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ELECTROLESS NICKEL – DATA SHEET

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The following is a brief guide on the preferred operation of electroless nickel plating systems. For additional information, please consult the Parameters Page for the specific system in use and your Surface Technology, Inc. Technical Service Representative.

EQUIPMENT

- The plating tank should be constructed of polypropylene, stainless steel (Type 316) or mild steel with a suitable tank liner depending on bath in use and other considerations. Stainless steel tanks may be anodically protected if desired.
- Filtration through a 10-micron or finer rated polypropylene filter bag system is suggested. Polypropylene wound cartridge filters are also permissible, but are not as easy to use as filter bags. The filtering pump system should turn the bath over at a rate of at least 10 times per hour. Filter bags should be well rinsed in hot water before use. For the specific filtration recommended for each bath, consult the appropriate Parameters Page.
- Agitation is useful in maintaining bath homogeneity and consistent finish. Air spargers with air from a high volume, low-pressure air blower is recommended. Compressed air is not recommended due to potential oil contamination. Other types of agitation, may also be used. For the specific agitation recommended for each bath, consult the appropriate Parameters Page.
- Heating of the bath may be accomplished by various methods including heat exchangers and immersion heaters. The bath temperature should be monitored and maintained closely.
- Cooling of the bath with an appropriate cooling apparatus should be done rapidly at the end of a shift or any time the bath will not be used for an extended period of time.
- Rack, barrel, and fixturing devices should only be constructed of compatible materials such as polypropylene, CPVC, stainless steel, PTFE, Viton, silicone rubber, and others that can withstand the chemicals and temperature of the plating bath and pretreatment process.
- Masking should only be accomplished with compatible materials such as certain vinyl tapes, stop-off paints, plugs and gaskets made of Viton, silicone rubber, and others that can withstand the chemicals and temperature of the plating bath and pretreatment process.

OPERATION

- Review the data sheet, MSDS, and product labels for all components for safety and handling information. Only operate according to this Data Sheet and the respective Parameters Page. Insure you are using the most current Data Sheet and Parameters Page available from STI.
- Consult your Surface Technology, Inc. Technical Support Representative for any assistance on the pretreatment of the base materials to be plated.
- The tank should be clean and passivated. The most common method is with a solution of 40-50% nitric acid for 2-3 hours at room temperature, followed by rigorous rinsing and neutralizing of the tank and verification that no nitrate contamination remains.

- Maintain the bath between 80% and 100% concentration of nickel, hypophosphite, stabilizers, or other chemicals. Tighter control will further help performance.
- Titrate the bath before and after every batch of parts that is plated. Replenish during plating cycles if the workload will lower the nickel concentration to 90% or less.
- Continual and accurate measurement of bath temperature, pH, and bath solution level is important. Evaporation will reduce bath volume and give false indication of actual concentration. Add DI water as needed during the plating cycle to keep solution at proper level.
- Only use reagent grade ammonium hydroxide (50% concentration or less) and reagent grade sulfuric acid (25% concentration or less) to adjust the pH of bath.
- Air quality in plating shop should be clean and ventilated. Do not draw dirty air over plating or other tanks.
- Deposition rate depends upon operating temperature, bath loading, pH, agitation, and age of the bath. The plating rate at the preferred conditions for a new bath is listed on the Parameters Page for each system.
- Plating thickness can be determined by various methods including witness panel measurements, electronic devices, and cross section analysis.

SOLUTION MAINTENANCE

- The plating bath is maintained by simple analytical procedures and subsequent replenishment.
- Nickel Metal Concentration
 - The following reagents are required: Concentrated ammonium hydroxide, Murexide indicator mix (2 grams murexide powder in 100 grams sodium chloride), and 0.0575M E.D.T.A. solution.
 - Analysis procedure:
 - Pipette 10 ml of plating solution into a 250 ml Erlenmeyer flask.
 - Add 100 ml of deionized water, 10 ml of concentrated ammonium hydroxide, and about 0.2 grams murexide indicator mixture and mix well.
 - Titrate immediately with standard 0.0575M E.D.T.A. solution to purple endpoint, and record the number of ml of the E.D.T.A. solution used.
 - The grams/liter of nickel metal in the bath = ml of 0.0575M EDTA used x 0.336.
- Replenishment
 - Replenish according to the Parameters Page for the system in use, based upon the ml of E.D.T.A. consumed in the titration procedure above.
 - If the nickel metal concentration in the bath falls below 80%, it is best to replenish in two steps to avoid over-stabilization.
 - Each replenishment component should be added separately to the bath.
 - All replenishments should be made slowly with good mixing, and not directly over the workload.
- Sodium Hypophosphite Concentration
 - With proper make-up and replenishment of the bath, the concentration of sodium hypophosphite should be self regulating. From time to time, however, it is recommended that this analysis for sodium hypophosphite be done independently to assure the bath is at its optimum concentration.
 - The following reagents are required: 6N HCl solution, 0.1N iodine solution, 0.1N sodium thiosulfate solution, and standard starch indicator.
 - Analysis procedure:
 - Pipette 5 mL of the plating bath into a 250 mL Erlenmeyer or iodine flask.
 - Add 25 mL of the 6N HCl to the sample.
 - Pipette 50 mL of 0.1N iodine solution to the sample.
 - Stopper the flask and let it stand in the dark for 30 minutes.

