



quality metal finishing guide

Electroless Nickel Plating

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mfsa quality metal finishing guide

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ELECTROLESS NICKEL PLATING

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INTRODUCTION

The Metal Finishing Suppliers' Association, Inc. is a trade association of companies and individuals engaged in providing the metal finishing industry with equipment, products and supplies. A primary objective of the Association is to promote the interests and further the welfare of its members and their customers while safeguarding the interests of the ultimate consumer. The Association, along with other groups in the industry, has long realized that the public, the industrial buyer and the designer are not too well informed about metal products finishing nor how to specify high quality coatings on industrial and consumer products.

In 1960 MFSA initiated action to upgrade the durability of metal finishes, in a program known as the "Quality Metal Finishing Project" or QMF. This project has been so successful that it is being continued and enlarged. It consists of programs aimed at: (1) promoting technically sound specifications or standards in cooperation with interested trade and technical societies; (2) providing information to both producers and buyers of metal products finishing; and (3) providing printed guides containing information on established standards and specifications, readily accessible for day to day use by the design engineer, the purchasing agent and all those involved in the use of metal product finishes.

The first volume of the series of guides dealt with standards for quality copper/nickel/chromium finishes. Published in 1965, that volume (Vol. I) has had several printings. It was followed by a second publication, Quality Metal Finishing Guide—Zinc and Cadmium Coatings. Because of the demand for more printings, the MFSA reissued both guides in a series of revisions constantly up-dated. In the decade of the '70s, QMF Guides on Mass Finishing and Tin and Tin Alloy Coatings were written and printed to further demonstrate our devotion to finish and/or coating quality.

Quality

As an association promoting the interest and welfare of the producers and customers of the metal finishing industry, MFSA realizes that all suffer loss when products, inadequately finished for their intended life, are sold. The consumer may be persuaded to shift to a product with a nonmetallic finish, or a nonmetallic product. MFSA wishes to encourage management to build in the right amount and kind of high quality needed to guarantee the expected or promised service of the unit.

Management needs to know how a high quality metal finish can be achieved, how to select proper specifications for a given service and how to control the release of products to insure that they meet the standards. At the same time management must be confident that this know-how is based upon sound engineering principles and incorporates a reasonable factor of safety.

WHAT IS ELECTROLESS NICKEL?

This volume is concerned with autocatalytic nickel plating, commonly referred to as electroless nickel plating. In contrast with electroplating, electroless nickel (EN) plating does not require rectifiers, electrical current or anodes. Deposition occurs in an aqueous solution containing metal ions, a reducing agent, chelates, complexing agents and stabilizers. Chemical reactions on the surface of the part being plated cause deposition of a nickel alloy.

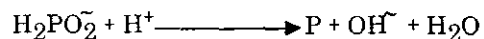
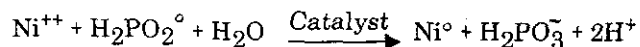
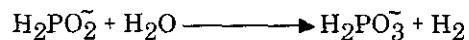
Since all surfaces wetted by the electroless nickel solution are plated, the deposit thickness is quite uniform. This unique property of EN makes it possible to coat internal surfaces of pipes, valves and other parts. Such uniformity of deposit thickness is difficult, if not impossible, to achieve by any other method.

The discovery of electroless plating is credited to Brenner & Riddell in the 1940's. Today EN has grown into a very substantial segment of the metal products finishing industry.

Compared with plating of other metals, electroless nickel (EN) plating is relatively young - being commercially available for less than 30 years; however, in the past decade the usage of the coating has grown to such proportions that electroless nickel plated parts are found underground, in outer space, and in a myriad of areas in between.

This guide seeks to provide the reader with more thorough understanding of the process. The volume includes descriptions of deposit properties, equipment required, process applicability and test procedures to the end that a high quality EN deposit can be achieved and maintained.

The chemical reactions that occur when using sodium hypophosphite as the reducing agent in electroless nickel plating are as follows:



An electroless nickel coating is a dense alloy of nickel and phosphorus. The amount of phosphorus generally varies from 3 to 12 pct, depending upon bath formulation, operating pH and bath age. The deposition process is autocatalytic; i.e., once a primary layer of nickel has formed on the substrate, that layer and each subsequent layer become the catalyst that causes the above reaction to continue. Thus, very thick coatings can be applied, provided that the ingredients in the plating bath are replenished in an orderly manner. In general commercial practice, thicknesses range from 0.1 mil to 5 mils.

Electroless nickel deposits are *functional* coatings and are rarely used for decorative purposes only. The primary criteria for using electroless nickel generally falls within the following categories:

- 1) Corrosion resistance.
- 2) Wear resistance.
- 3) Hardness.
- 4) Lubricity.
- 5) Solderability and bondability.
- 6) Uniformity of deposit regardless of geometries.
- 7) Nonmagnetic properties of high-phosphorus nickel alloy.

These items will be discussed at greater length in the Properties section of this book.

In the early years, platers encountered many problems with electroless nickel because of poor formulations, inferior equipment, misapplications and a general misunderstanding of the process and the deposit. In the first decade and a half of its existence, electroless nickel plating had an aura of "black magic" attached to it. Modern bath formulations, however, use only the purest grades of chemicals, delicately balanced and blended to give the processor plating baths with long life, exceptional stability, consistent plating rates, self-maintaining pH and most importantly, reproducible quality. In addition, advancements in tank design, filtration systems, heating and agitation have virtually eliminated the problems that plagued the user years ago.

Furthermore, in the past decade, advancements have been made in autocatalytic nickel plating solutions. Reducing agents other than sodium hypophosphite are used for special applications; composites of nickel with diamonds, silicon carbide and PTFE are available; and ternary alloys may be applied. Also, baths have been formulated to yield specific results; i.e., high corrosion resistance, brightness, high plating rate, improved ductility and low or high levels of magnetic response.

It has taken many years of hard work and cooperative effort by the suppliers and users to arrive at the present state of the art.

TYPES OF ELECTROLESS NICKEL

All electroless nickels are not the same. Different types have been developed to provide special properties, depending on the end-use requirement. Table I lists deposit characteristics and suggests suitable EN types or systems.

Nickel-phosphorus Baths

Acid nickel-phosphorus. Most commonly used for engineering applications, acid nickel-phosphorus baths typically deposit 88 to 93 pct nickel and 7 to 12

TABLE I

AUTOCATALYTIC NICKEL SYSTEMS MOST SUITABLE FOR OBTAINING SPECIFIC DEPOSIT CHARACTERISTICS*

Characteristics Desired	Autocatalytic System Most Suitable
Corrosion resistance	1. Nickel-phosphorus, acid solution. 2. Composite coatings.
Wear resistance	1. Nickel-phosphorus, acid solution formulated for maximum corrosion resistance on the basis metal selected.
Hardness	1. Nickel-phosphorus, acid solution - heat treated. 2. If heat treatment is not practical, use nickel-boron of 3 pct or more boron. 3. Composite coatings.
Adhesion	1. Nickel-phosphorus, acid solution - highest phosphorus content. 2. Teflon® composite or impregnation.
Electrical resistance	1. Nickel-phosphorus, acid solution.
Solderability	1. Nickel-boron - low boron content, less than 1 pct. 2. Polyalloys - exhibit excellent shelf-life solderability.
Welding	1. Polyalloys. 2. Nickel-boron, less than 1 pct boron.
Magnetic	1. Polyalloys.
Electric (for memory)	1. Nickel-cobalt-phosphorus. 2. Nickel-cobalt-boron. 3. Cobalt-phosphorus. 4. Nickel-cobalt-iron-phosphorus.
Electrical conductivity	1. Nickel-boron with boron less than 0.3 pct (resistivity is about 5.8-6.0 microohms/cm/cm ²).
Electrical resistance	1. Some polyalloys. 2. Nickel-phosphorus - high phosphorus content.
Galvanic replacement	1. Nickel-boron, 1-3 pct boron.
Electrolytic replacement	1. Nickel-boron - low boron for soldering, 0.1-0.3 pct B; higher boron for contacts, 0.5-1 pct B. 2. Polyalloys of phosphorus or boron if below 0.5 pct P or B.

*Action depends on severity of requirements and on economics. Boron-reduced systems are about five times more costly in terms of \$/mil/sq ft. However, thinner deposits may be used.

phosphorus. Deposits from these baths are quite hard, have natural lubricity and excellent wear and corrosion resistance. Some of the available baths are formulated with complexing agents and stabilizers which produce deposits highly resistant to chemical attack. These generally produce deposits containing a minimum of 9.5 pct phosphorus.

alkaline nickel-phosphorus. These baths plate at relatively low temperatures, making them suitable for plating on plastics. In addition, because of the low phosphorus content deposited (3 to 4 pct), they are used in many applications in the electronics industry, where enhanced solderability is often required.

