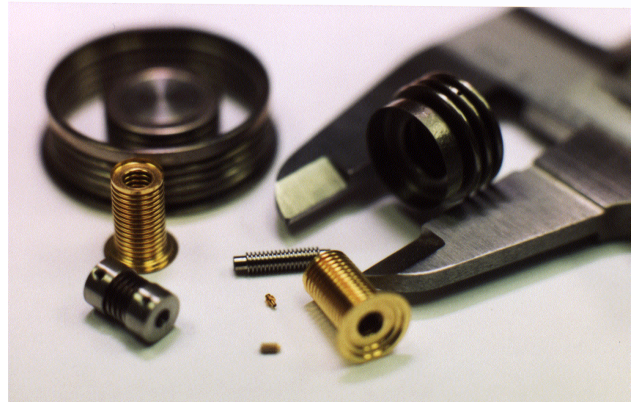


AUTONIC HX, 850

Production High Phosphorous ASTM B733 Type V

PROCESS:

AUTONIC HX is a, high-phosphorus electroless nickel process designed for processing in high reliability applications like space born metallic optics, high reliability electrical connectors, electroless forming and other applications. The applications require minimal textual defects and generally require a high level of corrosion protection.



FEATURES:

AUTONIC HX is a high-phosphorus electroless nickel process using a high purity formulation and highly developed formula producing exceptional results.

AUTONIC HX Deposit provides wear and corrosion resistance to metal parts in a wide range of environments. Typical hardness values are 420-470 Knoop at 100 g load (KHN100) in the as plated condition and 885-980 (KHN100) in the heat treated condition of ASTM B733 or Mil-C-26074E, Class 2.

AUTONIC HX process is designed for high production operations with simplicity and ease of operations. Replenishment of the process involves the use of two chemicals, AUTONIC HXPC and AUTONIC LNS, ultra pure carbon treated Liquid Nickel Sulfate. The products are liquid and can be added to the process while plating. These additions will maintain all the constituents and pH of the process providing for 5 plus metal turnovers of processing.

AUTONIC HX process provides excellent corrosion protection due to the purity of alloy, low porosity, and high phosphorus saturated nickel phosphide composition.

AUTONIC HX process provides a non-magnetic deposit for mechanical and electronic applications. Typical applications include computer memory discs, relay pull plates, magnetic switch components replacement for hard chrome, optical applications, and internal components to disc drives.

Feature Summary:

STAPLETON TECHNOLOGIES

Providing Electroless Technologies Since 1960

HOT LINE (800) 266-0541

<http://www.stapletontech.com>

DEPOSIT PROPERTIES AUTONIC HX DEPOSIT

Electroless Nickel Deposits can be classified into four types. These are based on the percent of phosphorus in the coating which can represent significant differences in performance of the alloys. The primary differences are in structure where ultra high phosphorus deposits of the AUTONIC HX, 11.5% to 13%, are free of Ni(111) both before and after heat treatment. This produces a deposit which is more corrosion resistant to most environments and is non magnetic.

PHYSICAL

Bond Strength to Steel	90,000 PSI - 620 MPa
Bond Strength to Aluminum	45,000 PSI - 310 MPa
Elongation	0.7 to 1.2 %
Tensile Strength	80 - 90 KSI
Internal Stress	2.2 KSI Tensile as plated
Thermal Coefficient of Expansion	$1.2 \text{ to } 1.4 \times 10^{-5}$
Melting Point	881° (C)
Electrical Resistivity	$80 - 110 \times 10^{-6}$ ohm-cm

COMPOSITION

Nickel	Balance
Phosphorus	10.2% to 11.5%
Trace	0.15 or less

HARDNESS

440 - 480 KHN _{100g}	As Plated
885 - 980 KHN _{100g}	Heat treated 700° (F) for 1 hour

STRUCTURE

In the as plated condition the deposit is amorphous. Upon heating to 220° (C) the deposit will start to crystallize and NiP₃ will form. The amount and orientation of the nickel will control the physical properties of the deposit.

CORROSION RESISTANCE

The AUTONIC HX deposit has excellent corrosion resistance in many Industrial environments.

