AUTONIC MX, 440

MID PHOSPHORUS, FUNCTIONAL MATRIX ELECTROLESS NICKEL

PROCESS:

AUTONIC MX is a functional, mid-phosphorus (7-9.5 %P) electroless nickel process designed for high production processing in engineering applications. The AUTONIC MX Matrix process represents extensive research and development in electroless nickel technology, combining industry requirements and unique advanced chemistry into an economical and efficient plating process.

Photo: 1 MX is used in petro chemical applications where salvage and corrosion protection combine to require thick and low stress deposits on steels

FEATURES:

AUTONIC MX is a mid-phosphorus functional electroless nickel process using advance organo-metalic stablilzers to produce an exceptionally efficient and uniform thick deposit. The chemistry supports producing thick deposit in excess of 100 mils without reactivation.

AUTONIC MX 1 Mil Deposits have been tested in Neutral Salt Spray in accordance to ASTM B117 for over 1000 hours with no red rust or substrate corrosion.

AUTONIC MX Deposit provides wear and corrosion resistance to metal parts in a wide range of environments. Typical hardness values are 470-520 Knoop at 100 g load (KHN100) in the as plated condition and 885-980 (KHN100) in the heat treated condition of Mil-C-26074, Class 2.

AUTONIC MX process is designed for high production operations with simplicity and ease of operations. Replenishment of the process involves pre-mixing MXMC with Sodium hypophosphite. This replenisher along with the AUTONIC LNS, Liquid Nickel Sulfate is then added to the working process. These additions will maintain all the constituents and pH of the process providing for 8-10 plus metal turnovers of processing.

AUTONIC MX process is designed for simple operation. The operator titrates for nickel and makes two liquid additions to the operating process to return the process to the working concentration for all the ingredients. These adds maintain the pH, nickel, hypo and additives.

AUTONIC MX process is stable and is capable of passing a 120 second palladium stability test. Typical electroless nickel solutions will only pass 5 to 10 seconds.

AUTONIC MX processes have been formulated for ease in waste treatment providing a process that can be plated down to less than 5 ppm in three hours.

DEPOSIT PROPERTIES AUTONIC DEPOSIT

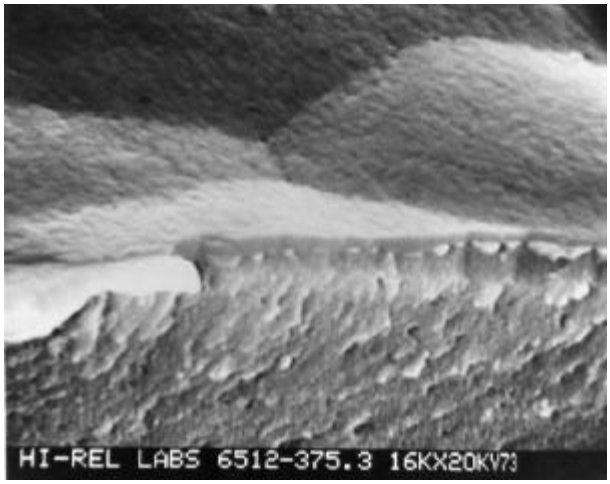


Photo: 2 Surface and cross section of MB Deposit. Oxide layer on surface is approximately 4 u" thick. 16KX

Electroless Nickel Deposits can be classified into four types. These are based on the structure and percent of phosphorus in the coating that in turn controls the deposit performance. These deposits are amorphous as plated and after heat treatment exhibit a mixture of ß and ? phases. Deposits produced by the MX process can be classified as ASTM B733 Type IV and V.

Mid phosphorus deposits are the most common. They are provided by AUTONIC MX, MB and MZ processes and meet all the requirements of MIL-C-26074.

PHYSICAL

Bond Strength to Steel	90,000 PSI	(620 MPa)
Bond Strength to Aluminum	45,000 PSI	(310 MPa)
Elongation	0.2 to 0.7 %	
Tensile Strength	40 - 60 KSI	
Internal Stress	1.2 KSI Tensile as plated	
Coefficient of Expansion	1.2 to 1.4 X 10^{-5}	
Melting Point	881^{o} (C)	
Electrical Resistivity	55 - 90 X 10^{-6} ohm-cm	

COMPOSITION

Nickel	Balance
Phosphorus	7.0% to 9.5%
Trace	0.15 or less

HARDNESS

470 - 520 KHN_{100g}	As Plated
885 - 980 KHN_{100g}	Heat treated 700^{o} (F) for 1 hr