Nickel-boron Baths

Nickel-boron baths are usually formulated using an amine borane as the chemical reducing agent. Alloy deposits can be plated from acid as well as alkaline baths and are harder, as plated, than nickel-phosphorus deposits. In addition, the melting point of nickel-boron alloys is higher.

Nickel-boron systems are most often used in electronic applications to provide specific deposit properties. They are sometimes used in industrial wear applications because of their high hardness levels. The chemical cost of these systems can range from five to 10 times that of nickel-phosphorus baths.

Low-boron-containing baths (less than one pct B) produce deposits having high electrical conductivity, good solderability and good ultrasonic bonding characteristics. Baths formulated to produce higher levels of boron (2 to 3 pct) in the deposit have very high hardness values as plated and tend to retard the formation of oxides on the surface of the deposit.

Sodium borohydride is sometimes used as the chemical reducing agent in nickel-boron systems. These baths codeposit higher levels of boron (5 to 6 pct), but are less stable than amine borane baths because of the high pH values required to prevent hydrolysis and solution decomposition.

Polyalloys

Several electroless nickel plating solutions produce deposits having three or four elements. These include nickel-cobalt-phosphorus; nickel-iron-phosphorus; nickel-tungsten-phosphorus; nickel-rhenium-phosphorus; nickel-cobalt-phosphorus; nickel-molybdenum-boron; nickel-tungsten-boron; and others.

Each of the above is designed to maximize qualities such as corrosion resistance, hardness, high-temperature resistance, electrical properties and magnetic or nonmagnetic characteristics.

Composite Coatings

The excellent wear resistance of electroless nickel can be further enhanced by codepositing hard particulate matter with the nickel-phosphorus alloy. Usually, particles of silicon carbide (4,500 VHN) or synthetic diamonds (10,000 VHN) are used in this process. A uniform dispersion of particles (20 to 30 pct by vol) is held in place in the deposit by the nickel-phosphorus matrix. These deposits are very brittle and require a sound substrate to prevent cracking in use. Composites containing silicon carbide are most often used in mold and die applications. Those containing diamonds have found use in textile applications.

Teflon® (PTFE) can also be codeposited with electroless nickel to provide even greater lubricity than that which naturally occurs in the nickel-phosphorus alloy deposit. Alternatively, Teflon® can be impregnated in the deposit as a post-plating operation. Both techniques produce an extremely slick surface which is useful in the packaging machinery industry, where minimum loads travel at maximum speeds.

conventional electroless nickel plating baths are not well suited to site plating, as the stabilizer is affected by the high concentration of sulfate matter.

APPLICATIONS OF ELECTROLESS NICKEL

Electroless nickel produces an alloy with truly unusual properties. These properties have made EN very useful in a broad range of functional applications. Most applications take advantage of the hardness, lubricity, corrosion resistance, electrical and magnetic properties of electroless nickel, as well as its ability to cover complex geometries and internal as well as external surfaces. Table II lists many of the common applications and features which properties of EN are of value in each of these applications.

PROPERTIES OF ELECTROLESS NICKEL

The properties of electroless nickel that are responsible for the rapid expansion of its use as a functional metallic alloy deposit in recent years. No other coating has the combination of properties offered by electroless nickel.

Corrosion Resistance

One of the most common reasons for selection of electroless nickel coatings in functional applications is its excellent corrosion resistance. In the very corrosive conditions encountered in drilling oil wells and pumping out the oil, for example, electroless nickel has shown its ability to withstand the combination of corrosive chemicals and abrasion.

Density

An electroless nickel deposit containing 3 pct phosphorus has a density of 7.92 g/cm³. An electroless nickel deposit with a 7.5 pct phosphorus content has a reported density of 7.92 g/cm³. These values are lower than those of pure metallurgical nickel (8.91 g/cm³).

The lower density of electroless nickel is caused by the presence of phosphorus as an alloying constituent. The most common range of phosphorus present in commercially applied deposits is generally 3 to 12 pct. Analysis has also shown minor levels of other elements present. These elements affect density and include hydrogen (0.0016%); nitrogen (0.0005%); oxygen (0.0023%); and carbon (0.04%).

Coefficient of Thermal Expansion

The coefficient of thermal expansion of a deposit containing 8 to 9 pct phosphorus is 13 to 14.5 x 10⁻⁶/°C. This compares to values for electroplated nickel of 14 to 17 x 10⁻⁶/°C.

Heat of Conductivity

The heat of conductivity for an electroless nickel deposit containing 8 to 9 pct phosphorus is 0.0105 to 0.0135 cal-cm/sec/°C. Pure metallurgical nickel has a value of 0.198 cal-cm/sec/°C.

Melting Temperature

The melting temperatures of electroless nickel deposits vary widely, depending upon the amount of phosphorus alloyed in the deposit. A generally accepted melting point is about 1616F (880C) for deposits from processes with approximately 7 to 9 pct phosphorus. This temperature corresponds to the melting point of nickel phosphide (NiP₃), which precipitates during heating of electroless nickel deposits.

Magnetism

Electroless nickel deposits containing greater than 8 pct phosphorus are considered to be essentially nonmagnetic as plated. The coercivity of 8.6 pct and 7.0 pct phosphorus content deposits has been reported at 1.4 oersteds and 2.0 oersteds respectively. A 3.5 pct phosphorus content deposit produces a magnetic coating of 30 oersteds. When the phosphorus content is increased to 10 pct, the deposit is nonmagnetic.

Coating thickness measurements with devices which rely on the nonmagnetic characteristic of the coating may become inaccurate and require special calibration if phosphorus content is below 8 pct.

Heat treatment of electroless nickel will increase the magnetism of the deposit. Most deposits which contain above 9 pct phosphorus will become slightly magnetic when heat treated above 518 to 536F (270 to 280C); however, some will show lower remnant magnetism. It is at this temperature that the solid solutions of phosphorus in nickel which occur in the as-plated deposit begin to form both nonmagnetic nickel phosphide (Ni₃P) and magnetic nickel.

Electrical Resistivity

The electrical resistivity of pure metallurgical nickel has a value of 6.05 microhm-cm. Electroless nickel deposits containing 6 to 7 pct phosphorus have values (as plated) which range from 52 to 68 microhm-cm. A deposit with 2.2 pct phosphorus has electrical resistivity of 30 microhm-cm, while a deposit with 13 pct phosphorus has a significantly higher resistivity—110 microhm-cm.

Heat treating electroless nickel reduces its electrical resistivity. Heat treatment up to 302F (150C) produces changes in the deposit primarily attributed to structural averaging of the phosphorus content and liberation of absorbed hydrogen. Beginning in the range of 500-536F (260-280C), heat treating produces a further decrease in electrical resistivity. This change is attributed to the precipitation of nickel phosphide (NiP₃) in the coating. An electroless nickel deposit with 7 pct phosphorus, heat treated to 140F (60C) was reportedly reduced from 72 to 20 microhm-cm.

APPLICATION	Base Metal	Deposit-Mils Approx.	Wear Resistance	Corrosion Resistance	Repair & Salvage	Fretting Resistance	Release Properties	Weldability	Solderability	Base for Electroplating	Infinite Throw	Thickness Control	Uniform Coating	Electroless Forming
AUTOMOTIVE														
Heat Sinks	Al	.4		X					X		X			
Pistons	Fe	.6	X	X										
Engine Bearings	Al		X	X										
Hose Couplings	Fe	.2	X											
Gears and Gear Assemblies	Fe	0.4-1.0										X	X	
AIRCRAFT														
Engine Overhaul	Fe	1.0+	X	X	X						X	X		
Struts	S/S	1.0-2.0			X							X		
Landing Gear	Fe	1.0+	X	X	X									
Hydraulics	Fe	1.0+												
Propellers	Fe	1.0+	X	X	X									
Hinges	Fe	.4	X						X					
Tape Guides	Al	.2	X											

Engine Mounts	4130 4140	1.0-2.0	X	X									X	
Turbine Parts	Fe	1.0-2.0											X	
Gyro Parts	Fe	0.5-1.0	X											
CHEMICAL EQUIPMENT														
Tanks	Steel and SS	2.0-5.0		X										
Pumps	Steel and SS	2.0-5.0	X	X										
Filters	Steel and SS	2.0-5.0	X	X										
Heat Exchangers	Steel and SS	2.0-5.0		X										
Spray Nozzles	Fe/Cu	0.3-1.0	X	X							X	X	X	
Tubing	Cu/Al Fe/SS	0.5-2.0	X	X							X	X	X	
RAILROAD														
Diesel Shafts	Fe	1.0+	X		X	X					X	X		
ELECTRICAL MOTORS														
Variable Speed Shafts	Fe	0.5+	X		X							X		
Rotor Blades, Starter Rings	Fe/Al	0.5-5.0	X	X										
PRINTING ROLLS	Fe/Cu	0.8-1.5	X	X									X	

Solderability/Weldability

Electroless nickel-phosphorus alloys are easily soldered with a highly active acid flux. Soldering without a flux or with mildly active fluxes can be more difficult if the parts are allowed to form oxides by extended exposure to the atmosphere. The heat processing of electroless nickel plated parts can make soldering very difficult unless a highly active acid flux is used.