Environments	Electrochemical	Immersion
Water, Brine 3.5% NaCl & Sat. CO ₂	9.8 mpy	5 mpy
Water, Brine 3.5% NaCl & Sat H ₂ S	0.02 mpy	nil
Water, Sea 3.5%	0.08 mpy	nil

(for a complete list of corrosion rates see "State of the Science")

Porosity

Ferroxyl on steel panels with 1 mil Passes with no blue spots
ASTM B117 Neutral Salt on steel panels with 1 mil has Passed 20,000 hours.

WEAR RESISTANCE

AUTONIC HX deposits are hard and resistant to abrasion and adhesive wear applications. Typical wear rates for the coating are 12 to 16 mg/1000 cycles Taber Abrader for the as plated condition and 6 - 12 gm/1000 cycles for the precipitate hardened deposits

OPERATING THE AUTONIC HX PROCESS

The AUTONIC HX Process is designed for ease of operations and process control. To operate the process the plater must be able to check the nickel concentration and pH and make adjustments to the process as required.

The plating process is operated in a specially designed process tank. This tank heats the solution to 90° C and filters it through a 5 micron bag at 10 to 20 times the volume of the tank each hour.

To maintain an efficient operation, the solution should be heated with a low density heat source and parts should be loaded when the operating temperature is reached. When all the parts are processed for the day the heat should be reduced to below 50° C with the filter operating and air agitation off.

The AUTONIC Series processes use a high purity nickel sulfate provided by STAPLETON TECHNOLOGIES. The AUTONIC LNS should be used to insure only the superior solution performance.

While the process is operating, the parts will consume nickel ions and reducing agent, thereby requiring their replacement to maintain the plating reaction. The amount of nickel consumed is generally measured in metal turnovers where 1 cycle is equal to a 100% replacement of the nickel metal in the original solution. The amount of reducing agent used is dependent on the amount of nickel consumed and will be replaced when the addition of AUTONIC HXPC.

Table 1, AUTONIC HX MAKE-UP FORMULA

Volume Tank		100	150	200	250	300	500	750	
AUTONIC HX _{PA}	150 ml/l	15	22.5	30	37.5	45	75	112	GAL
AUTONIC LNS(5#/gal)	50 ml/l	5	7.5	10	12.5	15	25	37	GAL

PROCEDURES FOR MAKING UP A NEW AUTONIC HX SOLUTION

1. Fill approximately 2/3 of the final working volume of cold deionized water into a clean plating tank.
2. Add the AUTONIC HX_{PA} to the plating tank.
3. Fill the tank to 95% percent volume with Deionized water.
4. Add the AUTONIC LNS and mix.
5. Record the new solution in a process log.
6. Start using your New AUTONIC Solution

Table 2, SUMMARY of OPERATING CONDITIONS

	At Start	UCL	LCL	Control Point	Frequency of Analysis
Conditions					
Nickel Metal	6.6 g/l	6.6 g/l	3.5 g/l	5.8 g/l	.5 - 4 hr.
Hypophosphite	35 g/l	42 g/l	33 g/l	35.5 g/l	4 - 8 hr.
pH (Electrometric)	4.4	4.7	4.1	4.4	.5 - 2 hr.
Temperature (C)	91	95	89	91	.5 - 2 hr.

PROCESS CONDITIONS and OPERATIONS

The AUTONIC HX process is simple to operate. A list of operating conditions can be found in TABLE 2. The major control is provided by checking for nickel using an EDTA Titration and from the results computing an addition of AUTONIC maintenance concentrates. These additions will maintain the chemistry, adding all the necessary chemicals to sustain the reaction. This procedure is required every time the solution uses 10% to 20% of the available nickel metal.

The Temperature and pH are also important in sustaining the reaction and therefore should also be controlled. The temperature causes the reaction to proceed and ultimately controls the rate and thickness of the plating. While the pH effects the alloy and plating rate it will also effect the plating rate. Lower temperature and pH will lower the plating rate.

In TABLE 2 the Upper Control Limit (UCL), Lower Control Limit (LCL), Control Point and recommended frequency of analysis are given. By adhering to these control points you will be able to produce consistent quality and high performance at the lowest possible cost.

