

# **Electroless Plating: Fundamentals And Applications**

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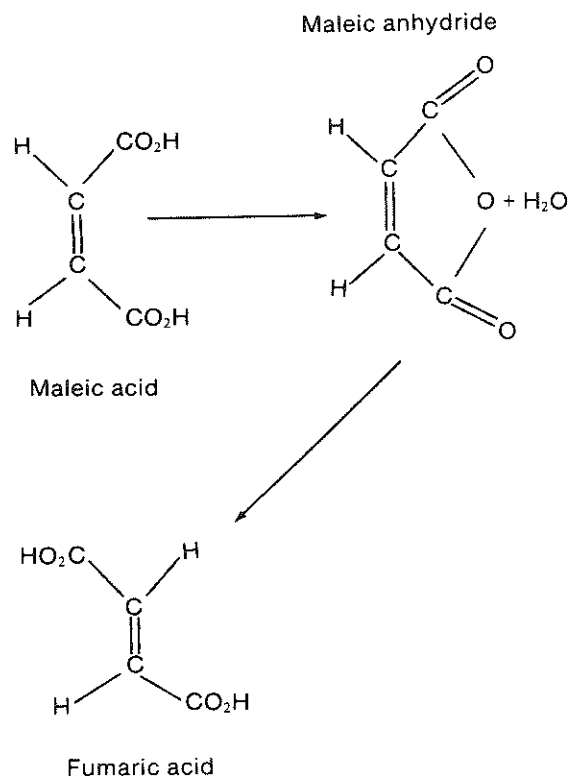


Fig. 1.19—Isomeric conversion of maleic acid to fumaric acid.

Although the above discussion on stabilizers was concerned with hypophosphite-reduced electroless nickel plating solutions, the principles also apply to amine borane-reduced baths. Stabilizers such as lead, and divalent sulfur compounds such as thiodiglycolic acid are commonly found in amine borane plating solutions. When a borohydride reducing agent is employed, the situation is different, however. As a result of the combination of high pH (>12.0) and high temperature (90° C), some of the stabilizers used in the former cases are not applicable because of their decomposition or precipitation. In borohydride baths, thallium salts at concentrations in the range of 40 to 50 ppm have proven to be extremely effective (16).

### Energy

Catalytic reactions, such as electroless nickel plating, require energy in order to proceed. The energy is supplied in the form of heat. Temperature is a measure of the energy (heat) content of the plating solution. In the context of this chapter, energy, which is added to the plating bath, is considered a bath variable like the other reactants. The quantity of energy required by the system or added to it is one of the most important factors affecting the kinetics and rate of the deposition reaction. The dependence of reaction rate on temperature is illustrated in Fig. 1.20. The rate-energy relation is shown schematically because the exponential nature of the relation is common to all electroless nickel plating systems, even though their absolute plating rates will differ.

The rate of a chemical reaction may be studied by measuring the rate of decrease in the concentration of the reactants or the rate of increase in the concentration of the products. In electroless nickel deposition, we measure the rate of formation of the deposit. These relationships are expressed mathematically by:

$$\text{Rate} = \pm \frac{dC_i}{dt} = K (\pi C_j^i) \quad [96]$$

where (+) is used if  $C_i$  is a product and (-) if  $C_i$  is a reactant;  $K$  is a numerical proportionality constant called the rate constant;  $C_j^i$  is the concentration of the  $i$ th reactant (product) present in the system at time  $t$ ; and  $j$  is the reaction order of the  $i$ th species.

To reiterate, the dominant variable affecting reaction rate is the temperature at which the process occurs. Many reactions that take place close to room temperature double their rates of reaction for each 10° C rise in temperature. Note that 100° C is considered close to room temperature.

The variation of rate constant  $K$  (in Eq. 96) with temperature  $T$  is given by the Arrhenius equation:

$$\frac{d}{dT} \ln K = \frac{E_a}{RT^2} \quad [97]$$

The quantity  $E_a$  is the activation energy of the reaction, and  $R$  is the gas constant. Since it is assumed that  $E_a$  is sometimes independent of temperature,  $T$ , integration of Eq. 97 yields:

$$\ln K = - \frac{E_a}{RT} + I_{nA} \quad [98]$$

where  $I_{nA}$  is the constant of integration.