Welding of electroless nickel deposits is not commonly done. There is a tendency of phosphorus to migrate to grain boundaries during cooling of the weld. This results in "hot cracks" or disintegration of the weld.

Adhesion

Excellent adhesion of electroless nickel deposits can be achieved on a wide range of substrates, including steel, aluminum, copper and copper alloys. Typical bond strengths reported for electroless nickel on iron and copper alloys range from 50 to 64 kpsi (345 to 441 M Pa). (kpsi = 1000 pounds per square inch, M Pa = Mega Pascal). The bond strength on light metals, such as aluminum, tends to be lower, in the range of 15 to 35 kpsi (103 to 241 M Pa) for most alloys.

Heat treatment is commonly employed to improve adhesion of EN on all metals, particularly on light metals such as aluminum or titanium. During this heat treatment diffusion occurs between the atoms of the coating and the substrate. Heat treatment is detailed later in this book in the "Post Treatments" section.

Thickness

Electroless nickel can be deposited to produce a wide range of coating thicknesses, with uniformity and minimum variation from point to point. This uniformity can be maintained in plating both large and small parts and in components which are fairly complex, with recessed areas. Electroplating of such parts, on the other hand, would produce thickness variation and possible voids in the plating when coating holes and inside diameters. The range of thicknesses for electroless nickel in commercial applications is 0.1 mil to 5 mils (2.54 to 127 μm), although deposits as thick as 40 mils have been reported. Normally thickness is built at the rate of 0.3 to 0.8 mil/hr (7.5 to 20 $\mu\text{m}/\text{hr}$).

The majority of commercial applications, except those involving corrosive service or heavy buildup of worn parts, utilize a thickness between 0.1 and 3.0 mil (2.54 and 25.4 μm). Thicknesses of 1.0 to 3.0 mils (25.4 to 78 μm) are common for corrosive service, while deposit thicknesses above 3.0 mils (78 μm) are typical of repair and rework applications. Deposition of these heavier coatings (3.0 mils) requires more careful attention to process control to avoid roughness and pitting.

Brightness

The brightness and reflectivity of electroless nickel vary significantly, depending on the specific formulation. The reflectivity is also affected by the surface finish of the substrate. Thus a very bright electroless deposit may appear dull if the substrate is rough.

The appearance of electroless nickel is similar to that of electrodeposited nickel, but slightly more yellow.

GOOD EN PLATING PRACTICE

Achieving the full potential of electroless nickel plating requires that the finisher pay attention to the original metallurgical surface condition before he ever begins to put the part in an electroless nickel processing line. In a like fashion, the part must be cleaned properly; the right equipment must be available for precleaning the parts and for operating the electroless nickel plating solution; the solution best able to produce the required properties must be used; the finisher must recognize the common processing problems and be able to correct them; and he must know how to heat treat and provide other postplate treatments required to achieve certain properties. The following sections elaborate on the metallurgy and processing methods important in producing sound deposits.

Substrate Effects

Substrate surface smoothness influences the protective value of electroless nickel deposits. The smoother the surface to be plated, the better the quality of the electroless nickel deposits.

Fabricating operations such as rolling, stamping, casting, shearing, lapping, drawing, machining and grit blasting can cause defects in the basis metal before it enters the electroless nickel process line. Inclusions in the substrate metal may cause the part to be hard to clean and not easily wetted. This will make uniform coverage with electroless nickel difficult even when thick deposits are applied. In addition, pores in the substrate can entrap preplate chemicals, which then "bleed out" during the plating cycle, causing inferior electroless nickel deposits at those sites.

When it is necessary to electroless nickel plate substrates with surface defects, the quality of plate can be improved by alternating hot and cold rinses during the preplate cycle, running the bath in a slow mode, increasing the rate of agitation and lowering the plating bath surface tension with an approved wetting agent.

Preparation of Metals for Electroless Nickel Plating

The importance of cleaning and activating metal surfaces prior to electroless

Nickel plating cannot be overemphasized. Many of the problems thought to be caused by improper electroless nickel plating are actually caused by failure to clean and pretreat surfaces adequately. Here are recommended preplate steps for each of the metals commonly plated with electroless nickel.

Nickel

- 1) Soak clean.
- 2) Rinses.
- 3) Anodic or periodic reverse electroclean.
- 4) Rinses.
- 5) Acid dip - 10% sulfuric acid.
- 6) Rinses.
- 7) Electroclean - anodic or periodic reverse.
- 8) Rinse.
- 9) Electroless nickel plate.

Copper

- 1) Soak clean.
- 2) Rinses.
- 3) Electroclean.
- 4) Rinses.
- 5) Dip in 5 to 10 pct sulfuric acid.
- 6) Rinses.
- 7) Contact the surface to be plated with a steel rod, or apply direct current momentarily to initiate deposition. Since copper is not catalytic, this is necessary to begin deposition.
- 8) Electroless nickel plate.

Aluminum

Single-zincate process.

- 1) Non-etch soak clean.
- 2) Rinse.
- 3) Alkaline or acid etch.
- 4) Rinse.
- 5) Desmut.*
- 6) Rinse.
- 7) Zincate.
- 8) Rinse.
- 9) Electroless nickel plate.

Double Zincate Process:

- 1) Non-etch soak clean.
- 2) Rinse.
- 3) Alkaline or acid etch.
- 4) Rinse.
- 5) Desmut.*
- 6) Rinse.
- 7) Zincate - long immersion.
- 8) Rinse.

- 9) Strip in 50 pct nitric acid.
- 10) Rinse.
- 11) Zincate - short immersion.
- 12) Rinse.
- 13) Electroless nickel plate.

*Desmut in nitric acid 50 pct, or nitric acid 50 pct plus sulfuric acid 15 pct, or nitric acid 50 pct plus sulfuric acid 25 pct plus hydrofluoric acid 5 pct.

Equipment

Tanks. The most basic item in a line for plating electroless nickel is a clean polypropylene tank of a known and calibrated capacity. Before the tank is used, a dilute caustic solution heated to 120F (49C) should be pumped into the tank and allowed to leach out impurities.

After the caustic has been drained out and the tank has been rinsed, a 30 pct solution of nitric acid should be pumped in and then recirculated through the tank and accessory equipment.

New stainless steel heaters should be immersed in the nitric solution overnight for adequate passivation. Either 304 or 316 stainless steel is acceptable. After use, the nitric acid solution should be pumped to a holding tank and saved for tank maintenance.

A good rinse should always follow each passivation period. A small amount of ammonia added to the final rinse and pumped through the system will neutralize residual acidity.

Filters. For the tank of 150 gallons or less, cartridge-type filter units will be adequate. Such units can be purchased in appropriate sizes, and can be connected to a distribution manifold.

Before new filters are used they should be rinsed thoroughly with a five pct sulfuric acid solution containing 0.1 pct of a suitable wetting agent. After this leaching operation the filter tubes or bags should be rinsed in deionized water before being used to filter the EN solution.

If polypropylene filter bags have been used before and subsequently stripped in nitric acid to remove nickel particles, they should be rinsed in deionized water containing a small amount of ammonia, to neutralize nitric residue, before being used again. Periodic laundering of these bags is advisable, as the nitric does not remove dirt. This dirt will cause more rapid plate-out in the filters, and subsequently lower the metal content of the bath.

Racks, Barrels. Consideration must be given to rack and barrel design and maintenance to minimize costly problems resulting from cross contamination of baths due to defective rack coatings and undersized holes in plating barrels (limiting solution transfer).

should be designed to provide for good drainage of processing solutions on racked parts. Certain shapes tend to entrap solution, which then causes contamination by carryover or dragin.

Makeup, Replenishment and Operation

proprietary electroless nickel product is a blending of many ingredients carefully balanced to provide long bath life and consistent, reproducible deposits. It is important that the user follow instructions provided by supplier so as not to inadvertently disturb this aforementioned balance. The following is a list of primary points of which the user should be cognizant.

