SOP: Electroless Zinc, Nickel, Gold plating on CMOS IC

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Minghao Li

Description

This SOP shows the detailed procedure for electroless Zn, Ni and Au plating on aluminum pads on CMOS IC. The cleaning part of this SOP is mainly based on the SOP [1] prepared by Dr Henry Lancashire, the etching, double zinc, nickel plating is inspired by reference [2] and the gold plating part uses the commercial solution.

In summary, the CMOS die is cleaned to remove organic contaminants, then it is etched slightly to remove aluminum oxide and to expose fresh aluminum. An intermediate double zincation step is used to replace a thin layer of aluminum with zinc to not only permit subsequent deposition and coating adhesion of a nickel film, but also prevent the aluminium from re-oxidation. Nickel is then deposited onto the zinc layer. The duration of this Ni deposition step determines the thickness of Ni, which, in turn, determines the thickness of the Au film, because the Au replaces the Ni.

Materials

- Sodium hydroxide (10% **NaOH**): 1 g in 10 mL DI water
- Nitric acid (20% **HNO3**): 2.2 mL in 20 mL (68% density) DI water
- Nickel sulphate (**NiSO4**): 0.28 g in 10 mL (28.8 g/l) DI water
- Sodium hypophosphite (**NaPO2H2**): 0.27g in 10 mL (27 g/l) DI water
- **Zn** eletroless plating solution ("Xenolyte Zincate CFA" from Xenolyte – use as it is)
- **Ni** eletroless plating solution (mix NiSO4 and NaPO2H2 in a 1:1 volumetric ratio)
- **Au** eletroless plating solution (The "Bright gold electroless plating solution", Sigma Aldrich)
- Isopropyl alcohol IPA (IUPAC name propan-2-ol)
- Acetone (IUPAC name propan-2-one)
- Deionised Water, dH2O
- Carbon-fibre tip forceps
- Glass Beakers with DI water (x6)

Read before start!

* Nickel sulphate (**NiSO4**) is a yellow crystal chemical which takes 1 day to be disolved in the water, Nickel sulfate hexahydrate (**NiSO4·6H2O**) is a blue crystal chemical which is much easier to be disolved. It's better to replace NiSO4 with NiSO4·6H2O. If you're going to use Nickel sulphate, make sure it is completely disolved. You can check this by keeping it still for 5 minutes until you do not see any precipitation. The solution will be clear green.
* This recipe has been proved to be repeatable when tried on two chips: Mooncake and Damocles.
* Never use nitrogen gun to clean the chip as it will break the metal structure on your chip!
*Be careful to use oxygen plasma as well, the silicon come out on one chip was found after oxygen plasma.
* Do not use silicon or glass as chip carrier in this process, as it is likely to react with some of the solutions (Dilute solutions of sodium hydroxide can etch silicon slowly, forming silicon dioxide (SiO2) and hydrogen gas., acetone can soften and weaken the epoxy) used in this recipe.
* To use this SOP you must read the relevant MSDS documents, these are available on RISKNet.

Step A: Cleaning

1. Clean in Acetone
    - Prepare 1 glass beaker containing acetone sufficient to cover the sample to be plated.
    - Place the sample in the acetone using carbon-fibre tip forceps.
    - Place the beaker in an ultrasound bath containing dH2O to an equal level to the beaker.
    - Ultrasound for 30 seconds.
2. Clean in IPA
    - Prepare 1 glass beaker containing IPA sufficient to cover the sample to be plated.
    - Place the sample in the acetone using carbon-fibre tip forceps.
    - Place the beaker in an ultrasound bath containing dH2O to an equal level to the beaker.
    - Ultrasound for 60 seconds.
3. Clean in DI water
    - Prepare 1 glass beaker containing dH2O sufficient to cover the sample to be plated.
    - Place the sample in the acetone using carbon-fibre tip forceps.
    - Place the beaker in an ultrasound bath containing dH2O to an equal level to the beaker.
    - Ultrasound for 2 minutes.

Step B: Aluminum Etching

NaOH is used as the Aluminum etchant as it is a mild alkaline solution.

1. Put the chip in a petri dish.
2. Put a few drops of the solution using pipette on the chip until it covers the entire chip.
3. Keep the chip under this condition for 2 minutes at room temperature.
4. Use dust-free paper to soak up the NaOH on the chip without touching it.
5. Using carbon-tip tweezers to place the chip in a glass beaker of dH2O for 1 minute.
6. Holding the chip using carbon-tip tweezers outside the petri dish and rinse it above a beaker for 30 seconds to prevent water from flowing everywhere.
7. Leave the chip in the DI water until next step.