Equation 98 is of the form  $y = mx + b$ , which is an equation for a straight line. Hence, a plot of  $\ln K$  vs. the reciprocal of the absolute temperature,  $1/T$ , should be a straight line whose slope is  $-E_a/R$ .

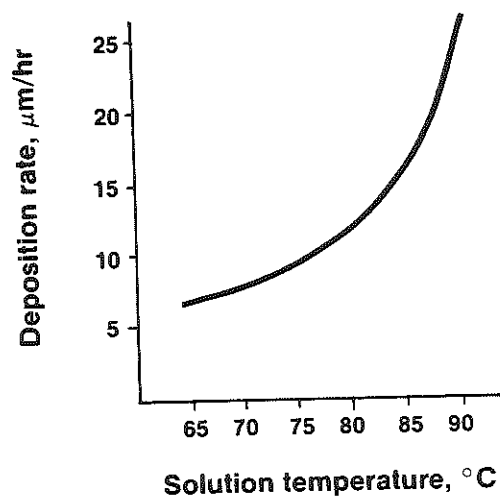


Fig. 1.20—Effect of temperature on plating rate.

Equation 98 yields an expression for the rate constant  $K$ :

$$K = A \exp\left(-\frac{E_a}{RT}\right) \quad [99]$$

Here  $A$  is called the *pre-exponential* or *frequency* factor. According to this equation, the reacting molecules or ions must acquire a certain critical energy  $E_a$  before they can react. The term  $\exp(-E_a/RT)$  is the Boltzmann factor, which denotes the fraction of the reacting species that have managed to attain activation energy  $E_a$ . The activation energy can be considered as a potential energy barrier that the reactants must climb before they can react.

Substituting the expression for  $K$  (Eq. 98), Eq. 96 can now be written as:

$$\text{Rate} = \pm \frac{dC_i}{dt} = A \left( \prod_{i,j} C_j \right) \exp\left(-\frac{E_a}{RT}\right) \quad [100]$$

Equation 100 shows that the reaction rate is dependent on the addition of energy to the system as measured by temperature.

When operating an electroless nickel plating bath, it is necessary to know which conditions yield deposits with the desired properties. The deposits should be produced with minimum cost and difficulty and optimum efficiency. In some cases, it is more efficacious to operate the plating bath at a temperature that does not give the maximum plating rate. The composition of Ni-P deposits can be altered slightly by merely raising (decrease P) or lowering (increase P) the temperature of hypophosphite-reduced nickel plating solutions. The amine borane reducing agents are more temperature sensitive and will hydrolyze excessively at high temperatures, causing wasteful side reactions.

In this chapter we have discussed, in some detail, the chemistry of the principal components of electroless nickel plating solutions. Each component was shown to have a unique effect on the course of the deposition reaction. The discourse was mainly concerned with the theoretical, or better, ideal aspects of the nickel reduction reaction.

However, in real time, EN solutions are used to plate a myriad of commercial and industrial substrates. In subsequent chapters, the real time operation of EN plating solutions will be discussed.

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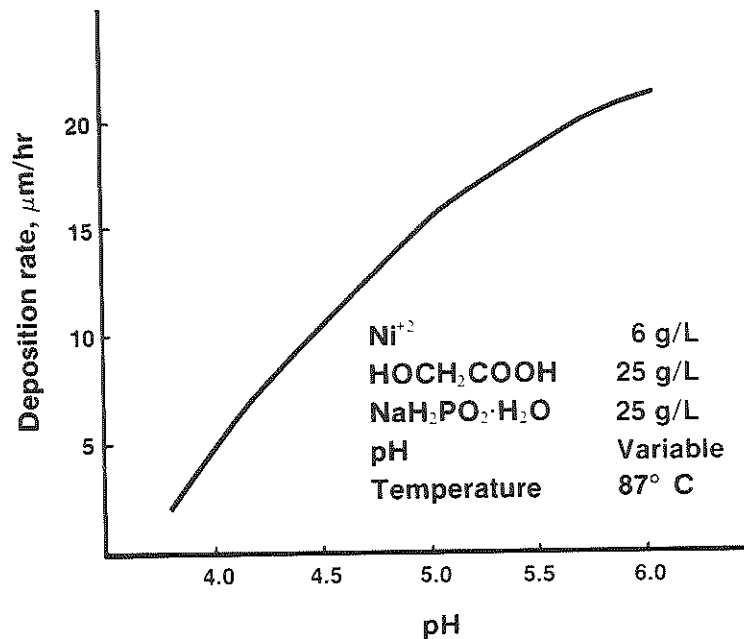


Fig. 2.1—Effect of pH on rate of deposition.