-) Tank volume should be known. Tank volume (gallons) = $\frac{\text{length(in.)} \times \text{X depth(in.)} \times \text{X width(in.)}}{231}$.
-) Deionized water should be used.
-) Temperature should be controlled.
-) The solution should be filtered at proper rates through appropriate media.
-) Makeup and replenisher components should be used according to supplier instructions.
-) Replenisher containers should be well marked.
-) The specified pH should be maintained.
-) A replenishment/control log should be maintained.
-) Standardized reagents should be used for analysis.
-) Bath should not be allowed to become too depleted, so that "shocking the bath" with large replenisher additions may be avoided.
-) Working solution volume level should be maintained, especially before analysis.
-) Racks, hooks, or barrels used for cadmium, chromium, lead, solder, tin or zinc plating should not be used in the electroless nickel solution.
-) Bath should not be heated for extended periods of time unnecessarily.
-) Chemical balance of the bath should not be upset by unauthorized additions by a "creative" operator.
-) Bath should not be used beyond its normal life.

Calculations required for an electroless nickel bath should be mixed according to instructions. The pH should be checked and adjusted to suggested working range. The tank should then be marked for continuous level observation. Care should be taken, at this point, to allow for solution contained in the pump and lines outside the tank. Not taking this into consideration can cause serious errors in the bath and subsequent titration errors.

At this point, a sample of bath should be taken and reserved for a laboratory for EDTA titrations. A graph should now be made of pct Ni content versus ml of EDTA. This will allow rapid checking of bath conditions during operation hours, without going through a series of calculations where errors are likely to occur. The bath is now ready to be brought to recommended operating conditions.

Bath pH ranges required vary with different bath formulations and different suppliers. Appropriate instructions should be followed. Since variations in pH can cause erratic operation, it is best to check pH frequently and make necessary corrections. Although most proprietary baths maintain pH within the desired range with proper replenishment, complex parts can cause excessive "dragin" to cause pH variations. When parts of this type are being processed, the pH should be checked continuously and appropriate adjustments should be made to keep the bath pH within the specified working range.

Some electroless nickel baths are extremely susceptible to failure after any substantial amount of dragin contamination; therefore, a concentrated effort must be made to minimize dragin.

Periodic titrations and accurate records of results are a must for any shop that wants to do government or other specification electroless nickel plating. Record keeping is very important.

It is not necessary to replenish every time a bath is titrated, unless the bath is out of optimum working range. Anything less than 85 pct could drop the deposition rate enough to slow production.

A good way to assure continued uniform deposition rates and eliminate unnecessary titrations is to measure thickness of plated panels. Panels of known, consistent original thickness may be hung on racks carrying parts through. Periodically, throughout the run, a panel may be removed from the rack and deposit thickness measured. Parts being plated will, of course, have this same deposit thickness. Also, deposition rate of the bath can thus be determined. A properly processed panel will demonstrate good plate adhesion.

Usually it is more economical to have various sizes of tanks containing electroless nickel solutions rather than to process all work in one large tank. Not only is it expensive to heat and maintain a large tank, but there is a detrimental effect on the bath itself if only a few small parts are being plated in a large solution volume.

EN solutions should not be heated unless they are to be used. If a bath is continuously heated and not used, or if just one or two small parts are to be plated daily, deposition rate will fall. Depending on the formulation, other problems also may occur.

An important item to be considered in production usage of electroless nickel is filtration. Continuous filtration using equipment able to filter at hourly rates equivalent to 10 times the solution volume is good practice. A 100 gal tank should have a 1,000 gph filter, for example.

Mechanical agitation is usually suggested for electroless nickel baths, but this is practical only in small tanks and then not without certain drawbacks. Most conventional baths will accept air agitation from an oil-free blower.

Air agitation will eliminate many roughness and gas-pitting problems. It will eliminate the need for continuous or periodic moving of parts hung in a bath. Better solution changeover will be produced in barrel plating, too.

Precautions must be taken in plating shapes which can trap air on entry into rinsing tanks. This air could block access of solution to areas of the part to be plated. Wherever air can be trapped, hydrogen or oxygen gas may also be evolved during a cleaning or plating step.

Rinsing is mandatory. Air-agitated counterflowed rinses after all plating steps are best. They may be supplemented with sprays where practical. All rinses should have overflows along the entire length of the tank to flush the whole surface of the tank continuously—not just at one corner. Rinsing should be kept at or near room temperature. Very cold rinses will cause certain cleaners on the part surface rather than remove them. A two-second dip in a rinse tank is not enough; rinsing should be thorough.

Users of electroless nickel use one tank of electroless nickel solution day after day. Even though an adequately filtered bath usually will last through a year, daily transfer is preferable.

Electroless nickel tanks are best set up in pairs. One is operated one day and the other the next. Nitric solution (30 pct) is left overnight in the previously used tank to remove any particulate matter and to passivate the heaters and other metal parts. This procedure should be followed even though the tank is clean. When the working tank is being heated and the first load of parts is in the plating cycle, the nitric solution should be pumped to a holding tank for the next day, and the plating tank rinsed out. Now the plater has a working tank and a clean spare.

For some reason the solution has to be drained immediately, it can be pumped to the clean tank, the heater turned on, and production may continue. It happens sometimes when a critical part is dropped, or when a triggering mechanism has started and must be stopped by filtering the entire bath quickly. Another reason for daily transfer, even when a high filtration rate is used, is that the geometry of a tank does not allow every bit of solution to be filtered, no matter how many times per hour the bath is turned over.

When parts become plated, they should be stripped in the stronger concentration of nitric acid. In most instances, 30 pct nitric is not strong enough to remove large quantities of nickel in a short period of time, especially after heaters have been used for a while.

Finally, a poly-lined drum of stronger solution can be kept without too much inconvenience. This will save the tank from unnecessary exposure to a strong oxidizing solution. Even with daily transfer practices and nitric exposure, these tanks will last two to five years.

This may seem to be a complicated procedure, but if practiced daily, it is a fast and inexpensive one. It will eliminate costly downtime and loss of baths by plate-out or triggering.

POST TREATMENTS

Heat Treatment

Heat treatment is frequently used to improve adhesion or to modify properties in order to satisfy the needs of a particular application. As a result of heat treatment, hardness, corrosion resistance, wear resistance, ductility and stress, fatigue properties, magnetic properties, and other qualities of the deposit can be affected. Figure 1 indicates changes of EN as a result of heat treatment. Figure 2 graphs changes in hardness and wear resistance of EN following heat treatment at different temperatures. (Additional details are provided in the "Deposit Properties" section of this book.)

Heat treatment is normally performed at temperatures of 200 to 750F (93 to 400C) for 30 minutes to several hours. Maximum hardness is produced by heating at 750F(400C), followed by cooling slowly to 390F (200C) or lower. The higher temperatures are less likely to be used in commercial practice, since most processors prefer to treat at lower temperatures for longer time periods. The normal range is 200 to 300F (93 to 149C) for 30 minutes to several hours. Heat treatment at above 500 to 550F (260 to 288C) will change physical,

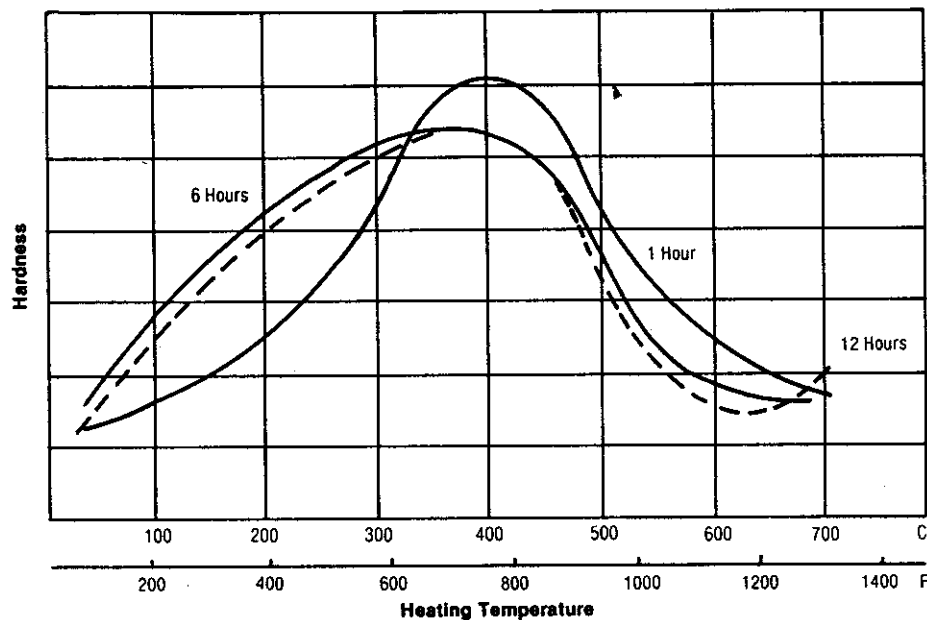


Fig. 1—Hardness versus temperature in heat treating electroless nickel.