Table 3, ADDITION SCHEDULE for AUTONIC HX Process

Concentration	Nickel concentration								
	6.6	6.2	5.8	5.4	5.0	4.6	4.2	3.8	g/l
Activity	100	93	88	81	75	69	63	57	%
EDTA Na ₂ mls	10	9.3	8.8	8.1	7.5	6.9	6.3	5.7	
100 GALLONS									
AUTONIC HX_{PC}	0	0.70	1.2	1.90	2.50	3.10	3.10	3.10	
AUTONIC LNS (5#/gal)	0	0.35	0.6	0.85	1.25	1.55	1.55	1.55	
250 GALLONS									
AUTONIC HX_{PC}	0	1.75	3.00	4.75	6.25	7.75	7.75	7.75	
AUTONIC LNS	0	0.87	1.50	2.38	3.12	3.87	3.87	3.87	

GETTING THE MOST OUT OF YOUR AUTONIC HX PROCESS

1. The solution operates most efficiently when it is plating with a load of between 30 to 90 square inches per gallon with mechanical agitation.
2. Possible contaminants from the shop or parts should be controlled as much as possible. These contaminants can be in the form of organics, metals, or solid materials. They can be airborne, on the part, or waterborne. A clean shop with processes that are maintained correctly will help insure excellent operations and maximum performance of the deposit with minimum costs of operations.
3. The AUTONIC Process will produce the most consistent deposits when it is operated in the 80% to 90% activity range. To operate in this range it is advisable to have the person who is plating, titrate and make the additions.
4. The AUTONIC Process is designed to be operated with continuous filtration through a 5 micron bag. It is advisable for the pump filter system be sized to move 20 plus times the volume of the solution in one hour. This high filtration will remove any unwanted particles which may have be in the process and insure that as the solution ages and at boiling the pump will still be providing in excess of 20 turnovers per hour.
5. When the process reaches about 350% or 3.5 times the original nickel metal it is time to think about plating the solution down to 50%, waste treating the solution and making up a new solution. With this type of approach you will be able to operate at the most economical point, using nearly 90% of all the nickel metal.
6. The AUTONIC HX Process can be loaded with parts up to 140 square inches per gallon. Working the solutions hard (60 to 90 in²/gal) after the first day of operation will provide the most efficient production for both chemicals and facility. Low loading (5 to 20 in²/gal) will have the effect of reducing the efficiency of the process and require additional replenishment chemicals to sustain the plating rate.

DAILY CARE

1. Cover the tank when you are not loading or unloading the tank.
2. Strip the tank with 20% to 30% nitric acid at the end of the production run if any significant nickel has plated onto the sides or bottom. This will help eliminate roughness and the significant loss of chemicals. After stripping the tank and piping you should always rinse and neutralize the system with a dilute solution of ammonium hydroxide. Small concentrations of nitric acid in the solution may cause a dull or streaked deposit. The addition of 5 g/l of sulfamic acid along with heating to plating temperature to 1 hour should correct the problem.
3. Turn off the heat when the solution is not going to be used for several hours. Prolonged heating with air agitation and no work in the tank will cause a slight oxidation of the reducing agents and subsequently reduce the efficiency of the process.
4. Always check the pH when you test for nickel. The results should be noted in a log and any correction required should be made using dilute sulfuric acid or ammonium hydroxide.

GENERAL EQUIPMENT INFORMATION

Filtration - Continuous filtration through 1 to 5 micron polypropylene filter bag that is elevated above the solution is recommended. This produces a pressure differential to cause filtration and removes the high concentration of heat from the filter area.

With the bag elevated, any plateout or activity in the solution can be detected and corrective action taken before a problem is evident.

Tank and Materials of Construction - All tanks, piping, racks and carriers which come in contact with the solution should be made of CPVC, Poly-propylene, 316LC Stainless Steel, or PVDF. The electroless nickel solution and stripping solutions are corrosive and care should be taken to prevent contact with electrical and mechanical systems.

Agitation - Mechanical agitation is recommended when the process is operating. A minimal loss of about 5% per day of reducing agent will occur when using air agitation. This loss can be minimized by not operating the air agitation and heaters when the tank is free of work. Prolonged heating and air will accelerate the oxidation of sodium hypophosphite.

Ventilation - The vapor from AUTONIC HX plating process is inherently corrosive due to the high concentration of salts in the material. All ventilation equipment should be constructed with poly-propylene, PVC or fiberglass to insure good equipment life and a healthy area for the platers.

TITLE: Analysis for Nickel Metal

SCOPE: This method is a direct titration for nickel metal using EDTA. The procedure is quick and will work with automatic titrators using ampermetric or spectrometric probes.

