

Iron fluoborate .....	30.3 oz/gal
Metallic iron .....	7.37 "
Sodium chloride .....	1.34 "
Baume at 80°F .....	19-21°
pH (colorimetric) .....	3.0-3.4
Temperature .....	135-145°F
Average tank voltage .....	2-6
Average cathode current density .....	20-90 amp/ft <sup>2</sup>
Anode to cathode ratio .....	1:1

Two-thirds of the required amount of water is placed in the plating tank and the specified amount of sodium chloride is added. When the salt has been completely dissolved, the ferrous fluoborate concentrate is added directly into the tank. Water is then added to bring the volume of the solution up to its required concentration. After this, the solution should be adjusted to the specified operating pH, clarified by treatment with activated carbon in a storage tank and then filtered. The carbon may be built-up on the filter pads and the solution circulated through the filter, but sufficient time must be allowed to remove the solid particles and other contaminants. Electrolysis at 3-5 amp/ft<sup>2</sup> is also recommended. The bath is then heated to operating temperature and is ready for use.

#### Sulfamate Bath:

The sulfamate bath is similar to the sulfate but permits higher cathode current density. Iron sulfamate is available as a purified solution concentrate. The general characteristics of the deposit with respect to stress, hardness and permissible current density is similar to those from other iron plating baths. A particular bath and its operating conditions are:

	Range	Recommended
Iron as ferrous ion .....	9-15 oz/gal	10 oz/gal
Ammonium sulfamate .....	3-6 "	4-5 "
Sodium chloride .....	3-8 "	5-6 "
pH .....	2.5-3.3	2.7-3.0
Temperature .....	110-150°F	120-140°F
Current density .....	10-100 amp/ft <sup>2</sup>	50 amp/ft <sup>2</sup>

Proprietary addition agents are available to reduce the stress and, as in other iron solutions, a wetting agent is recommended to eliminate pitting. In general, precautions against contamination, and the usual purification procedures apply to this solution.

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## NICKEL PLATING

by George A. DiBari

International Nickel Inc., Saddle Brook, NJ

Nickel plating for decorative, engineering and electroforming purposes is possible because the properties, including the appearance, of electrodeposited nickel can be controlled and varied over broad ranges.

Decorative nickel coatings are obtained by plating from solutions containing organic addition agents. The coatings are protective, mirror-bright and smooth.

Nickel coatings for engineering purposes are usually prepared from solutions that deposit pure nickel. The property sought in engineering end-uses is generally corrosion resistance, but wear resistance, solderability, magnetic and other properties may be relevant in specific applications.

Electroforming is a specialized use of the electroplating process in which nickel is deposited and subsequently removed from a mandrel to yield an all-nickel component or article. Electroformed nickel tools, such as molds, dies, record stampers, seamless belts and textile printing screens are important commercial products.

The processes used for decorative, engineering and electroforming purposes have a great deal in common, but differ in detail. The details are described below after considering what the various processes have in common.

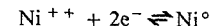
### BASIC CONSIDERATIONS

All nickel plating processes have certain features in common and are limited by natural laws which determine the rate of deposition, the thickness of the coating and its distribution. These laws make it possible to control the process and operate it continuously.

#### The Process:

Nickel plating is the electrolytic deposition of a layer of nickel upon a substrate. The process involves the dissolution of one electrode (the anode) and the deposition of metallic nickel on the other electrode (the cathode). Direct current is applied between the anode (positive) and the cathode (negative). Conductivity between the electrodes is provided by an aqueous solution of nickel salts.

When nickel salts are dissolved in water, the nickel is present in solution as divalent, positively charged ions (Ni<sup>++</sup>). When current flows, divalent nickel ions react with two electrons (2e<sup>-</sup>) and are converted to metallic nickel (Ni<sup>0</sup>) at the cathode. The reverse occurs at the anode where metallic nickel dissolves to form divalent ions. The electrochemical reaction in simplest terms is:



Because the nickel ions discharged at the cathode are replenished by the nickel ions formed at the anode, the nickel plating process can be operated for long periods of time without interruption.

## Estimating Nickel Thickness:

The amount of nickel that is deposited at the cathode is determined by the product of the current (amperes) and the time (hours). Under ideal conditions, 26.8 amperes flowing for one hour will deposit 29.4 grams of nickel (1.095 grams per ampere-hour).

If the area being plated is known, the average thickness of the nickel coating can be estimated. For example, if 29.4 grams of nickel are deposited on one square foot of area, the thickness of the deposit is 0.0014". (Thickness equals the weight of nickel divided by the product of the area and the density of nickel. It is important to use consistent units. The density of nickel is 0.322 lbs/in<sup>3</sup>).

Because a small percentage of the current is consumed at the cathode in discharging hydrogen ions, the efficiency of nickel deposition is less than 100%. This fact must be taken into account in estimating the weight and the thickness of nickel that will be deposited under practical plating conditions.

Table I is a data sheet on depositing nickel based on 96.5% cathode efficiency. The table relates coating thickness, weight per unit area, current density and time of plating. Some factors useful in making nickel plating calculations are given in Table II.

