MTL	STANDARD OPERATING PROCEDURE	**Revision Number**	
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	5/18/2022	Made steps more generalized.
Title	Procedure for Room-Temperature Metallization of Electroless Nickel Plating	

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1. Summary

- This procedure outlined below will describe how to perform room-temperature metallization (RTM) of electroless nickel plating. The process includes the methods to prepare parts to be plated as well as the plating process itself. In good conditions, the nickel will deposit at 2.54 microns (0.1 mils) over 20 minutes of plating time.

2. Scope

- This procedure applies to the RTM electroless nickel kit purchased from Transene® for the plating of ceramics, and other dielectric materials. Plating this way also extends to certain metals compatible with nickel.

Room Temperature Metallizing for Alumina

Room-Temperature nickel metallizing process for alumina ceramics and other dielectric materials – strongly bonded nickel can be plated, soldered, brazed, or welded.

UNIQUE FEATURES

- Low-cost metallization process.
- Excellent bond strength – 100 psi typical.
- Withstands temperature extremes: – 65 °C. to + 850 °C.
- Passes Mil specifications for temperature cycling and shock.
- Hermeticity – leak rate < 10—8cc helium/second.
- Permits registration of metallization patterns with good resolution using photolithographic techniques and mechanical masking.
- Process applicable to beryllia, barium titanate, ferrites, epoxies, mylar and other dielectrics.

The nickel deposit is found to be unstressed, highly conductive, and extremely adherent with strong bond strength exhibited from –60 °C. to + 850 °C. It has a hardness of at least 500 (Vickers) as deposited. The adherent nickel deposit may be plated, soldered, brazed, or welded.

Applications

The RTM process is applicable to metallizing alumina substrates in hybrid and microcircuits, insulated heat sinks, ceramic tubes, windows and terminals. Strong bond structures are produced. Structures with hermetic seals are formed as well.

The RTM process is also useful for metallizing barium titanate and ferrites. In addition, many polymeric materials, notably polyesters, epoxies, cellulosic, mylar and acrylonitrile-butadiene-styrene (ABS terpolymer) may be metallized following suitable modifications of the etch (Solution A).

RTM lends itself productively to the application of photolithographic technology using photoresist materials to register metallization patterns. In this manner, conductive pads may be formed for thin film and hybrid circuits. Base plates for building capacitor structures are also quite feasible.

Method of Plating

The RTM process involves first etching the surfaces of the part to be treated, to increase surface roughness and improve nickel adhesion. This is material dependent, but usually involves an acid to partially dissolve the surface layers.

Post-etch, the part is sensitized and activated catalytically for the nickel. This is done with palladium chloride, a tried-and-true activator.

Finally, the palladium acts as auto catalyst for the nickel sulfate solution to reduce on. The nickel will self-plate. Similarly for other metals.

3. Determine Etching Pre-treatment if Applicable

Before we begin plating, the parts to be plated must be prepared for the treatment. The first step is to determine whether the surface of these parts should be chemically etched. Etching increases surface roughness and improves adhesion of the nickel layers during plating. Transene® recommends a surface roughness of about 20 microinches before plating. Consult the following table for recommended etching pre-treatments based on material.

Warning: Some etching components use dangerous caustics, such as hydrofluoric acid (HF). Consult the dedicated SOPs for those specific steps. This is only a general guide, with emphasis on the plating process itself.

SUBSTRATES	PRETREATMENT	ETCH	SENSITIZER & CONDITIONER	FIXING	NICKEL DEPOSITION OPER.TEMP.
Alumina	Polimet or Equivalent	Solution A	Solution B	Solution C	Solution D
Beryllia	“	HF (1 1/2 min)	“	“	65 °C
Magnesium Oxide	“	Chromic Acid (2 min.)	“	“	50 °C
Steatite	“	“	“	“	50 °C
Barium Titanate	“	Solution A (2 min.)	“	“	65 °C
Ferrites	“	HCl 20% (1 min.)	“	“	65 °C
Epoxies	“	Chromic Acid (3 min.)	“	“	45 °C
Mylar	“	Chromic Acid (3min.) followed with 25% KOH dip and rinse	“	“	45 °C
Polyethylene	“	“	“	“	45 °C
Polystyrene	“	“	“	“	45 °C
Lucite (Acrylic)	“		“	“	45 °C
Cellulose Acetate	“	Chromic Acid (2 min.)	“	“	45 °C
Lead Zirconate-Lead Titanate	“	Special*	“	“	90 °C

Applied to Lead Zirconate-Lead Titanate Pre-treatment*

1. Enough surface is required to obtain surface finish of 25-30 microinches average.
2. Soak in chromic-sulfuric HF solution 1 or 2 minutes, then rinse.
3. Soak in Solution A for 1 minute, then rinse.
4. Soak in potassium hydroxide 20% for l~ minutes. Rinse.
5. Soak in stannous chloride solution for 4 minutes. Follow by 1 mm. rinse in distilled or deionized water.
6. Continue RTM stops with steps III, IV, and V, using the solution B, C and D as indicated. Plate for 15 minutes.