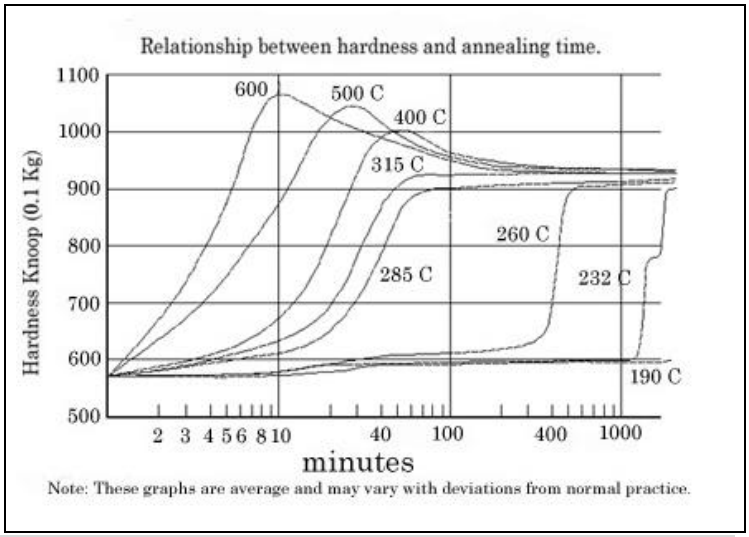
STRUCTURE

In the as plated condition the deposit is amorphous. Upon heating to 220^{o} (C) the deposit will start to crystallize into a beta phase and NiP_3 will form. The amount of phosphorus and orientation of the nickel phosphide controls the physical properties of the deposit. An alpha phase exists between 154 (C) and 220 (C) and will remain unstable and loading forces and temperature will control orientation. In delicate mechanical sensing applications the AUTONIC HX deposit should be used to minimize variations in the modulus due to this mechanical instability.

HEAT TREATMENT TO INCREASE HARDNESS

The MX deposit can be hardened to near the hardness of hard chromium. This is caused by the precipitation of phosphorus and nickel forming nickel phosphide. The graphs on the right show differing treatments and the resultant hardness over time. These curves are approximations and should only be used as a guide to achieve specific hardnesses.

Another factor in controlling the hardness is the thermal inertia of the part while heating and cooling which will effect the time and temperature to achieve maximum hardness.



CORROSION RESISTANCE

The AUTONIC MX deposit has excellent corrosion resistant in many industrial environments. The deposits are extremely well suited for brine and alkali salts. In most of these applications the corrosion rate is less than 0.1 mils/year. Mineral acids and strong oxidizers will provide increased corrosion rates on parts and diminished service.
The corrosion rate determination by calculating weight loss from exposure and conducting hundreds of immersion and electrochemical corrosion tests reveals the potential for protection of base metals over a wide range of environments. A full list of environments is available in the State of the Science document that is included in this electronic document.

Environments	Electrochemical	Immersion
Water, Brine 3.5% NaCl & Sat. CO_2	9.8 mpy	5 mpy
Water, Brine 3.5% NaCl & Sat H_2S	0.02 mpy	nil
Water, Sea 3.5%	0.08 mpy	nil

Porosity

- Ferroxyl on steel panels with 1 mil Passes with no blue spots
- ASTM B117 Neutral Salt on steel panels with 1 mil Passes 1000 hours.

WEAR RESISTANCE

AUTONIC MB, MX and MZ deposits are hard and resistant to abrasion and adhesive wear applications. These AUTONIC electroless nickel deposits have similar wear properties to engineering chromium deposits. In many applications the increased corrosion protection over chromium with nearly equal hardness affords AUTONIC superior service to chromium.

For applications which can't be heat-treated for maximum hardness the AUTONIC LX process is recommended. This deposit is significantly harder in the as plated condition than mid phosphorus deposits and provides excellent wear resistance in many applications.

Typical wear rates for the AUTONIC 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 MX PROCESS

The AUTONIC MX 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^{o} 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^{o} C with the filter operating and air agitation off.

The AUTONIC Series processes use AUTONIC LNS, carbon treated and submicron filtered Liquid Nickel Sulfate to insure optimum process 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 MXPC or MXPD is made.

MAKEUP

TABLE 1, AUTONIC MX MATRIX MAKE-UP FORMULA

AUTONIC MXMA	80 ml/l	8	12	16	20	24	40	gal
Sodium hypophosphite	24 g/l	22	33	44	55	66	110	lbs
AUTONIC LNS (5#/gal)	50 ml/l	5	7.5	10	12.5	15	25	gal

PROCEDURES FOR MAKING UP A NEW AUTONIC MX SOLUTION

1. Fill approximately 2/3 of the final working volume of cold deionized water into a clean plating tank.

2 Add the AUTONIC MXMA to the plating tank.

3. Add the Sodium hypophosphite to the plating tank and mix.

4. Fill the tank to 95% percent volume with Deionized water.

5. Add the AUTONIC LNS and mix.

6. Record the new solution in a process log.

MIXING REPLENISHER

The AUTONIC MX Matrix Process requires Sodium hypophosphite to prepare the Replenisher Mix for use. This is an economical and convenient way of using electroless nickel chemistry.