**Table I. Data Sheet on Depositing Nickel  
(Based on 96.5% Cathode Efficiency)**

Thickness in Inches	Thickness in Micrometers	Oz/ft <sup>2</sup>	G/ft <sup>2</sup>	Amp-hr	Minutes for Obtaining Coating at Various Current Densities, A/ft <sup>2</sup>			
					10	20	50	100
0.0001	2.5	0.0721	2.04	1.99	12	6	2.4	1.2
0.0002	5.1	0.144	4.08	3.98	24	12	4.8	2.4
0.0005	12.7	0.360	10.20	9.95	59.6	30	12.2	6.1
0.0008	20.3	0.578	16.32	15.9	95.6	47.8	19.2	9.6
0.0010	25.4	0.721	20.40	19.9	119.2	59.6	23.9	12.0
0.0015	38.1	1.082	30.60	29.8	178.8	89.3	35.7	18.0
0.0020	50.8	1.44	40.80	39.8	238.4	119	47.7	23.9

**Table II. Factors Useful in Making Nickel Plating Calculations  
(Assume 100% Cathode Efficiency)**

Multiply	By	To Estimate
Wt of NiSO <sub>4</sub> · 7H <sub>2</sub> O	21%	Wt of Ni contained
Wt of NiSO <sub>4</sub> · 6H <sub>2</sub> O	22%	Wt of Ni contained
Wt of NiCl <sub>2</sub> · 6H <sub>2</sub> O	25%	Wt of Ni contained
Wt of NiCl <sub>2</sub> · 6H <sub>2</sub> O	30%	Wt of Cl contained
Wt of nickel carbonate	50%	Wt of Ni contained
Amp-hrs nickel plating	1.095 0.0386	g nickel deposited oz nickel deposited
Amp-min/ft <sup>2</sup>	0.000869	Mils nickel deposited
Mils thickness	19.18 1151 0.0226 0.742	amp-hr/ft <sup>2</sup> amp-min/ft <sup>2</sup> g/cm <sup>2</sup> oz/ft <sup>2</sup>



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Anode efficiency is normally 100%. Because anode efficiency exceeds cathode efficiency by a small percentage, nickel ion concentration and pH will rise as the bath is used.

Drag-out of nickel plating solution may compensate for nickel metal build-up in solution to some extent, but at some point it may be necessary to remove a portion of solution from the plating tank and replace the solution removed with water and other constituents. The pH of the solution is maintained by adding acid.

#### Metal Distribution:

The relation between current and thickness distribution is an important basic consideration. It is desirable to apply uniform thicknesses of nickel on all significant surfaces to achieve predictable service life and to meet plating specifications which require minimum coating thickness values at specified points on the surface.

The amount of metal that will deposit on the surface of any object being plated will be proportional to the current that reaches the surface. Recessed areas on the surface will receive less current.

The current density and, consequently, the rate of metal deposition in the recessed area will be lower than at points which project from the surface. The electrodeposited coating will be relatively thin at recessed areas, and relatively thick on projecting areas (Fig. 1).

The thickness of the deposit at the cathode and the distribution of the coating can be controlled by proper racking and placement of the parts in solution, and by the use of thieves, shields and auxiliary anodes. Parts can be designed to minimize problems. It may be necessary to deposit more nickel than is specified to meet a minimum thickness requirement on a specific article.

The nickel processes used for decorative, engineering and electroforming purposes thus all have the same electrochemical reaction in common. The weight of nickel deposited at the cathode is controlled by natural laws which make it possible to estimate the thickness of the nickel deposited. These estimates must be adjusted to account for variations in cathode efficiencies for specific processes.

Normally, cathode efficiency values are between 93 and 97% for most nickel processes. The actual thickness at any point on a shaped article depends on current flow. In practice, it is necessary to measure coating thicknesses on actual parts before thickness can be controlled within a specified range.

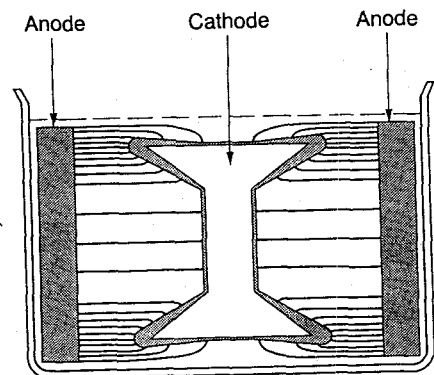


Fig. 1. Current distribution is not uniform over a shaped article. Areas remote from the anode will receive a smaller share of the available current than areas near to the anode.

## DECORATIVE NICKEL PLATING

The technology of decorative nickel plating has undergone many improvements over the years. The introduction of bright and semi-bright, nickel solutions, the development of multilayer nickel coatings, and the use of conventional, microporous and microcracked chromium in combination with multilayer nickel coatings are some examples.

#### Decorative Processes:

The solutions used for decorative plating differ from those for other applications in that they contain organic addition agents which modify the growth of the nickel deposit to produce fully-bright semi-bright and satin-like surfaces. The basic constituents — nickel sulfate, nickel chloride, and boric acid — serve the same purposes as they do in the Watts solution (Table III).

Nickel sulfate is the principal source of nickel ions; nickel chloride improves anode dissolution and increases solution conductivity; boric acid helps to produce smoother, more ductile deposits.

Anionic anti-pitting or wetting agents are required to reduce pitting due to the clinging of hydrogen bubbles to the products being plated. Nonfoaming wetting agents which lower surface tension are available for air-agitated solutions.

The composition and operating conditions given in Table III for the Watts solution are typical of many decorative nickel plating solutions, but wide variations in the concentrations of nickel sulfate and nickel chloride are possible. Since most decorative nickel plating processes are proprietary, composition and operating conditions should be controlled within the limits recommended by the suppliers.

