NITRIC ACID CONTROL PROCEDURES

Nitric acid solutions used to strip and passivate electroless nickel plating tanks may become ineffective if their nickel content becomes too high or their acid content becomes too low. These solutions can be controlled by analyzing for these components using the following procedures. Nitric acid passivation solutions are typically made up at a concentration of 30 percent by volume.

Acidity. The acidity of nitric acid solutions is determined by titrating a sample with a 1 normal sodium hydroxide solution. The procedure for this titration is described in the following:

Reagents

1. 1N NaOH solution
2. Methyl orange indicator

Procedure

1. Pipette 5 mL of the nitric acid solution into a 250 mL Erlenmeyer flask.
2. Add 3 or 4 drops of methyl orange indicator.
3. Titrate the sample with 1N NaOH solution until the color of the solution changes from red to yellow.
4. Calculate the acidity of the nitric acid solution from the volume of NaOH solution used.

Calculation

(mL 1N NaOH) x 1.27 = percent HNO_3 in solution (by volume)

To be effective, the strength of a nitric acid stripping solution should be maintained above about 15 percent, although it will *slowly* remove plated out electroless nickel even at acidities as low as 5 percent.

Nickel Content. When necessary, the nickel content of nitric acid solutions is determined by titrating a sample with a 0.1 normal EDTA solution. These titrations are usually only required when the solution's color is dark green and its nickel content exceeds 10 g/L. The procedure for this titration is described in the following:

Reagents

1. 0.1N EDTA solution
2. 50 percent (by volume) ammonium hydroxide solution
3. Murexide indicator

Procedure

1. Pipette 10 mL of the nitric acid solution into a 100 mL volumetric flask and dilute it to 100 mL volume with deionized water (to make a one to 10 dilution).
2. Mix the diluted solution carefully.
3. Pipette 5 mL of the diluted solution into a 250 mL Erlenmeyer flask.
4. Dilute the sample with about 50 mL of deionized water.
5. Add approximately 25 mL of 50 percent ammonium hydroxide to the sample. Its color should change from green to blue.
6. Add a small amount of murexide indicator to the sample until its color changes to a pale straw color.
7. Titrate the sample with 0.1N EDTA solution until the color of the solution changes to violet.
8. Calculate the nickel content of the nitric acid solution from the volume of EDTA solution used.

Calculation

(mL 0.1N EDTA) x 1.174 x 10 = g/L Ni in solution

Nitric acid stripping solutions can contain significant amounts of nickel metal and still provide effective passivation. For ease of control and waste treatment, it is usually good practice to maintain the nickel concentration of the solution below 50 g/L.

One method of optimizing the performance and life of nitric acid solutions is to *bleed and feed* them, by periodically removing about 5 percent of the old solution and replacing that amount with concentrated nitric acid.

REFERENCES

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2. Gregoire Gutzeit, Outline of the Chemistry Involved in the Process of Catalytic Nickel Deposition From Aqueous Solution, Plating, Vol 46, No 10 (1959) p 1158.

3. Ron Duncan, unpublished data.