1000ml flask labeled “Ni Sensitize Solution” | Reagent grade Tin Tetrafluoroborate, 50% w/w Aqueous (PCB p/n 100-17230-20) |
| 1000ml flask labeled “Ni Seed Solution” | Reagent grade Palladium Chloride (PCB p/n 100-17230-10)  |
| 1000ml Pyrex beaker labeled “Nickel A” | Reagent grade Nickel Sulphate (PCB p/n 100-17229-70) |
| 1000ml Pyrex beaker labeled “Nickel B” | Reagent grade Sodium Citrate (PCB p/n 100-17230-00) |
| 150mm x 75mm Pyrex crystallizing dish labeled “50/50” | Reagent grade Sodium Acetate (PCB p/n 100-17229-80) |
| 150mm x 75mm Pyrex crystallizing dish labeled “Ni Sens” | Reagent grade Sodium Hypophosphite (PCB p/n 100-17229-60) |
| 150mm x 75mm Pyrex crystallizing dish labeled “Ni Seed” | Reagent grade Magnesium Sulfate (PCB p/n 100-17229-90) |
| 150mm x 75mm Pyrex crystallizing dish labeled “Nickel” | Reagent grade 37.3% Hydrochloric Acid (PCB p/n 100-8374-30) |
| 150mm x 75mm Pyrex crystallizing dish labeled “E-Gold” | Transene Bright Electroless Gold Solution (PCB p/n 100-8766-00) |
| 150mm x 75mm Pyrex crystallizing dish labeled “DI Water” | 50/50 Solution of Reagent grade 69.7% Nitric Acid (PCB p/n 100-8374-20) and DI water |
| 150mm x 75mm Pyrex crystallizing dish labeled “Methanol” | Methanol (PCB p/n 100-8374-70 / 100-17228-40) |
| One gallon bottle marked “Nickel “A” | DI water |
| One gallon bottle marked “Nickel “B” |  |
| Hot Plate and Magnetic Stirrers |  |
| 2x 190mm x 100mm Pyrex dishes with a bed of alumina beads, for hot water baths |  |
| 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 90°C |  |
| Digital Thermometer with probe |  |
| Plastic-tipped tweezers |  |
| Paper towels |  |
| Kim wipes |  |
| Lab timers |  |