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**Table III. Typical Compositions, Operating Conditions and Approximate Mechanical Properties of Deposits from Several Electrolytes**

Constituents	Typical Electrolyte Compositions—g/L(oz./gal)																	
	All Chloride		Chloride Sulfate		All Fluoborate		All Sulfate		All Sulfamate		Chloride Sulfamate		Hard Sulfamate		Watts		Hard Watts	
Total nickel	75	(10)	86.4	(11.5)	75	(10)	70	(9.3)	84	(11.2)	84	(11.2)	45	(6)	78	(10.4)	60	(8)
Nickel chloride	300	(40)	158	(21)					330	(44)	22.5	(3)	7.5	(1)	60	(8)	45	(6)
Nickel sulfate			188	(25)							428	(57)	248	(33)			262	(35)
Nickel sulfamate					300	(40)			450	(60)	428	(57)	248	(33)				
Nickel fluoborate							30	(4)	37.5	(5)	37.5	(5)	30	(4)	30	(4)	30	(4)
Boric acid	30	(4)	37.5	(5)	30	(4)	30	(4)	X		X		X				X	
Anti pitter	X		X		X													
Addition agent													Optional stress reducer				Optional stress reducer	
<b>Operating Conditions</b>																		
Temperature °C (°F)	55	(130)	55	(130)	55	(130)	55	(130)	60	(140)	60	(140)	55	(130)	55	(130)	50	(122)
pH	2.0		3.0		2.7		1.5		4.0		4.0		5.0		3.0		5.0	
Current density, A/m <sup>2</sup> (A/ft <sup>2</sup> )	540	(50)	540	(50)	320	(30)	400	(40)	540	(50)	540	(50)	600	(60)	540	(50)	540	(50)
<b>Mechanical Properties</b>																		
Tensile strength, MPa(ksi)	690	(100)	517	(75)	400	(58)	480	(70)	413	(60)	758	(110)	896	(130)	413	(60)	1170	(170)
Hardness, DPN (100 g load)	240		230		150		170		190		300		470		150		350	
Elongation, % in 50.8 mm (2 inches)	14		20		30		28		30		5		6		28		2	
Internal stress, MPa(ksi)	345	(50)	220	(32)	138	(20)	117	(17)	14	(2)	69	(10)	Variable according to addition agent		138	(20)	Variable according to addition agent	

**Bright Nickel Solutions:**

Bright nickel solutions contain at least two classes of organic addition agents which complement each other and yield fully-bright nickel deposits.

The first class produces deposits that are mirror-bright initially, but are unable to maintain the mirror-like appearance of the deposit as its thickness is increased. This class includes compounds like benzene disulfonic acid, benzene trisulfonic acid, naphthalene trisulfonic acid, benzene sulfonamide and sulfonamides, such as saccharin. The presence of the sulfon group and an unsaturated bond adjacent to the sulfon are critical characteristics.

Adsorption of the addition agent occurs by virtue of the unsaturated bond onto growth sites, points or edges of crystals and at dislocations. The organic compound is reduced electrochemically at the cathode, and this is accompanied by the reduction and incorporation of sulfur (as the sulfide) in the deposit. Fully bright nickel deposits typically contain 0.06 and 0.10% sulfur. These reactions control the structure and growth of the nickel as it is deposited.

The second class of addition agents may be termed leveling agents because they make the surface smoother as the thickness of the deposit is increased. They are sulfur-free, bath soluble organic compounds containing unsaturated groups and generally introduce small amounts of carbonaceous material into the deposit. Typical examples of second class brighteners are formaldehyde, coumarin, ethylene cyanohydrin and butyrol.

The combination of these two classes of organic addition agents makes it possible to obtain brilliant, lustrous deposits which are smooth over wide ranges of coating thickness. The deposits have a bonded structure consisting of closely spaced laminations believed to be related to the codeposition of sulfur.

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Other investigations concluded that chromium deposits with high porosity or crack densities on a microscopic scale would be preferable. This led to the development of microdiscontinuous chromium deposits of two types: microporous and microcracked.

These processes greatly improve corrosion performance by distributing the available corrosion current over a myriad number of tiny cells on the surface of the coating. As a result, corrosion proceeds uniformly over the entire surface, instead of concentrating at one or two pores, and the rate of pit penetration is slowed dramatically.

Double-layer nickel coatings 40 micrometers thick (1.5 mils) electroplated with either microporous or microcracked chromium and applied uniformly resisted corrosion in severe service for more than 16 years.

The progress made in the technology of decorative nickel plating has led to great improvement of the corrosion resistance of nickel plus chromium coatings. What is not often appreciated is that this has been accomplished without increasing the thickness of the nickel coating. This has kept nickel plating practical and cost competitive.

### ENGINEERING NICKEL PLATING

Engineering or industrial applications for electrodeposited nickel exist because of the useful properties of the metal. Nickel coatings are used in these applications to modify or improve surface properties, such as corrosion resistance, hardness, wear and magnetic characteristics.

Although the appearance of the coating is important and the plated surface should be defect-free, the lustrous, mirror-like deposits described in the preceding section are not usually required.

#### Engineering Plating Processes:

Table III gives typical compositions and operating conditions for many electrolytes suitable for engineering applications.

#### Mechanical Properties:

The mechanical properties are influenced by the chemical composition and the operation of the plating bath, as indicated by the information included in Table III. The tensile strength of electrodeposited nickel can be varied from 410 to 1170 MPa (60 to 170 psi), and the hardness from 150 to 740 DPN, by varying the electrolyte and the operating conditions.