Step C: Double Zinc process

1. Set the temperature of oven at 55 °C.
2. First Zinc:
    - Place the chip in the glass beaker of Zincate.
    - Place the glass beaker into the oven for 1 minute.
    - Take the chip out of the glass beaker.
    - Holding the chip using carbon-tip tweezers and place it directly above an empty beaker and rinse with DI water for 1 minute.
3. Remove
    - Put the chip in a petri dish.
    - Put a few drops of HNO3 to cover the sample.
    - Keep it for 45 seconds at room temperature.
    - Take the chip out of the glass beaker.
    - Holding the chip using carbon-tip tweezers and place it directly above an empty beaker and rinse with DI water for 1 minute.

4. Second Zinc:
    - Place the chip in the glass beaker of Zincate.
    - Place the glass beaker into the oven for 1 minute.
    - Take the chip out of the glass beaker.
    - Holding the chip using carbon-tip tweezers and place it directly above an empty beaker and rinse with DI water for 1 minute

Step D: Nickle Plating

1. Set the temperature of oven at 85 °C.
2. Nickle plating
    - Place the chip in the glass beaker of Nickle plating solution.
    - Place the glass beaker into the oven for 25 minute.
    - Take the sample out and wash in the ultrasonic bath.
    - Holding the chip using carbon-tip tweezers and place it directly above an empty beaker and rinse with DI water for 1 minute

Step E: Gold Electroless Plating

1. Set the temperature of oven at 90 °C.
2. Gold plating
    - Place the chip in the glass beaker of gold plating solution.
    - Place the glass beaker into the oven for 15 minutes.
    - Take the chip out and wash in the ultrasonic bath.
    - Holding the chip using carbon-tip tweezers and place it directly above an empty beaker and rinse with DI water for 1 minute

Step F: Characterization

A scanning electron microscopy (SEM), atomic force microscopy (AFM) and energy dispersive analysis of x-rays (EDX) can be used to characterize the morphology of sensor surface. The operation details can be found in SOP: SEM, EDX and AFM operation manual. It is important to follow the written guideline in these documents to set the correct parameters for measurement.

The typical composition percentage value of electroless zinc and nickel can be found in [3][4] to be 10 wt.% and 90 wt.% respectively. The findings are in line with the studies of Lin et al. [5], who found that zincating for more than a minute gave a high density and thick zinc layer, resulting in a rough surface.

For nickel plating, It can be clearly seen from [4] that the intensity of the nickel element peaks continuously increases with the electroless plating time. The nickel particle become bigger and bigger with time. The EDX results followed by this SOP has proved the phenomenon stated above. The typical mean value and standard deviation of the percentage of each metal after the above stages are as follows

EDX results

Zn： 10.22±1.56 %
Ni: 89.20±0.51 %
Au: 97.15±1.06 %

AFM is used as a measurement tool to test the morphology of the sample, so that we can get the roughness and thickness of different metal layers. The thickness of the layers deposited using the above SOP can be calculated by comparing the difference height with the passivation layer around the electrode. The typical RMS roughness and thickness values are as follows.

rms area roughness:

Al: 11.2 nm
Zn: 125.7 nm
Ni: 72.3 nm
Au: 43.8 nm

Layer thickness:

Zn: 103 nm
Ni: 692 nm

Note: The AFM results is based on one Mooncake and one Damocles, it will be updated after testing more chip.

References:

1. SOP: Gold Plating of ASICs, Henry Lancashire, 2019
2. S. Hwang, et al, CMOS Microelectrode Array for Electrochemical Lab-on-a-Chip Applications, in *IEEE Sensors Journal*, vol. 9, no. 6, pp. 609-615, June 2009.
3. Othman, et al. Impact of single and double zincating treatment on adhesion of electrodeposited nickel coating on aluminium alloy 7075. Journal of Advanced Manufacturing Technology. 2008.
4. Wei shang, et al. Deposition mechanism of electroless nickel plating of composite coatings on magnesium alloy. vol. 207, pp. 1299-1308, 2019.
5. A.M. Elsherik and U. Erb, Synthesis of Bulk Nanocrystalline Nickel by Pulsed Electrodeposition", Journal of Materials Science, vol. 30, no.33, 1995.