COMPOSITION OF ETCH SOLUTIONS

Buffered Etch (Solution A) – a buffered solution **hydrofluoric acid**.

Chromic Acid

Chromium Trioxide	7.5 grams
Sulfuric Acid	25 mL
Water to	1 liter

Metal Etch

Nitric Acid	50% by volume
Glacial Acetic Acid	40% by volume
Water	10% by volume

Chromic-Sulfuric-HF- Solution

Chromium Trioxide	5% by volume
Sulfuric Acid	10% by volume
Hydrofluoric Acid (4896)	19% by volume

Stannous Chloride Solution

Stannous chloride 2% with hydrochloric acid added to obtain a clear solution.

4. List of Equipment and Resources Required

- 3 beakers or vats large enough to fully submerge parts to be plated
- 1 additional beaker or vat **compatible with the etching solution used**
- Hot plate and magnetic stirrer
- Plastic or glass stirring rod
- Non-metallic tweezers or pliers
- Thermometer to monitor bath solution temperature

5. Procedure

Detailed instructions are provided with each of the solutions used in the RTM process for alumina ceramics. For many other applicable materials, suitable etchants or process modifications are recommended. Steps 1 and 2 may be omitted as needed.

Etching, Sensitizing, and Fixing solutions should fully submerge the parts in question. Nickel plating solution (Solution D) used should form a bath whose volume is comparatively much larger than the part.

PROCEDURE FOR RTM PROCESS

1. **STEP I.** Preheat Electroless Nickel Deposition (Solution D) to optimum operating temperature of 90°C ready for use during the RTM Process (65°C with additive).

2. **STEP II.** Surface Pretreatment (Polimet)
The ceramic surface should exhibit surface roughness of at least 20 microinches, average, as shown by measurement with Brush Surf- indicator, Model MS-lOOO. If necessary, lap surface with Polimet to obtain adequate surface roughness of 25 microinches. Other means to obtain adequate surface roughness are applicable, E.G., sand- blasting. Polimet is used by converting it to a thin paste with water on a glass plate. The specimen is worked on the glass plate until the entire ceramic surface appears uniformly lapped. Rinse well in water to remove lapping compound.

3. **STEP III.** Etch (Solution A) Place the lapped ceramics in the appropriate buffered etch solution from the chart above (**CAUTION! Solution A contains HF**. If using it, Solution A **must** be contained in a polyethylene beaker), for the time listed in the same chart. Finally remove ceramic pieces from the etch solution and rinse thoroughly in water.

4. **STEP IV.** Surface Sensitizing (Solution B) Expose etched ceramics to Solution B for 4 minutes. Do not stir or agitate the solution. Avoid contamination from metal objects. Use glassware and plastic-coated tweezers or transfer racks for handling. Remove surface-sensitized ceramics, and without rinsing transfer the ceramics to the next solution. Solution contains palladium chloride, activating the nickel plating process.

5. **STEP V.** Fixing (Solution C) The ceramics should be allowed to stand in Solution C for 4 minutes. Again, do not agitate or stir. Finally remove ceramics and carefully rinse in distilled or deionized water by limiting the water rinse to a maximum of 30 seconds. Transfer to next step immediately.

6. **STEP VI.** Electroless Nickel Deposition (Solution D) must be held at optimum operating temperature of 90°C ready for use during the RTM Process. Addition of 12 gm/gal dimethylamine borane (Solution D Additive) prior to using Solution D is an effective means of lowering the plating temperature to 65°C. The ceramic pieces are placed in this special electroless nickel solution without agitation for first few minutes.
Approx. Plating Rate: 7.62 microns (0.3 mils) per hour immersion time of optimally run plating

CARE OF RTM SOLUTION

Special Electroless Nickel (Solution D) has a pH of 8.0. This pH should not be allowed to drop below 7.0; although the solution is well buffered, the addition of ammonium hydroxide from time to time may be required. The use of pH test paper is satisfactory for control.

The electroless nickel solution should be filtered to extend the usefulness of the solution whenever particles occur. The glass vessel used for RTM may sometimes develop a deposit of nickel, due to overheating. The glass vessel should then be cleaned as well.

Sufficient quantities of RTM solutions should be used in the RTM Process. These used solutions, however, should not be returned to the original container to guard against contamination.

6. **STEP VII.** Once desired thickness is achieved, finally remove ceramics and rinse in water.

6. Training Plan and Measures of Competency

- If using any etching solution that contains any amount of hydrofluoric acid (HF), then the user is required to take MIT's specific HF course.

7. Work Health and Safety Considerations

- Any use of etching solutions, including Transene Solution A, poses a safety hazard. Consult with the individual SOPs for those procedures.

8. References, Related Resources, and Acknowledgments

1. Transene Company, Inc.

9. Attachments or Related Documents

1. https://transene.com/rtm-process/