The operating conditions significantly influence the mechanical properties of electrodeposited nickel. Figures 2, 3 and 4 show the influence of pH, current density and

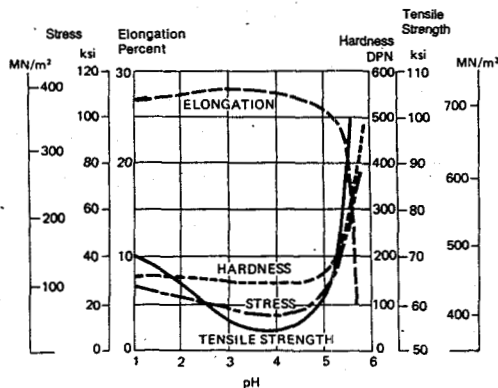


Fig. 2. Variation in internal stress, tensile strength, ductility and hardness with pH. Watts bath 54°C (130°F) and 495 A/m<sup>2</sup> (46 A/ft<sup>2</sup>).

Fig. 3. Variation in internal stress and hardness with current density. Watts bath 54°C (130°F) and pH 3.0.

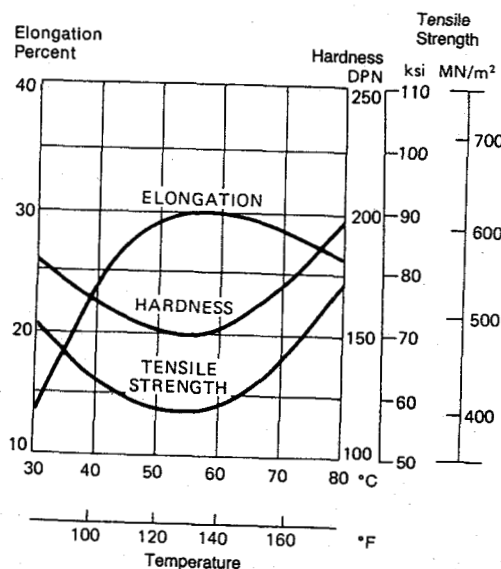
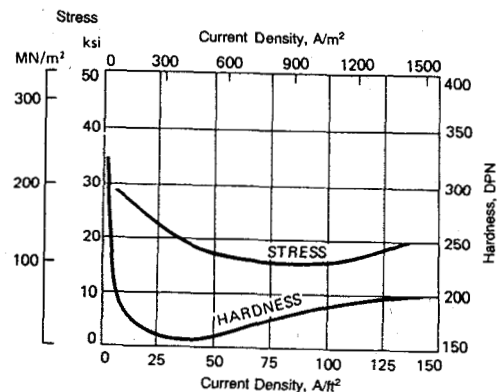


Fig. 4. Variation in elongation, tensile strength and hardness with temperature. Watts bath pH 3.0 and 495 A/m<sup>2</sup> (46 A/ft<sup>2</sup>).

temperature on the properties of nickel deposited from a Watts bath. Additional information is given in Tables IV and V which describe how various properties are affected by changes in operating variables and solution composition.

The mechanical properties of electrodeposited nickel vary with the temperature to which the coatings are exposed, as summarized in Fig. 5. The tensile strength, yield strength and ductility of electrodeposited nickel decrease steadily with an increase in temperature from -240 to 870°C (-400 to 1600°F).

The ductility of electrodeposited nickel reaches low values above 480°C (900°F). Nickel deposits from sulfamate solutions are stronger at cryogenic temperatures than deposits from the Watts bath. The electrolytes used to prepare the deposits for these studies are those given in Table VI.

**Table IV. Variables Which Affect Mechanical Properties of the Deposit-Watts Solution**

<i>Property</i>	<i>Operational</i>	<i>Solution Composition</i>
Tensile strength	Relatively independent of plating solution temperature within range suggested.	Increases with increasing nickel content.
	Relatively independent of changes in cathode current density.	Increases with increasing chloride content.
	Relatively independent of pH variation within range suggested.	
Elongation	Increases with temperature to 55°C (130°F) followed by slight decrease at higher temperature.	Decreases with increasing nickel content.
	Relatively independent of pH variation within range suggested.	
Hardness	Decreases with temperature rise to 55°C (130°F) but increases with higher temperatures.	Increases with increasing nickel content.
	Decreases significantly with increasing cathode current density to 540 A/m <sup>2</sup> (50 A/ft <sup>2</sup> ). At higher current densities the hardness increases with increasing current density.	Increases with increasing chloride content.
Internal stress	Relatively independent of plating solution temperature.	Increases slightly with increasing nickel content.
	Decreases slightly, then increases with increasing cathode current density.	Increases markedly with increasing chloride content.
	Relatively independent of pH variation within range suggested.	

**Table V. Variables Which Affect Mechanical Properties of the Deposit-Nickel Sulfamate Solution**

<i>Property</i>	<i>Operational</i>	<i>Solution composition</i>
Tensile strength	Decreases with increasing temperature to 49°C (120°F), then increases slowly with further temperature increase.	Decreases slightly with increasing nickel content.
	Increases with increasing pH. Decreases with increasing current density.	
Elongation	Decreases as the temperature varies in either direction from 43°C (110°F).	Increases slightly with increasing nickel content.
	Decreases with increasing pH.	Increases slightly with increasing chloride content.
	Increases moderately with increasing current density.	
Hardness	Increases with increasing temperature within operating range suggested.	Decreases slightly with increasing concentration of nickel ion.
	Increases with increasing solution pH.	Decreases slightly with increasing chloride content.
	Reaches a minimum at about 1300 A/m <sup>2</sup> (120 A/ft <sup>2</sup> ).	
Internal stress	Decreases with increasing solution temperature.	Relatively independent of variation in nickel ion content within range suggested.
	Reaches a minimum at pH 4.0-4.2.	Increases significantly with increasing chloride content.
	Increases with increasing current density.	