TABLE III

FAULTY DEPOSITS—POSSIBLE CAUSES, SUGGESTED REMEDIES

PROBLEMS	POSSIBLE CAUSES	SUGGESTED REMEDIES	
1) Skip plating, poor coverage, edge pullback and frosted edges.	a) Improper cleaning.	a) Temperature should be checked as well as purity and concentration of cleaner, other pretreatment solutions.	
	b) Improper activating.	b) Activators should be checked. Some metals and alloys, such as leaded steels, brasses, copper, aluminum and magnesium, require special preparation.	
	c) Improper rinsing.	c) Rinse temperature and rinsing time should be checked. Too long a time in a rinse may cause an oxide film to form; too short a time may not remove residual films. Rinses must be kept clean.	
	d) Metallic contamination.	d) Bath should be dummied, or discarded and replaced.	
	e) Organic contamination.	e) Bath should be cooled, carbon treated and filtered. Rinses should be checked for residual drag-in.	
	f) Too much air agitation.	f) Air agitation should be reduced.	
	2) Roughness in deposit.	a) Contamination from solid particles; i.e., dust, loose nickel or metal chips.	a) Solution should be filtered through 5 micro filter at high flow rates.
		b) Turbidity of solution.	b) May be result of high pH, which aids in formation of metallic hydroxides. pH should be checked and adjusted. Complexers may be added.
		c) Heavy or too rapid EN solution replenishment while work is being plated.	c) Replenisher should be added slowly and mixed thoroughly. Additions should be made in area as far away from work as possible.
		d) Contaminated makeup water.	d) Quality of water should be checked. Water may require carbon treatment and filtration.
e) Localized overheating.	e) Air agitation should be checked or temperature adjusted. Derated heaters should be used.		
f) Particles of metal or soil on work.	f) Cleaning and rinsing should be improved.		
g) Contaminated filter cartridges, liners or bags.	g) Liners should be leached prior to makeup. Filters should be changed.		
h) Only one side of work affected.	h) Agitation around work should be increased. Work rod or solution agitation should be provided.		
i) High sodium phosphate concentration.	i) All or part of the solution should be discarded.		

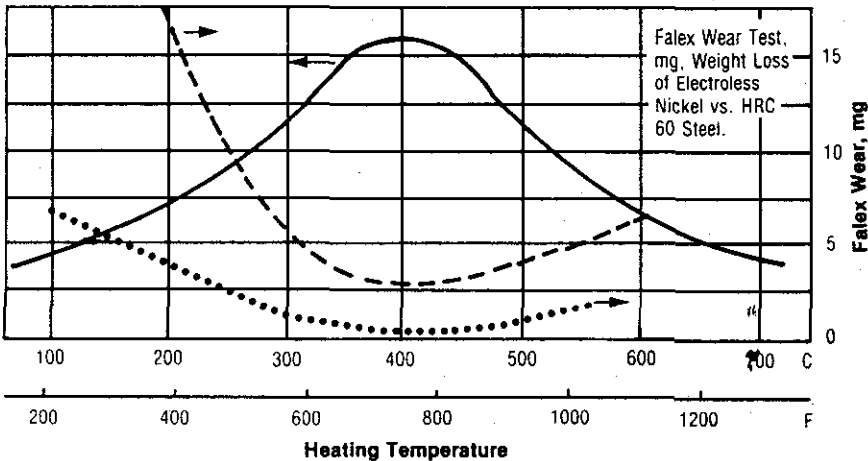


Fig. 2—Hardness and wear resistance of electroless nickel versus heat treatment.

mechanical and protective properties of EN. This is due to precipitation of nickel phosphide, which begins to occur in this range.

Heat treatment at temperatures and times beyond those required to develop maximum hardness increases the ductility of the deposit. Typical coatings will withstand six pct elongation without failure, provided that the basis metal is not stressed beyond its elastic limit.

Heat treating should be carried out in an inert atmosphere such as one of argon or nitrogen, in order to minimize oxidation. If the temperature is increased beyond 500F (260C) in air, a highly discolored surface results. In addition to the poor appearance of such oxides, there are problems in soldering heavily oxidized surfaces.

Chromate Coatings

Proprietary chromating solutions are sometimes used to passivate and help protect the substrate from corrosive attack.

CAUSES OF FAULTY DEPOSITS

Some common problems in electroless nickel plating, possible causes of the problems, and suggested remedies are contained in Table III.

TABLE III (continued)

PROBLEMS	POSSIBLE CAUSES	SUGGESTED REMEDIES	
Streaks in deposit.	a) Gas streaks from position of work.	a) Either solution or work rod agitation should be provided. Work should be repositioned periodically. Agitation should be increased.	
	b) Silicate dragin	b) Non-silicated cleaners should be used. Rinsing should be improved.	
	c) Poor rinsing.	c) Process cycle should be checked, rinsing improved.	
	d) Improper cleaning.	d) Cleaning and/or rinsing should be improved.	
	e) High concentration of metals.	e) Bath should be dummied to remove metals. Bath may have to be discarded and replaced with new solution.	
	f) Organic contamination.	f) Bath should be carbon treated and filtered, or discarded and replaced with new solution.	
	g) Poor agitation.	g) Air agitation should be increased, or a different air pattern developed.	
	h) Low surface area.	h) Surface area should be increased to recommended range.	
	i) Low reducer content.	i) Reducer should be checked and adjusted.	
	j) Too much agitation.	j) Agitation should be reduced.	
	k) Too much complexer.	k) Complexer should be reduced.	
	Pitting	a) Heavy metal contamination.	a) Bath should be dummied, or discarded and replaced.
		b) Ethylene glycol contamination from jacketed tank.	b) Bath should be discarded and replaced. Ethylene glycol must be kept from bath.
c) Basis metal pitted.		c) Basis metal should be checked after each step in the cycle.	
d) Organic contamination.		d) Bath should be cooled, carbon treated and filtered.	
e) Improper cleaning.		e) Cleaning and rinsing should be improved.	
f) Excessive bath activity.		f) pH should be lowered.	
g) Copious evolution of hydrogen.		g) Surface area being plated should be reduced. Reducing agent concentration should be checked and adjusted. If problem is being caused by plating of tank walls and equipment, it may be necessary to clean tank and equipment.	
h) Low complexer concentration.		h) Complexer should be added in small increments.	
i) Improper reducer concentration.		i) Bath should be analyzed and adjusted.	
Dull or matte deposit.	a) Organic contamination.	a) Bath should be cooled, carbon treated and filtered.	
	b) Improper cleaning.	b) Cleaning should be improved. Electrocleaning may be required.	

TABLE III (continued)

PROBLEMS	POSSIBLE CAUSES	SUGGESTED REMEDIES
	c) Bath very old (more than 15 regenerations).	c) Bath should be discarded, and new bath prepared.
	d) High or low pH.	d) pH should be adjusted with acid or alkali.
	e) Bath imbalance.	e) Nickel and reducer should be checked and bath adjusted.
	f) Poor quality substrate.	f) Substrate should be improved.
	g) Low temperature.	g) Temperature should be adjusted.
	h) Excessive brightener dragout.	h) Solution chemistry should be balanced.
	i) Metallic contamination.	i) Bath should be dummied onto a large surface.
	j) Low reducer concentration.	j) Reducer should be added.
	k) Low metal content.	k) Metal should be added.
	6) Poor adhesion on aluminum.	a) Metal contamination.
b) Improper surface preparation.		b) Cleaning and rinsing should be improved.
c) Improper zincate or other pretreat condition.		c) Concentration and aluminum substrate should be checked.
d) Reoxidation.		d) Transfer times should be reduced.
e) Dragin of acid inhibitors.		e) Uninhibited acid should be used; rinsing improved.
f) Organic contamination.		f) Bath should be carbon treated and filtered.
g) Improper heat treatment.		g) Heating time and temperature should be corrected.
h) Quality of alloy.		h) Alloy quality should be checked.
7) Frosted deposits.	a) Low work load.	a) Work load should be increased.
	b) Metallic contamination.	b) Bath should be dummied or discarded and replaced.
8) Step plating.	a) Metallic contamination.	a) Bath should be dummied or discarded and replaced.
	b) Certain alloys, such as lead, steel, brass or copper.	b) A copper or nickel strike should be used prior to plating, followed by galvanic activation for copper.
	c) Bath imbalance.	c) Bath should be analyzed and adjusted.
9) Laminar deposits.	a) Poor temperature, pH and/or bath control.	a) Uniform temperature, pH and bath control must be maintained.
10) Poor wear resistance.	a) Low heat-treatment temperature and/or short time.	a) Temperature and time cycle should be adjusted.