The replenisher concentrate is added to a clean mixing container and the Sodium hypophosphite is added. The Sodium hypophosphite will dissolve into the liquid producing a ready to use Replenisher Mix. Minimal agitation is required to dissolve this material and it can be ready for use in 10 minutes.

Using Sodium hypophosphite to mix the AUTONIC Matrix replenisher provides a 20% to 40% savings over standard production electroless nickel chemistry due to the increased cost of inventory and related storage.

The replenishment mix should be prepared before you start plating. The following table shows the formula and how to mix the material.

TABLE 2, REPLENISHER FORMULA for mixing MXPC

Products		Mix Tank Volumes		
MXMC or MXMD	800 ml/l	10 gal	56 gal	
+	+	+	+	
Sodium hypophosphite	330 g/l	35.0 lbs	196 lbs	
	=	=	=	
= MXPC	1000 ml	12.5 gal	70 gal	pH 5.3
or MXPD				PH 10.7

The AUTONIC MXPC Replenisher has a pH of 5.3 and is inherently safe to handle. Adding Sodium and Ammonium bicarbonate and raising the pH to 10.7 provides for self-adjusting pH. This product is available as MXMD. Filtration is not necessary if the plating process is being continuously filtered. The material can be stored for up to a year in this manner.

TABLE 3, SUMMARY of OPERATING CONDITIONS

	at make-up			Control Point	Frequency of Analysis
Nickel Metal	6.6 g/l	6.6 g/l	3.5 g/l	5.8 g/l	0.5 - 4 hr
Hypophosphite	24 g/l	35 g/l	15 g/l	28 g/l	4 - 8 hr.
pH(Electrometric	4.6	4.9	4.0	4.6	0.5 - 2 hr
Temperature (C)	91	95	89	91	0.5 - 2 hr.

PROCESS CONDITIONS and OPERATIONS

The AUTONIC MX Matrix process is simple to operate. A list of operating conditions can be found in TABLE 3. The major control is provided by checking for nickel using an EDTA Titration and, from the results, computing an addition of AUTONIC replenisher 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 is the major factor in controlling the deposition rate. It is important to maintain a consistent temperature to insure the rate can be predicted. The pH effects the alloy and plating rate and also needs to be controlled.

In TABLE 3 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.

AUTOMATIC REPLENISHER PUMPS

Automatic feeding systems are available from Stapleton Technologies to monitor the nickel and pH and automatically add the replenisher chemicals back to the plating process. For more information contact Stapleton Technologies.

TABLE 4, ADDITION SCHEDULE for AUTONIC MX Process

	Nickel concentration								
	6.6	6.2	5.8	5.4	5.0	4.6	4.2	3.8	g/l
	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	
Volume of Tank									
100 gal									
MXPC or MXPD	0	0.70	1.2	1.90	2.50	3.10	3.10	3.10	gal
LNS (5#/gal)	0	0.35	0.6	0.85	1.25	1.55	1.55	1.55	gal
250 gal									
MXPC or MXPD	0	1.75	3.00	4.75	6.25	7.75	7.75	7.75	
LNS (5#/gal)	0	0.875	1.50	2.385	3.125	3.875	3.875	3.875	

GETTING THE MOST OUT OF YOUR AUTONIC MX PROCESS

1. The solution operates most efficiently when it is plating with a load of between 10 to 40 square inches per gallon with mechanical agitation. The process can be loaded to in excess of 90 to as high as 140 in2/gal.
2. Possible contaminates from the shop or parts should be controlled as much as possible. These contaminates 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 times the volume of the solution in one hour. This high filtration will remove any unwanted particles that may be in the process and insure that as the solution ages and at boiling the pump will still be providing in excess of 10 turnovers per hour.
5. When the process reaches about 450% or 4.5 times the original nickel metal has been added it is time to think about plating the solution down to 50%, destructing 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 95% of all the nickel. The process can be operated until exhaustion that can range in life from 6 to 20 MTOs depending on the dragout.

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 of nitric with a dilute solution of ammonium hydroxide. If nitric acid is present in the solution a dull or streak deposit may develop.

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 5 to 15 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 that come in contact with the solution should be made of PTFE, Polypropylene, 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 MX plating process is inherently corrosive due to the high concentration of salts in the material. All ventilation equipment should be constructed with polypropylene, PVC or fiberglass to insure good equipment life and a healthy area for the platers.

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

STORAGE

AUTONIC MX products are concentrated and will freeze. The temperature should remain above 30^{o} (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 and by FAX (562) 437-8632 and e-mail at info@stapletontech.com. 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.

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