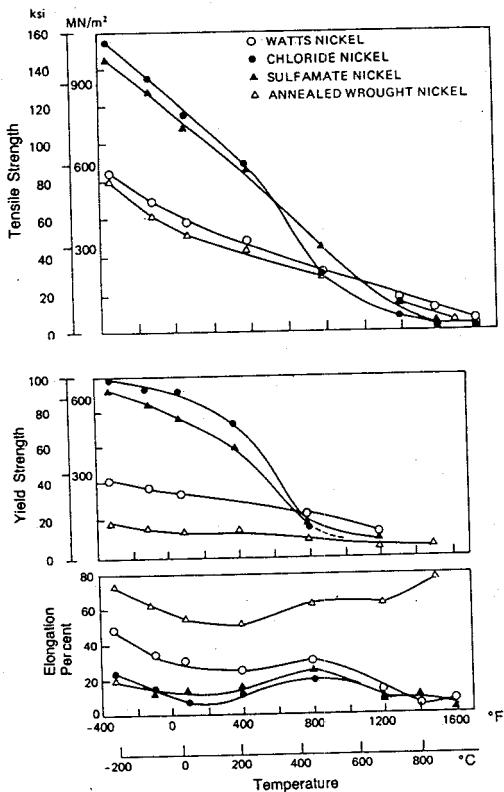


Fig. 5. Effect of temperature on the tensile strength, yield strength and elongation of electrodeposited nickel.

Table VI. Composition and Operating Conditions of Typical Industrial-Type Electrolytes

Constituent and Conditions	Plating Bath		
	Watts	All-Chloride	Sulfamate
Nickel sulfate, NiSO <sub>4</sub> · 7 H <sub>2</sub> O .....	300 g/L (40 oz/gal)	—	—
Nickel chloride, NiCl <sub>2</sub> · 6 H <sub>2</sub> O .....	60 g/L (8 oz/gal)	300 g/L (40 oz/gal)	—
Nickel sulfamate, Ni(H <sub>2</sub> NSO <sub>3</sub> ) <sub>2</sub> .....	—	—	400 g/L (60 oz/gal)
Boric acid, H <sub>3</sub> BO <sub>3</sub> .....	37.5 g/L (5 oz/gal)	37.5 g/L (5 oz/gal)	30 g/L (4 oz/gal)
pH .....	3.0	3.0	4.5
Temperature .....	60°C (140°F)	60°C (140°F)	57°C (135°F)
Current density, A/m <sup>2</sup> (A/ft <sup>2</sup> )	430 (40)	430 (40)	430 (40)

Note: Proprietary wetting agents were used to prevent pitting.

## Corrosion Resistance:

Engineering nickel coatings are frequently applied in the chemical, petroleum, food and beverage industries to prevent corrosion, maintain product purity and prevent contamination. As a general rule, oxidizing conditions favor corrosion of nickel in chemical solutions, whereas reducing conditions retard corrosion.

Nickel also has the ability to protect itself against certain forms of attack by developing a passive oxide film. When an oxide film forms and is locally destroyed as in some hot chloride solutions, nickel may form pits. In general, nickel is resistant to neutral and alkaline solutions, but not to most of the mineral acids.

Corrosion resistance in engineering applications is controlled by optimizing nickel thickness. The thickness of the nickel to be used is dependent on the severity of the corrosive environment. The more corrosive the service conditions the greater the thickness of nickel required. Thickness generally exceeds 0.003" (75  $\mu$ m).

## Nickel Plating and Fatigue Life:

Thick nickel deposits applied to steel may cause significant reductions in the composite fatigue strength in cyclical stress loading. The reduction in fatigue strength is influenced by the hardness and strength of the steel and the thickness and internal stress of the deposits.

Elimination of, or the lowering of, internal stress of the deposits is an important factor. Fatigue life is enhanced by increasing the hardness and strength of the steel, and by specifying the minimum deposit thickness consistent with design criteria.

Compressively stressed nickel deposits are beneficial. Shot peening the steel prior to plating helps minimize reduction in fatigue life upon cyclical stress loading.

## Hydrogen Embrittlement:

Highly stressed, high strength steels are susceptible to hydrogen embrittlement during normal plating operations. Because nickel plating is highly efficient, hydrogen embrittlement is unlikely to occur as a result of nickel plating, *per se*.

The pretreatment of steel prior to plating, however, may require exposing the steel to acids and alkalis. During these operations, excessive amounts of hydrogen may be evolved which may damage steels susceptible to hydrogen embrittlement.

Steels that are susceptible to hydrogen embrittlement should be heat treated at 205°C (440°F) for several hours to remove the hydrogen. The time required may vary from eight to 24 hours depending on the type of steel and the amount of hydrogen to be removed.

## NICKEL ELECTROFORMING

Nickel electroforming is the production or reproduction of products by electroplating on a mandrel which is subsequently separated from the deposit.