APPARATUS:

1. Erlenmeyer flask 250 ml
2. Automatic burette
3. Chemicals
 - a. Distilled water
 - b. Ammonium hydroxide
 - c. 35% Triethanolamine
 - d. Murexide Indicator in sodium chloride
 - e. 0.0575 M EDTA Na₂ Solution

PROCEDURE:

1. Add exactly 5.0 ml of plating bath into the Erlenmeyer flask.
2. Add about 100 mls of distilled water to the flask.
3. Add 20 mls of Ammonium hydroxide to the flask.
4. Add 10 mls of 35% Triethanolamine
5. Add about 0.2 g of Murexide Indicator Mix to the flask. The solution should be light straw color.
6. Titrate immediately with standard 0.0575 M EDTA Na₂ Solution to purple endpoint.

ANALYSIS:

$$\text{Nickel oz/gal} = (\text{MLS } 0.0575\text{M EDTA Na}_2) \times .0884$$

$$\text{Nickel g/l} = (\text{MLS } 0.0575\text{M EDTA Na}_2) \times 0.6624$$

TITLE: Analysis for Sodium Hypophosphite

SCOPE: This is a direct titration for hypophosphite using indirect titration for iodate using thiosulfate. The method takes 30 minutes and can be automated using automatic titrators.

APPARATUS:

1. Erlenmeyer flask 250 ml with stopper
2. 2 Automatic burettes, 25ml
3. 1x10 ml pipette
4. Chemicals
 - a. Distilled water
 - b. 0.1N Iodine
 - c. 0.1N Thiosulfate
 - d. 6N H₂SO₄ Analytical Grade
 - e. Starch Indicator

PROCEDURE:

1. Add exactly 2.0 mls of plating bath into the Erlenmeyer flask.
2. Add approximately 20 ml of 6N H₂SO₄ to the flask.
3. Add exactly 25 ml 0.1N Iodine to the flask.
4. Stopper flask and place in a dark place for exactly 30 minutes.
5. Remove from dark area and wash stopper and neck of flask into the sample contents with deionized water.
6. Titrate immediately with standard 0.1N Thiosulfate Solution to light yellow point. Add a small amount of starch indicator. Solution will go black. Continue titration until solution goes clear.

ANALYSIS:

Hypophosphite oz/gal = (MLS 0.1N Iodine (25) - MLS 0.1N Thiosulfate) x 0.3535

Hypophosphite g/l = (MLS 0.1N Iodine (25) - MLS 0.1N Thiosulfate) x 2.65

CAPABILITY INDEXES & FAX Service

Producing Quality plating requires a team effort by many people. Having the products made to high standards means that every step of the way people attend detail. At Stapleton Technologies we use a three level lot control program and operate to a state of the art quality assurance program.

One means of establishing this attention to detail is the use of Capability Indexes. This technique establishes the amount of performance variation that is occurring in the manufacturing process. By tracking this variation and controlling it the process of manufacturing always yields quality products.

For users of AUTONIC that rely on statistical process control internally and for purchasers of the coating we will provide capability indexes on a monthly basis.

HANDLING

AUTONIC HXPA, is a neutral pH product and is quite safe under almost all conditions. The solutions should not be taken internally and if contact with skin has occurred it should be washed with copious amounts of water. AUTONIC HXPC is a high pH product and will cause chemical burns if contact occurs. Wash with copious amounts of water while scrubbing with soap and warm water. (see MSDS)

STORAGE

AUTONIC HX products are concentrated and will freeze. The temperature should remain above 38°(F). Products should be shipped in heated trailers during the winter months.

MSDS, Material Safety Data Sheets

MSDS Sheets are available upon request from our facilities in Long Beach, California. Call or write (562) 437-0541 and specify mail or FAX for return.

ORDERS

Orders are accepted by phone (800) 266-0541, by mail, e-mail and by FAX (562) 437-8632. Specify trucking company and delivery date required.

TERMS

AUTONIC Products are sold FOB, Long Beach at NET 30 days unless otherwise specified at the time of shipment. Annual contracts and purchase agreements are available to provide secure prices and delivery of materials. Consult Stapleton Technologies for further details.

NON-WARRANTY

The information contained in this Data Sheet is believed by Stapleton Technologies to be accurate, true and complete. Since, however final methods for use of this product are in the hands of the customer and beyond our control, we cannot guarantee that the customer will obtain the results described in this Data Sheet, nor can we assume any responsibility for the use of this product by the customer in any process which may infringe the patents of third parties.