- Titrate the sample with 0.1N sodium thiosulfate solution, adding starch indicator when a pale yellow color is reached.
- Continue to carefully titrate until the purple starch color disappears.
- Calculate the hypophosphite content of the plating solution from the volume of thiosulfate solution used: $5 - (0.1 \times mL \text{ thio used}) = meq \text{ hypophosphite in solution}$
- $(meq \text{ hypo}) \times (10.5) = g/L \text{ of hypophosphite}$

POST TREATMENT

- Consult your Surface Technology, Inc. Technical Support Representative for any assistance on the treatment of your parts following plating, such as rinsing, passivation, heat treatment, mechanical finishing, stripping, etc.

WASTE TREATMENT

- Consult your Surface Technology, Inc. Technical Support Representative for any assistance on the proper treatment/disposal methods of the used plating bath.
- All usage and waste treatment of the chemicals associated with this process must comply with all federal, state, and local regulations for health, environmental and safety requirements.

TROUBLE SHOOTING

The following are problems that may occur in plating, plus a listing of possible causes and suggested remedies. Consult your Surface Technology, Inc. Technical Support Representative for any additional trouble shooting and resolution assistance.

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| <ul style="list-style-type: none"> • Skip plating, pitting, edge pull-back, step plating, dark or laminar deposit <ul style="list-style-type: none"> ○ See: 1, 2, 3, 8, 11 • Roughness in deposit <ul style="list-style-type: none"> ○ See: 4, 5, 6, 7, 11 • Streaks in deposit <ul style="list-style-type: none"> ○ See: 1, 3, 8, 9, 11 • Dull or Matte Deposit <ul style="list-style-type: none"> ○ See: 1, 3, 10, 11, 14 • Poor adhesion <ul style="list-style-type: none"> ○ See: 1, 14, 15 | <ul style="list-style-type: none"> • Poor corrosion and/or chemical resistance <ul style="list-style-type: none"> ○ See: 1, 3, 4, 12, 13, 14 • Slow Plating Rate <ul style="list-style-type: none"> ○ See: 2, 3, 5, 8, 9, 10, 11, 14 • Fast Plating Rate <ul style="list-style-type: none"> ○ See: 4, 6, 14 • Short Bath Life <ul style="list-style-type: none"> ○ See: 2, 3, 10, 11, 14, 15 |
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Possible Causes/Remedies:

1. Improper Pretreatment - Temperature should be checked as well as purity and concentration of cleaner, activator, and other pretreatment solutions. Some metals and alloys, such as leaded steels, brasses, copper, aluminum and magnesium, require special preparation. Rinsing, temperature and rinsing time should be checked. Consider using an electro-cleaning, ultrasonic cleaning, and/or other methods of cleaning. Minimize transfer times between pretreatment steps. Consider a double zincate process for aluminum work pieces.
2. Over stabilization of bath - Bath should be dummied or discarded and replaced. Review replenishment history. Insure adequate work load in the bath, and add additional surface area if needed.
3. Chemical contamination - Bath should be dummied or discarded and replaced. Insure no sources of nitric acid, heavy metal or other contamination. Use only proper quality deionized water. Avoid drag-in.

4. Particulate contamination from solid particles; i.e. dust, loose nickel or metal chips - Avoid contamination and/or improve workload cleaning and rinsing, and bath filtration. Demagnetize ferromagnetic substrates if possible. Use only proper quality DI water.
5. Excessive solution replenishment while work is being plated – Replenishments should be added slowly and mixed thoroughly, as far away from work piece(s) as possible.
6. Low stabilizer content. – Increase the stabilizer content in the plating bath.
7. Only one side of work affected - Agitation around work pieces should be increased and/or work pieces should be rotated while plating.
8. Improper agitation - Agitation around work pieces should be improved and/or work pieces should be rotated while plating.
9. Low surface area - Surface area should be increased to recommended range.
10. Bath very old - Bath should be discarded, and new bath prepared.
11. Bath Imbalance - Nickel and reducer should be checked and bath adjusted.
12. Improper phosphorous content in the deposit – Adjust plating bath parameters or replace with alternative nickel-phosphorous alloy plating bath.
13. Pitting in base metal and/or deposit – Inspect base metal and remedy plating bath as needed.
14. Poor bath control - Uniform temperature should be maintained, pH and replenishment controlled.
15. Zincate build up in plating bath - Bath should be used for non-aluminum parts or discarded. Double zincate processing will reduce the rate of zinc contamination.

SURFACE TECHNOLOGY, INC.