### Nickel Electroforming Processes:

The composition, operating conditions and mechanical properties of deposits from two electrolytes most often used for electroforming are given in Table VII. Nickel sulfate solutions are the most popular because the deposits are low in stress, high rates of deposition are possible and thickness of the deposit is less affected by variations in current densities than are deposits from Watts solutions.

Table VII. Nickel Electroforming Solutions

	Watts Nickel	Nickel Sulfamate
Electrolyte composition, g/L (oz/gal)	NiSO <sub>4</sub> ·6H <sub>2</sub> O—225-300 (30-40)	Ni(SO <sub>4</sub> NH <sub>4</sub> ) <sub>2</sub> —315-450 (42-60)
	NiCl <sub>2</sub> ·6H <sub>2</sub> O—37.5-52.5 (5-7)	H <sub>2</sub> BO <sub>3</sub> —30-45 (4-6)
	H <sub>2</sub> BO <sub>3</sub> —30-45 (4-6)	NiCl <sub>2</sub> ·6H <sub>2</sub> O—0-22.5 (0-3)
<b>Operating Conditions</b>		
Temperature	44-66°C (115-150°F)	32-60°C (90-140°F)
Agitation	Air or mechanical	Air or mechanical
Cathode current density	270-1075 A/m <sup>2</sup> (25-100 A/ft <sup>2</sup> )	50-3225 A/m <sup>2</sup> (5-300 A/ft <sup>2</sup> )
Anodes	Soluble nickel	Soluble nickel
pH	3.0-4.2	3.5-4.5
<b>Mechanical Properties</b>		
Tensile strength:		
MPa	345-482	410-620
ksi	50-70	60-90
Elongation, %	15-25	10-25
Hardness		
(Vickers hardness, 100 g load)	130-200	170-230
Internal stress:		
MPa	125-186 tensile	0-55 tensile
ksi	18-27	0-8

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## High-Speed Nickel Sulfamate Solution:

A typical high-speed nickel sulfamate solution for electroforming has a nickel sulfamate concentration of 550 to 650 g/L, a nickel chloride concentration of 5 to 15 g/L and a boric acid concentration of 30 to 40 g/L. It is operated at a pH of 4.0, a temperature of 140 to 160 °F (60 to 71 °C) and at current densities as high as 800 A/ft<sup>2</sup>.

The high rates of plating are made possible by the high nickel concentration. When the bath is properly conditioned and operated, it is possible to control internal stress and vary it over broad ranges.

The conditioning, which can be done continuously, involves electrolyzing the solution in a separate tank using an anode of low activity; i.e., regular electrolytic nickel. The conditioning tank should have 10 to 20% of the capacity of the plating tank so that the entire solution can be circulated through the conditioning tank from two to five times per hour.

The average current density on the anode and cathode in the conditioning tank should be 5 to 10 A/ft<sup>2</sup>. The current in the conditioning tank should be about 3% of the current through the main tank.

The anode material used in the main tank should be the active type; i.e., electrolytic nickel with sulfur. With a correctly conditioned bath, zero deposit stress can be achieved at 165 A/ft<sup>2</sup> at 140 °F (60 °C) or at 300 A/ft<sup>2</sup> at 158 °F (70 °C).

The internal stress in deposits from sulfamate solutions is influenced by reactions at the nickel anode material. When a nickel anode dissolves at relatively high potentials, a stress-reducing compound is produced by anodic oxidation of the sulfamate anion.

The use of pure nickel in the conditioning tank and active nickel in the main tank is designed to control the nature and amount of the stress-reducer formed in this high-speed bath. Although the process was developed many years ago, there appears to be renewed interest because of the need to obtain zero stress in certain critical applications.

## "Electroplating & Related Processes"

by: J. B. Mohler ..... \$45.00

This is a good introductory volume to the industry. The explanations are clear and the material is uncluttered with verbiage or meaningless illustrations. The reader wastes no time while preparing the groundwork for further study.

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### METAL FINISHING

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Nickel electroforming is the first and oldest practical application of nickel plating and it remains an important industrial activity about 150 years after it was conceived.

## NICKEL PLATING QUALITY CONTROL

Quality control is an important consideration whether the nickel plating process is used for decorative, engineering or electroforming purposes. Controlling quality involves maintaining the purity of the plating solutions and the properties of the deposits.

### Purification of Solutions:

Nickel plating baths freshly prepared from technical salts contain a number of organic and inorganic impurities that must be removed before the bath is operated. Older baths gradually become contaminated from drag-over from preceding treatments, from components that are allowed to fall off the rack and are allowed to remain in the tank, from corrosion products from auxiliary equipment, from tools dropped into the tank and from other sources.

It is probably more effective to wage a continuous campaign to keep impurities out of the plating bath than to deal with rejects and production interruptions resulting from the use of impure solutions.

The maximum concentrations of impurities normally permissible in nickel plating solutions and recommended treatments for their removal are shown in Table VIII.

The electrolytic treatment referred to in the table involves placing a large corrugated cathode in the solution and plating at low current densities, 2 and 5 A/ft<sup>2</sup>. Copper, lead and certain sulfur-bearing organic addition agents are best removed at 2 A/ft<sup>2</sup>, whereas iron and zinc are more effectively removed at 5 A/ft<sup>2</sup>.

A corrugated cathode is preferred because it gives a wider current density range. At 2 A/ft<sup>2</sup> impurities should be removed after the solution has been operated for 2 ampere hours per gallon; at 5 A/ft<sup>2</sup>, 5 ampere-hours per gallon should be sufficient.