TABLE III (continued)

PROBLEMS	POSSIBLE CAUSES	SUGGESTED REMEDIES
	b) Low phosphorus content.	b) Phosphorus content should be increased by lowering pH and temperature. Bath should be analyzed and chemical balance restored.
Poor corrosion and/or chemical resistance.	a) Low phosphorus. b) Metallic contamination. c) Poor bath control.	a) Phosphorus content should be increased by lowering pH and increasing reducer concentration. b) Bath should be dummied or discarded and replaced. c) Uniform temperature should be maintained, pH and replenishment controlled.
Dark to black deposits.	a) High stabilizer content. b) Metallic contamination. c) Bath imbalance. d) Poor activation. e) Organic contamination. f) Improper surface preparation. g) Inadequate rinse. h) High work load. i) Low work load. j) Low reducing agent. k) Low metal concentration.	a) Bath should be dummied or diluted. b) Bath should be dummied or diluted. c) Bath should be analyzed and adjusted. d) Reactivate. e) Bath should be carbon treated and filtered. f) Cleaning, pickling and rinse cycles should be improved. g) Rinsing should be improved. h) Work load should be reduced. i) Work load should be increased. j) Reducing agent should be increased. k) Metal content of the bath should be increased.
Blistering.	a) Inadequate surface preparation. b) Improper zincating of aluminum. c) Organic contamination. d) Metallic contamination. e) Improper pH. f) Improper heat treatment.	a) Cleaning, pickling and rinsing should be improved. b) Concentration of zincate, processing time should be checked. c) Bath should be carbon treated and filtered. d) Bath should be dummied or discarded and replaced. e) pH should be checked and adjusted. f) Heat treating temperature and time should be checked and corrected.

STRIPPING ELECTROLESS NICKEL

though modern techniques for electroless nickel plating have greatly reduced the need for stripping of unsatisfactory deposits, selective stripping

of these metallic coatings may be required, either immediately after plating, or after plated parts have been in service and require rejuvenation.

Most electroless nickel deposits are highly resistant (passive) to chemical attack. The most prevalent basis metal on which electroless nickel is deposited is steel, usually of complex geometry, and *seldom* resistant to chemical attack.

Cleaning and activation of the electroless nickel surface is necessary before stripping can begin. Components which have seen severe service may require rigorous cleaning to remove organic soils, carbonaceous deposits, and other incrustations. Thorough cleaning by vapor degreasing, followed by alkaline soak and electrocleaning is recommended.

Electroless nickel deposits which have aged or have been heat-treated should be activated in acid between the cleaning and stripping cycles. Inhibited hydrochloric acid (30 to 50 pct by volume), mixed acids (40 pct by volume hydrochloric acid and 10 pct by volume sulfuric acid), or proprietary acid salts may be used for activation.

Stripping Solutions

Electroless nickel deposits are commonly stripped by immersing them in aqueous chemical solutions. Since in most instances, electroless nickel is applied to components which do not lend themselves to electroplating because of their complex geometry, electrolytic stripping or "deplating" has rarely been employed for stripping these deposits.

Immersion strippers may be classified in two chemical categories: alkaline (cyanide and non-cyanide), and acid. Alkaline solutions incorporating nitro-organic compounds and cyanide are recommended for stripping electroless nickel deposits (containing up to approximately 7 pct by weight phosphorus) from steel and steel alloy substrates. For maximum bath life and efficiency, alkaline cyanide strippers should not be operated at temperatures greater than 140F (60C).

Alkaline non-cyanide solutions utilize nitro-organic oxidizers, and replace cyanide with amino compounds. These solutions are recommended for stripping electroless nickel deposits with higher phosphorus content (8 to 14 pct by weight) from steel and steel alloy substrates. Proprietary processes of this type are available which incorporate inhibitors and permit the solutions to strip these high-phosphorus nickel deposits from certain copper and copper alloys. Alkaline non-cyanide electroless nickel strippers are more stable than the cyanide type, and may be operated at temperatures up to 200F (95C).

Solutions of nitric acid (40 to 50 pct by volume) are recommended for stripping electroless nickel from aluminum and most aluminum alloys.

ing Bath Operation

Concentration. Proprietary electroless nickel stripping solutions must be prepared at the concentrations recommended by the supplier. Overly concentrated solutions may lower the solubility of the deposit being stripped and cause localized and/or overall etching of the basis metal. Conversely, dilute solutions may reduce the rate of stripping, and cause localized solution breakdown and inadequate inhibition.

Organic and metallic impurities may adversely affect the performance of the stripping process.

Immersion nickel strippers become saturated with dissolved metal, and should be discarded (in accordance with pertinent regulations governing disposal of such chemicals). Since the stripping rate of these strippers is proportional to the concentration of dissolved metal, the economies of operation when the dissolved metal reaches the saturation point are far greater than those to be realized by trying to prolong bath life by chemical additions beyond the saturation point. (Saturation is reached when further additions of chemicals do not significantly increase the stripping rate.)

Temperature. Certain types of electroless nickel strippers are designed to operate within specific temperature ranges. Temperature control, therefore, is of great importance for good stripping results.

Agitation. Electroless nickel stripping solutions may require agitation when used. In most cases, agitation of the solution or the parts to be stripped will increase the rate of stripping. The primary function of agitation is to keep the stripping solution moving past the work surface. Air or gas bubbles clinging to the surfaces of the parts being stripped result in non-uniform stripping. More importantly, as the metal stripping reaction commences and progresses, a localized depletion of the chemical reactants (e.g., oxidizers, accelerators, inhibitors) occurs at the interface of the solution and the metal being stripped. Agitation of the solution or the parts serves to displace reacted products with fresh reactants.

Excessive overheating of stripping solutions may occur near heating devices, and much higher temperatures are attained than those recommended for operation of the stripping solution. Agitation serves to prevent this disfunction.

Equipment. The importance of equipment specifications relating to electroless nickel stripping cannot be overstated. Specifications must take into account:

- a) resistance to chemical attack from the operation of the stripping process.
- b) accommodation of temperature extremes in the operating solution.
- c) operator safety and prevention of health hazards.
- d) adequacy of materials of construction.
- e) prevention of electrical problems.

The dimensions of the vessel containing the stripper solution must provide clearance between the parts being stripped and the tank bottom, with an additional 12-inch clearance at the bottom for sludge accumulation. Heating and/or cooling coils, and the parts to be stripped, must be electrically insulated from the tank. All other equipment such as mechanical agitators and temperature-controlling devices in contact with the stripping solution should also be insulated to prevent stray currents from entering the tank.

Since fumes often evolve from solutions being used to strip electroless nickel, ventilation is required. Suitable corrosion-resistant materials should be used for ducts and exhaust systems.

Substrate Metallurgy. Since mechanical working, prior heat treatments, and exposure to severe service environments may result in stressed or otherwise deformed basis metal components, the metallurgy of the basis material is an important consideration in the prevention of subsequent problems during the stripping operation.

Brazed components often present unique problems with respect to substrate metallurgy. Although the electroless nickel stripper is designed to be selective for a particular brazing alloy, etching or pitting may occur. Etching or pitting usually begins where the braze interfaces with the basis metal. It is believed that these sites are vulnerable to intergranular attack because of diffusion alloying. Preliminary tests to determine the rate of attack should be made on any brazed component before deciding on any method of or solution for stripping electroless nickel deposits.

High-strength, hardened and tempered steels may also be subject to pitting and etch. Machining, drawing, stamping, and other mechanical working operations affect substrate metallurgy. Components made from these types of steels and alloys usually experience wear, damage, and corrosion in service. Consequently, the substrate may be non-heterogeneous with respect to its electrochemical activity in an electroless nickel stripping solution.

Once all of the electroless deposit has been removed, the stripping solution may preferentially attack certain areas along grain boundaries in the substrate, even though the stripper has been properly formulated and maintained. The selective stripping of electroless nickel deposits should be regarded as a finishing process. The course of the stripping action must be monitored. It is good practice to remove the components from the solution as soon as the electroless nickel deposit has been stripped. With good equipment, careful selection of the stripping material, and controlled operation of the stripping solution, valuable components, which would otherwise be scrapped, can be salvaged.

Typical stripping rates are 0.4 to 0.6 mil/hr except for very high phosphorus alloyed deposits and certain heat-treated or aged deposits. These will strip at a rate of 0.2 to 0.4 mil/hr.

SPECIFICATIONS AND TESTS FOR ELECTROLESS NICKEL PLATE

There are a number of specifications and test methods commonly used to judge the quality of electroless nickel. The tests described below apply to nickel-phosphorus deposits. They do not cover testing of deposits from solutions reduced by borane, hydrazine or other reducing agents. The tests mentioned do not cover all possible physical properties, but do cover those normally of interest to users of electroless nickel: hardness, thickness, porosity, corrosion resistance, solderability and phosphorus content. A number of the tests are those developed by the American Society for Testing and Materials (ASTM), 916 Race Street, Philadelphia, PA 19103 (tel. 215-299-5400). Further information is available from ASTM.