Surface Technology, Inc. offers a full line of proprietary electroless nickel plating solutions and coating services including low, medium, high phosphorous, nickel-boron, and composites with diamond, PTFE, silicon carbide, boron nitride, identification/authentication, and other materials. All solutions are formulated to the strictest specifications for quality and consistency and are available in **NiPLATE™** and **ADDPLATE™** packaging options.

To compliment its line of plating solutions, Surface Technology, Inc. offers a wide array of pretreatment and waste-treatment chemicals; systems for plating on plastics; nickel sulfate and sodium hypophosphite; nickel strippers; consulting services and contract research and development projects.

Surface Technology, Inc. holds more than ninety U.S. and foreign patents, which have helped establish STI's global reputation as an innovative leader in the metal finishing industry. This strength has resulted in over forty license agreements with companies around the world including: Allied-Kelite, Atotech, Cookson Electronics-Enthone, Coventya, Electrochemicals, ESK, Okuno, OMG-Fidelity, Hitachi, IBM, Lea Ronal, MacDermid, Olin Hunt, Q&M, Rohm and Haas, Schering, Shipley, SurTec, Schlotter, Uyemura, and many others.

WARRANTY

We warrant our goods to conform to our standard specifications. This Warranty is in lieu of any and all other warranties or guarantees and our obligation hereunder for breach of such warranty is limited to either refund of the purchase price or replacement of said goods, as we may elect. We make no other warranties, express or implied, including those of merchantability and fitness for a particular purpose. We believe that the statements, technical information and recommendations contained herein are reliable, but they are given without warranty or guarantee of any kind except as specified above. Beyond that specified above we assume no responsibility for any loss, damage or expense, direct or consequential, arising out of the use of, or inability to use, our goods. Their quality and suitability for any particular purpose or use should be confirmed by the user's own tests.

Use of this product may be covered by one or more of the following US Patents: 5,863,616; 5,834,065; 6,306,466; 7,744,685; 8,147,601 and/or others pending. NiPLATE™ and ADDPLATE™ are Trademarks of Surface Technology, Inc... Composite Diamond Coating® and TraceCoat® are Registered Trademarks of Surface Technology, Inc.

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The 100 system is an economical fully bright electroless nickel process designed to deposit a uniform, medium phosphorous-nickel alloy. The NiPLATE™ 100 system is supplied in three liquid concentrates: NiPLATE™ 100 A for bath make-up and replenishment, NiPLATE™ 100 B for bath make-up, and NiPLATE™ 100 C for replenishment.

Bath Make-Up

Volume =	100	Gallons
NiPlate 100 A		5 Gallons
NiPlate 100 B		15 Gallons
DI Water		Balance

Parameters Page Instructions

- 1) Insert desired volume units in the yellow cell
- 2) Insert the bath volume in the green cell
- 3) Chemical usage values will be automatically calculated

Instructions:

- Fill a cleaned tank with DI water about half way.
- Add the NiPlate 100 B with good mixing
- Add the NiPlate 100 A with good mixing
- Add DI water to final bath level
- Adjust pH to the desired level
- Heat and use according to the specified conditions

IMPERIAL

METRIC

<u>Operating Conditions</u>	<u>Range</u>	<u>Optimum</u>	<u>Units</u>	<u>Range</u>	<u>Optimum</u>	<u>Units</u>
Temperature	185 - 198	190	°F	85 - 92	88	°C
pH	4.6 - 5.2	4.8	pH	4.6 - 5.2	4.8	
Bath Loading	0.3 - 1.0	0.6	ft ² /gal	0.75 - 2.5	1.5	dm ² /l
Nickel Metal Concentration	0.65 - 0.85	0.8	oz/gal	4.9 - 6.4	6	g/l
Hypophosphite Concentration	3.5 - 4.5	4	oz/gal	26.3 - 33.8	30	g/l
Plating Rate	0.8 - 0.9		mils/hr	20 - 22		microns/hr
Agitation	Mechanical or Filtered Air			Mechanical or Filtered Air		
Filtration	10 micron or finer			10 micron or finer		

Replenishment

ml 0.0575M EDTA consumed	17.8	16.9	16	15.1	14.2	13.4	12.5
g/l nickel metal present	6	5.7	5.4	5.1	4.8	4.5	4.2
% nickel metal present	100	95	90	85	80	75	70
NiPlate 100 A to add	Gallons	0	0.25	0.5	0.75	1	1.25
NiPlate 100 C to add	Gallons	0	0.5	1	1.5	2	2.5

TYPICAL DEPOSIT PROPERTIES

Phosphorus Content	6-9% by wt.
Hardness:	
As plated	550 VHN ₁₀₀
Heat treated 1 hr. @ 400C	900 VHN ₁₀₀
Salt Spray (ASTM-B-117)	90-100 hrs.
Stress	slightly tensile
Magnetic Properties	slightly magnetic
Deposition rate	0.8 – 0.9 mil / hour

SYSTEM FEATURES

Fully bright deposit
Consistent deposit properties
Excellent economy of use
Corrosion resistance
Outstanding stability
Ease of use
Long bath life
Meets MIL-C-26074D and AMS 2404C