Table VIII. Maximum Permissible Concentration of Impurities

Iron	0.15	g/L
Copper	0.04	"
Zinc	0.05	"
Lead	0.002	"
Aluminum	0.06	"
Hexavalent chromium	0.01	"

### Purification Treatments:

Impurity	Treatment (See Text)
Iron	High pH or electrolysis
Copper	High pH or electrolysis
Zinc	High pH or electrolysis
Lead	Electrolysis
Chromium	High pH. It may be necessary to precede this with a potassium permanganate-lead carbonate treatment, followed by lead removal.
Aluminum	High pH
Organic Impurities	Activated carbon, and activated carbon plus electrolysis.

The high pH treatment requires transferring the nickel solution to an auxiliary treatment tank. Sufficient nickel carbonate is added to bring the pH above 5.2. About 0.5 to 1.0 ml/L of 30% hydrogen peroxide is added.

The bath is agitated and kept warm for two hours. The pH is adjusted to the optimum level after the bath is filtered back into the main plating tank. The solution may then be electrolyzed at low current density until deposit quality is acceptable.

When organic impurities are to be removed, activated carbon is added prior to the high pH treatment described above. About 0.13 to 0.4 oz/gal (1 to 3 g/L) of activated carbon is commonly added to the solution in the auxiliary treatment tank.

The nickel carbonate and hydrogen peroxide are then added, and the solution is filtered. Electrolytic purification is often desirable at this point.

After a new bath has been prepared, the high pH treatment, treatment with activated carbon and electrolysis at low current densities are performed sequentially until the quality of the deposit as determined by the tests discussed in the next section is acceptable.

### Controlling the Properties of Nickel Deposits:

Methods that measure thickness, adhesion and corrosion resistance of nickel coatings are available for quality control. Properties such as porosity, ductility, tensile strength, internal stress, hardness and wear resistance are important and are measured to control the quality of electroplated articles. These properties may be measured by the following methods.

#### Thickness:

Micrometer readings are often used to determine the thickness of a coating at a particular point when the deposit thickness exceeds 125  $\mu\text{m}$  (0.005").

More intricate and accurate methods for determining the thicknesses of electrodeposited coatings can be found in ASTM standards. ASTM standard B 487 describes a method based on metallographic examination of cross-sections of the plated object. Alternate tests involve magnetic (ASTM B 530) and coulometric (ASTM B 504) measurements of thickness.

#### Corrosion and Porosity Tests:

Examination of the coated part after immersion in hot water for two to five hours for rust is one technique used in studying the corrosion resistance of plated steel. The number of rust spots in a given area is then used as the qualification for accepting or rejecting the piece.

Modifications of this test include immersion for up to five hours in distilled water, in distilled water saturated with carbon dioxide, or in distilled water containing 0.5% wt sodium chloride, at test temperatures of 82 to 85 °C (180 to 185 °F).

Several salt spray tests have been used to simulate marine environments. These tests are commonly used to evaluate nickel and nickel-chromium coatings on ferrous and nonferrous substrates.

The salt spray tests are also used as accelerated quality control tests and are described in the following ASTM standards:

1. Salt spray (ASTM B 117).
2. Acetic acid salt spray (ASTM B 287).
3. Copper accelerated acetic acid salt spray (CASS Test: ASTM B 368).

The ferroxyl test is another porosity test which is employed for coatings on ferrous metal substrates and involves the formation of Prussian blue color within exposed pits.

The solution utilizes sodium chloride and potassium ferricyanide as reagents to develop the color.

The only truly satisfactory method of establishing the relative performance of various coating systems is by service testing. Care should, therefore, be exercised in interpreting the results of accelerated corrosion tests.

Once an acceptable service life has been determined for a specific thickness and type of coating, the performance of other candidate coatings may be compared in accelerated corrosion tests to the performance obtained with the coating for which an acceptable service life has been established.

#### Hardness:

Hardness measurements involve making an indentation on the surface (or cross-section for thin coatings) of the deposit. The indenter has a specified geometry and is applied with a specified load.

In the case of industrial nickel coatings, the most common hardness determination is the Vickers method of forcing a diamond point into the surface under a predetermined load (normally 100 grams). This provides a measure of that surface to permanent deformation under load.

The figure obtained is not necessarily related to the frictional properties of the material nor to its resistance to wear or abrasion.

#### Tensile Strength:

The measurement of the tensile strength of an electrodeposited coating is performed after the coating is separated from the basis metal. The coating is then pulled in a tensile machine. In general, plating plant operators seldom perform this test.

#### Internal Stress:

The magnitude of internal stress obtained in deposits is determined by plating onto one side of a thin strip of basis metal and measuring the force causing the strip to bend.

One method used in commercial practice involves plating the exterior surface of helically wound strip and measuring the resultant change of curvature. Another method is based on the flexure of a thin metal disc.

#### Ductility:

Most of the tests that have been used for evaluating the ductility of plated coatings are rather qualitative in nature. Two bend tests are described in ASTM B 489 and B 490. Both of these procedures require a minimum amount of equipment.

Another method for measuring the ductility of thick deposits is to determine the elongation of a specimen in a tensile testing machine. This method is limited to relatively thick foils of controlled geometry and thickness. A method specifically designed for plated thin foils has been used and is known as the hydraulic bulge test.

#### Adhesion:

The adhesion of a coating to the basis material is important. In general, the adhesion between a nickel coating and the basis material exceeds the tensile strength of the weaker material.