Hardness

Hardness may be determined by the method outlined in ASTM B-578 "Microhardness of Electroplated Coatings", using a 100-gram load and a deposit thickness of two mils unless otherwise specified.

Thickness

Thickness of deposits may be determined by microscopically examining a cross-section, by beta backscatter methods, or by x-ray fluorescence. The deposit thickness also can be measured by using a micrometer before and after processing the article or a test specimen.

The microscopic examination of the cross-section of the article to be tested should be in accordance with ASTM B-478 "Standard Method for Measurements of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross-Section." Deposits on metals which have an atomic number less than 18 or greater than 40 can be measured by the use of a beta backscatter device. This test should be in accordance with ASTM B-567 "Standard Method for Measurement of Coating Thickness by the Beta-backscatter Principle."

Porosity

Plated parts may be inspected for pits and porosity by a number of methods.

Ferroxyl test. This test is for use with EN on steel and iron basis metals. Prepare a test solution by mixing 25 grams of potassium ferricyanide and 15 grams of sodium chloride in one liter of deionized water. Clean the article and immerse in test solution for five seconds. Blue spots visible on the surface indicate pore sites.

Copper sulfate test. This test is also for deposits on iron and steel basis metals. Immerse or swab the deposits for 15 seconds using 60 g/liter copper sulfate acidified to pH 3.0 with sulfuric acid. Pore sites will be indicated by copper-colored spots on the deposit.

Alizarin test. This test for deposits on aluminum alloys is performed by wiping a test specimen with a 10 pct sodium hydroxide solution. After three minutes, rinse and apply the solution of Alizarin sulfonate.

After four minutes, apply glacial acetic acid until the violet color disappears. Red spots indicate pore sites. The Alizarin sulfonate solution is prepared by dissolving 1.5 grams of methyl cellulose in 90 milliliters of boiling deionized water, to which, after cooling, a solution of 0.1 gram of Alizarin sulfonic acid dissolved in five milliliters of ethanol is added.

Hydrochloric spot test. This test is for deposits on aluminum alloys. It is performed by immersing the article which has been plated into a solution of 50 pct hydrochloric acid at room temperature for two minutes. Black spots on the surface indicate pore sites.

Five pct neutral salt spray test. This test may be used on all alloys and should be in accordance with ASTM Standard B-117, "Method of Salt Spray (fog testing)."

Electrochemical pitting test. This test also can be used on any basis material, in accordance with ASTM G-61, "Standard Practice for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion."

Corrosion

Corrosion test methods may be used to determine the corrosion rate of the deposit in various environments.

The immersion weight loss test is performed on all types of basis materials, in accordance with ASTM G-1, "Standard Recommended Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens."

Electrochemical test methods also can be used on all types of basis materials, in accordance with the following methods: ASTM G-3, "Standard Recommended Practice for Electrochemical Measurements and Corrosion Testing"; ASTM G-5, "Standard Recommended Practice for Standard Reference Methods for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements"; ASTM G-59, "Standard Practice for Conducting Potentiodynamic Polarization Resistant Measurements".

Solderability

Solderability tests may be performed by heating a plated article to 450F (232C) and applying a 60-40 tin-lead solder. This solder shall wet the surface, indicating that the deposit is solderable.

Other tests are available for electroless nickel-phosphorus deposits, and they should be agreed upon between the purchaser and the applicator of the coatings.

TERMINOLOGY

Autocatalytic	Deposition of a metal coating by controlled chemical reduction, catalyzed by the metal or alloy being deposited.	Hydrogen Embrittlement	Embrittlement of a metal or alloy caused by absorption of hydrogen during a pickling, cleaning, or plating process.
Abrasive Blasting	A process for cleaning or finishing by means of an abrasive directed at high velocity against the work piece.	Oxidizing Agent	A compound that causes oxidation, thereby itself becoming reduced.
Activation	Elimination of a passive condition on a surface, usually by chemical removal of oxides.	pH	The cologarithm (negative logarithm) of the hydrogen ion activity; less than precisely, the acidity or alkalinity of a solution. Low pH indicates acidity, high pH, alkalinity.
Adhesion	The attractive force that exists between a deposit and its substrate. Can be measured as the force required to separate a deposit from its substrate.	Passivity	The condition of a metal that retards its normal reaction in a specified environment. Associated with the assumption of a potential more noble than its normal potential. Less precisely, an oxidized or inert surface.
Amorphous	Noncrystalline, or devoid of regular structure.	Peeling	The detachment or partial detachment of an electrodeposited coating from a basis metal or undercoat.
Barrel Plating	Plating or cleaning in which the work is processed in bulk in a rotating container.	Pickling	The removal of oxides or other compounds from a metal surface by means of a chemical solution.
Basis Metal	Material upon which coatings are deposited.	Pit	A small depression or cavity produced in a metal surface during deposition or by corrosion.
Blister	A dome-shaped imperfection or defect, resulting from loss of adhesion between a metallic deposit and the substrate.	Reducing Agent	A compound that causes chemical reduction, thereby itself becoming oxidized.
Buffer	A compound or mixture that, when contained in solution, causes the solution to resist change in pH. Each buffer has a characteristic limited range of pH over which it is effective.	Regeneration	The replacement of 100 pct. of the original nickel metal content of an electroless nickel bath.
Carryover	See: Dragin .	Resist	A material applied to a part of the surface of an article to prevent metal deposition on or metal removal from that area during chemical or electrochemical processes.
Chelate Compound	A compound in which the metal is contained as an integral part of a ring structure and is not readily ionized.	Shelf Roughness	Roughness on upward facing approximately horizontal surfaces where undissolved solids have settled on parts during a plating operation.
Cleaning	The removal of grease and other foreign materials from surfaces.	Step Plating	Nonuniform thickness, striations, skip-type plating. Also referred to as edge pullback and/or worm tracking.
Contact Plating	Deposition of a metal by the use of an internal source of current, by immersion of the work in solution in contact with another metal.	Strip	To remove a coating from the basis metal or undercoat.
Corrosion	Gradual solution or oxidization of a metal.	Triggering	Spontaneous decomposition of the bath caused by over-concentration or introduction of contaminants such as metal filings or particles.
Cycle	See: Regeneration . Also, processing steps in order through which work is taken to accomplish electroless nickel plating.	Turnover	See: Regeneration .
Degreasing	The removal of grease and oils from a surface.	Water Break	The appearance of a discontinuous film of water on a surface, signifying nonuniform wetting and usually associated with a surface contamination.
Dragin	The water or solution that adheres to the objects introduced into a bath.	Work (Plating)	The material, parts or products being plated or processed.
Dragout	The solution that adheres to the objects removed from a bath.		
Ductility	The ability of a material to defoam plastically without fracturing.		
Electroless Plating	Deposition of a metallic coating by a controlled chemical reduction that is catalyzed by the metal or alloy being deposited.		
Electroplating	The electrodeposition of an adherent metallic coating upon an electrode for the purpose of producing a surface with properties or dimensions different from those of the basis metal.		
Etch	To dissolve unevenly a part of the surface of a metal, resulting in low-reflectivity matte finish.		
Filter Aid	An inert, insoluble material, more or less finely divided, used as a filter medium or to assist in filtration by preventing excessive packing of the filter cake.		
Hard Chromium	Chromium plate for engineering rather than decorative applications. Not necessarily harder than decorative chromium.		

SOURCES OF INFORMATION

Societies and Associations

ALUMINUM ASSOCIATION
818 Connecticut Ave., N.W.
Washington, D.C. 20006
(202) 862-5100

**AMERICAN ELECTROPLATERS & SURFACE
FINISHERS SOCIETY, INC.**
12644 Research Parkway
Orlando, FL 32826
(305) 281-6441

**AMERICAN SOCIETY FOR
ELECTROPLATED PLASTICS, INC.**
1133 Fifteenth Street, N.W.
Washington, D.C. 20005
(202) 429-9440

AMERICAN SOCIETY FOR METALS
Metals Park, OH 44073
(216) 338-5151

**AMERICAN SOCIETY FOR TESTING AND
MATERIALS (ASTM)**
1916 Race Street
Philadelphia, PA 19103
(215) 299-5400

**METAL FINISHING SUPPLIERS'
ASSOCIATION, INC. (MFSAI)**
801 N. Cass Avenue
Westmont, IL 60559
(708) 887-0797

**NATIONAL ASSOCIATION OF
CORROSION ENGINEERS (NACE)**
Post Office Box 218340
Houston, TX 77218
(713) 492-0535

**NATIONAL ASSOCIATION OF METAL
FINISHERS (NAMF)**
401 N. Michigan Avenue
Chicago, IL 60611
(312) 644-6610

MFSA MEMBERSHIP ROSTER AS OF JANUARY, 1991

A-10 EQUIPMENT CORP.
120 W. Bellevue Drive #104
Pasadena, California 91105

AA-GEM CORPORATION
4221 N. Lawndale Avenue
Chicago, Illinois 60618

A BRITE COMPANY
8005 Sovereign Row
Dallas, Texas 75247

ABTREX INDUSTRIES INCORPORATED
28530 Reynolds Avenue
Inkster, Michigan 48141

ACME MANUFACTURING COMPANY
650 W. 12 Mile Road
Madison Heights, Michigan 48071-2484

ACI CHEMICALS, INC.
140 Industrial Street
Lancaster, Texas 75134

AD-TECH ASSOCIATES
7115 Ashlawn Drive
Brecksville, Ohio 44141

ADVANCED CHEMICAL SYSTEMS, INC.
2515 Three Mile Road, N.W.
Grand Rapids, Michigan 49504

AEON CORPORATION
7202 N. Shadeland Ave., Suite 114
Indianapolis, Indiana 46250

AGATE LACQUER MANUFACTURING CO., INC.
11-13 Forty-Third Road
Long Island City, New York 11101

ALCHEM CORPORATION
3615 Superior Avenue
Cleveland, Ohio 44144

ALCOA SEPARATIONS TECHNOLOGY, INC.
181 Thorn Hill Road
Warrendale, Pennsylvania 15086

ALDOA COMPANY
12727 Westwood Avenue
Detroit, Michigan 48223

ALDONEX, INC.
1917 St. Charles Road
P.O. Box 148
Bellwood, Illinois 60104

**ALLIED-KELITE DIVISION
WITCO CHEMICAL CORPORATION**
2701 West Lake Street
Melrose Park, Illinois 60160

ALLIED PLATING SUPPLIES, INC.
A Chromadyne Corporation
P.O. Box 2127
5000 East 10th Court
Hialeah, Florida 33012

ALLOYCRAFT LIMITED
12 Raitherm Road
Toronto, Ontario M6B 1S6, Canada

AMERICAN CHEMICAL & REFINING CO., INC.
P.O. Box 120
Waterbury, Connecticut 06720

AMERICAN PLATING SYSTEMS
2150 Maple Privado
Ontario, California 91761

AMERICAN TRIPOLI
222 Oneida Street
Seneca, Missouri 64845

**AMERICHEM DIVISION
INTERNATIONAL METALS & CHEMICALS, INC.**
5129 Unruh Avenue
Philadelphia, Pennsylvania 19135

AMES METAL PRODUCTS CO.
4323 S. Western Boulevard
Chicago, Illinois 60609

AMETEK, INC.
Haveg Division
900 Greenbank Road
Wilmington, Delaware 19808

ANODE PRODUCTS CO., INC.
9425 Seymour Avenue
Schiller Park, Illinois 60176

AQUALOGIC, INCORPORATED
30 Devine Street
North Haven, Connecticut 06473

ATLANTES CHEMICAL SYSTEMS, INC
303 Silver Spring Road
Conroe, Texas 77303

ATOCHEM NORTH AMERICA
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Woodbridge, New Jersey 07095

ATOCHEM NORTH AMERICA
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(EL JOHNSON (CANADA) INC.
Process Equipment Division
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BAKER BROTHERS/SYSTEMS
Division of Systems Eng. & Mfg. Corp
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Northampton, Massachusetts 02072

E. BAKER COMPANY
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Northampton, Massachusetts 02138

ASF CORPORATION
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Eastland, Michigan 48192-3736

AYLIS AMERICAN CO., INC.
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ELKE MANUFACTURING COMPANY
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BENCHMARK, INC.
660 13th Street
Eastland, Michigan 48192

BREWERY RECOVERY TECHNOLOGIES, INC.
390 Hopkins Street, Unit #4
Whitby, Ontario L1N 2C3, Canada

BIO-RECOVERY SYSTEMS, INC.
1001 Copper Avenue
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BIRCHWOOD CASEY
Division of Birchwood Laboratories, Inc.
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Eden Prairie, Minnesota 55344

BISON CORPORATION
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Canton, Ohio 44707

BROCO PRODUCTS, INC.
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Cleveland, Ohio 44110

THE BUCKEYE PRODUCTS COMPANY
7020 Vine Street
Cincinnati, Ohio 45216

CP CHEMICALS INC.
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Fort Lee, New Jersey 07024

CAMAC INDUSTRIES
18 Commerce Road
Fairfield, New Jersey 07004

CANADA COLORS AND CHEMICALS LIMITED
80 Scarsdale Road
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P.O. Box 67
Jamestown, New York 14702

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Sterling Heights, Michigan 48312

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CHEMICAL FINISHING, INC.
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CHEMICAL SYSTEMS, INC.
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CHEMIX CORPORATION
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CHEMJAY INC.
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CLAYTON ENVIRONMENTAL CONSULTANTS, INC.
22345 Roethel Drive
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Wilmington, Delaware 19802

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1955 Stephenson Highway
Troy, Michigan 48083

CORNING INCORPORATED
Corning Process Systems
Big Flats Plant
Big Flats, New York 14814

CORROTEC INC.
P.O. Box 1693
Springfield, Ohio 45501

FRANK J. COSCINA ASSOCIATES, INC.
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COSMOS MINERALS CORPORATION
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DEVECO CORPORATION
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E.I. DU PONT DE NEMOURS & COMPANY, INC.
Industrial Chemicals Division
Chemicals & Pigments Dept.
10th and Market Streets
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DURIRON COMPANY, INC.
Filtration Systems Division
9542 Hardpan Road
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EG&G ROTRON
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ENGELHARD CORPORATION
Specialty Chemicals Division
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IN F. FLETCHER, INC.
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Monticello, New York 13902

FORBES COMPANY, INC.
Box 14828
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Tribal Dryer Department
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Hollidaysburg, Pennsylvania 17557

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Ann Arbor, Michigan 48207

FOTHERINGHAM, INC.
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New York, New York 14213

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GOAD COMPANY
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FREDERICK GUMM CHEMICAL COMPANY, INC.
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HAMPDEN/MATHIEU CHEMICAL
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Springfield, Massachusetts 01101

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HAVILAND PRODUCTS COMPANY
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Grand Rapids, Michigan 49504

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HOECHST CELANESE CORP.
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IMPERIAL SMELTING CORPORATION
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INCO LIMITED
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**INDUSTRIAL CHEMICAL PRODUCTS OF
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Conover, North Carolina 28613

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Wheeling, Illinois 60090

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2745 Bond Street
Rochester Hills, Michigan 48309

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11110 Deerfield Road
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Birdsboro, Pennsylvania 19508

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KRAFT CHEMICAL COMPANY
1975 North Hawthorne Avenue
Melrose Park, Illinois 60160

KURTS & WOLFE CHEMICAL COMPANY
960 Close Avenue
Bronx, New York 10473

THE LEA MANUFACTURING COMPANY
237 East Aurora Street
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Waterbury, Connecticut 06720

LEA PRODUCTS COMPANY INC.
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Montreal, Quebec H1E 3H4, Canada

LEARONAL, INC.
272 Buffalo Avenue
Freeport, New York 11520

LIQUID DEVELOPMENT CO., INC.
3748 East 91st Street
Cleveland, Ohio 44105

DAVID K. LUCAS & ASSOCIATES
16918 Landing Drive
Spring Lake, Michigan 49456

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1180 Corporate Drive
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MAC DERMID CHEMICALS INC.
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MAC DERMID INCORPORATED
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Cleveland, Ohio 44113

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MIDWESTERN PROCESSES
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MILLHORN CHEMICAL AND SUPPLY CO.
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MISSOURI ELECTROCHEM, INC.
10958 Lin-Valle
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Jerky Rack - A Mitchell-Bate Affiliate
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MITCHELL-BRADFORD INTERNATIONAL CORP.
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MONSANTO CHEMICAL COMPANY
300 N. Lindbergh Boulevard
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MYRON L COMPANY
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Carlsbad, California 92009

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NAPCO INC.
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OAKITE PRODUCTS, INC.
50 Valley Road
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OCCIDENTAL CHEMICAL CORPORATION
360 Rainbow Blvd. South
Niagara Falls, New York 14303

OCCIDENTAL CHEMICAL CORPORATION
ED&S Division
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PALM INTERNATIONAL, INC.
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PARISH-O CO., INC.
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PARKER AMCHEM
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PLATING SYSTEMS INC.
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PLATING SYSTEMS & TECHNOLOGIES, INC.
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TECHNIC, INC.
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Fairfield, New Jersey 07006

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W. W. WELLS, LIMITED
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