As a result, when a force is applied to a test specimen which tends to pull the coating away from the basis metal, separation occurs within one of the two metals rather than at the boundary between the basis metal and the nickel coating.

**Table IX. ASTM Recommended Practices for Preparing Metals for Plating**

Basis Metal	ASTM Designation	Conditioning Step 1		Conditioning Step 2 (if needed)		Final Rinse Before Plating
		Solution	Operation	Solution	Operation	
Aluminum alloys	B 253	Alkaline zincate	Immerse long enough to deposit 0.02 to 0.05 mg Zn/cm <sup>2</sup>	Rochelle type copper strike. pH 10.2 to 10.5	Deposit Cu at 258 A/m <sup>2</sup> (24 A/ft <sup>2</sup> ) for 2 min then 129 A/m <sup>2</sup> (12 A/ft <sup>2</sup> ) for 4 min	Double rinse or spray
Copper alloys	B 281	Sulfuric or hydrochloric acid soln.	Acid dip			Single rinse or spray
Iron castings	B 320	Sulfuric or hydrochloric acid soln. Room temp.	Brief dip Cold water rinse	Anodic clean	Anodic at 646 to 1076 A/m <sup>2</sup> (60 to 100 A/ft <sup>2</sup> ) to remove smut	Rinse, acid dip, then cold water rinse
Lead alloys	B 319	Fluoborate dip 10% HBF <sub>4</sub>	Dip 10 to 15 seconds			Cold water rinse and spray
Nickel	B 343	Acid nickel chloride soln. 240 g/L (32 oz/gal) plus HCl 94 ml/L (12 fl oz/gal)	323 A/m <sup>2</sup> (30 A/ft <sup>2</sup> ) anodic 2 min cathodic 6 min			None
Stainless steels	B 254	65% H <sub>2</sub> SO <sub>4</sub>	Cathodic 2 min	Acid nickel chloride 240 g/L (32 oz/gal) plus HCl 94 ml/L (12 fl oz/gal)	Cathodic 2 min at 1614 A/m <sup>2</sup> (150 A/ft <sup>2</sup> )	None
Steels, low carbon	B 183	Alkaline anodic clean	Anodic at 6 volts for 1 to 2 min. Cold water rinse	4 to 10 vol % H <sub>2</sub> SO <sub>4</sub>	Dip at room temp. for 5 to 15 sec.	Cold water rinse
Steels, high carbon	B 242	Smut removal sodium cyanide soln.	Dip or short anodic treatment. Rinse	H <sub>2</sub> SO <sub>4</sub> 250 to 1000 g/L (34 to 134 oz/gal) plus 125 g/L (17 oz/gal) Na <sub>2</sub> SO <sub>4</sub> . Keep below 25 °C (77 °F)	Anodic at 1076 to 4304 A/m <sup>2</sup> (100 to 400 A/ft <sup>2</sup> ) for not more than 60 sec.	Fast rinse
Zinc alloys	B 252	Copper strike soln. Rochelle type	Deposit 1.3 μm (50 millionths inch) Cu	Cyanide copper plating bath	Copper plate 5 μm (0.2 mil) thick	Cold water

A number of qualitative tests have been used which utilize various forces applied in a multitude of directions with regard to the composite basis metal and coating, such as hammering, filing, grinding and deforming.

#### PREPARATION OF BASIS METALS FOR PLATING

Nickel plating requires a sound bond between the substrate and the coating. The coating should adhere tightly to the basis material. A sound metallurgical bond may be achieved on most materials.

The selection of grinding, polishing, pickling and conditioning treatments for a variety of basis metals varies from one material to another and depends on the initial surface condition of the metal.

The activating treatments which follow polishing and cleaning operations are listed in Table IX for the basis metals most commonly plated. The ASTM standards referred to provide additional information.

Nonconductive plastics and other materials can be plated by metalizing the material, using etching and catalyzing techniques. The mechanism of adhesion between metal and plastic is not fully known. A mechanical "locking" or "keying" action may take place at the plastic to metal interface and thereby provide for a moderately adherent deposit.

#### NICKEL ANODE MATERIALS

Significant developments in nickel anode materials and their utilization have taken place. Of utmost significance was the introduction of titanium anode baskets in the 1960s. Today the use of expanded or perforated titanium anode baskets filled with nickel of a selected size has become the preferred method of nickel plating.

Titanium anode baskets are preferred because they offer the plater a number of advantages. Primary forms of nickel can be used, which provide the least costly nickel ion source. Anode replenishment is simple and can be automated. The constant anode area achieved by keeping baskets filled improves current distribution and conserves nickel.

Several forms of primary nickel are currently being used in baskets. These include electrolytic nickel squares, rectangles, or rounds which contain a small controlled amount of sulfur. Nickel pellet produced by the carbonyl process and pellets containing a controlled amount of sulfur are also in use.

Prior to the introduction of titanium anode baskets, wrought and cast nickel anode materials were widely used. These are still used, but not to the extent they were before 1960.

The wrought and cast anode materials comprise rolled bars containing about 0.15% oxygen; rolled nickel containing about 0.20% carbon and 0.25% silicon; and cast bars containing about 0.25% carbon and 0.25% silicon. Soluble auxiliary anodes are generally carbon and silicon-bearing small-diameter rod.

With the exception of the sulfur-bearing materials, nickel anodes require the presence of chloride ion in the plating bath to dissolve efficiently. Rolled or cast carbon-bearing materials are used up to about pH 4.5. Oxygen-bearing, rolled depolarized anode bars can be used above pH 4.5.