Equation 1 shows that there are at least five variables (components) that influence the deposition reaction: the reactants  $\text{Ni}^{+2}$  and  $\text{H}_2\text{PO}_2^-$ ; the products  $\text{H}^+$ ,  $\text{H}_2\text{PO}_3^-$ , and  $\text{L}^-$ . To these five variables, at least three more are added: temperature, the anion of the nickel salt used (e.g., sulfate from  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ ), and one or more stabilizers. Hence, there are at least eight variables that must be monitored during electroless nickel plating.

The number of variables illustrates the complexity of operating and maintaining these solutions. As an aside, it should be pointed out that the suppliers of proprietary electroless nickel plating solutions have reduced the number of variables the plater must be concerned with through judicious formulations and clever packaging of replenishing solutions.

#### The Influence of $\text{H}^+$

In laboratory tests, it is observed that for every mole of  $\text{Ni}^{+2}$  deposited, three moles of  $\text{H}^+$  are generated, which is in agreement with Eq. 1. The accumulation of hydrogen ions ( $\text{H}^+$ ) in the plating bath lowers the pH of the solution. When the pH decreases, the most noticeable change in the plating process is a concurrent decrease in the rate of deposition. If the pH is allowed to drop too far in acid solutions (pH < 4.0 for commercial plating baths), a very low plating rate is observed. Figure 2.1 illustrates the effect of solution pH on deposition rate. The

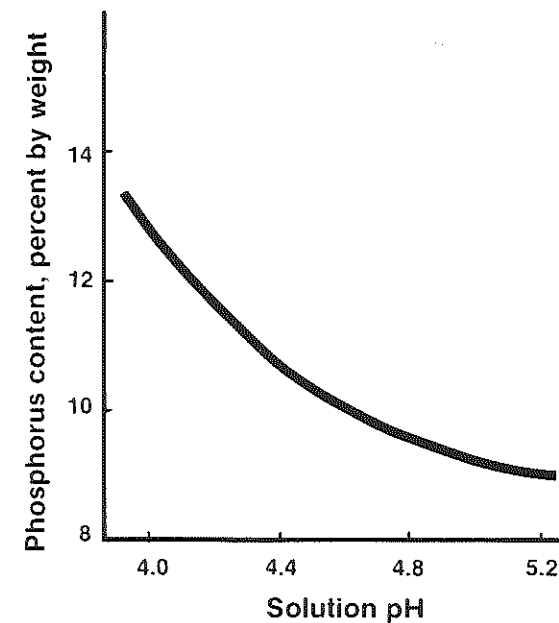


Fig. 2.2—Effect of pH on phosphorus content.

most dramatic effect of lowering the pH is on the composition, and concomitantly, the properties of the Ni-P deposit. The effect of solution pH on alloy composition is shown in Fig. 2.2.

The physical and chemical properties of electroless Ni-P deposits are dependent on the composition of the alloy in question. (A detailed discussion of the properties of electroless nickel-phosphorus deposits is covered elsewhere in this book.) As a rule of thumb, when the phosphorus content of the deposit is in excess of 10 percent by weight, the Ni-P alloy has the following characteristics:

- Low internal intrinsic stress, usually near zero or slightly compressive.
- Good corrosion resistance; low porosity.
- Non-magnetic in as-plated state.

If one or more of these properties is required, then a decrease in plating bath pH is not harmful, since the phosphorus content will be increased, allowing the deposit to achieve and maintain the desired property. On the other hand, if a particular application requires that the phosphorus content remain invariant or within a narrow range, say between 5.5 and 6.0 percent by weight, then a moderate decrease in pH can result in increasing the phosphorus content beyond the specified limit.

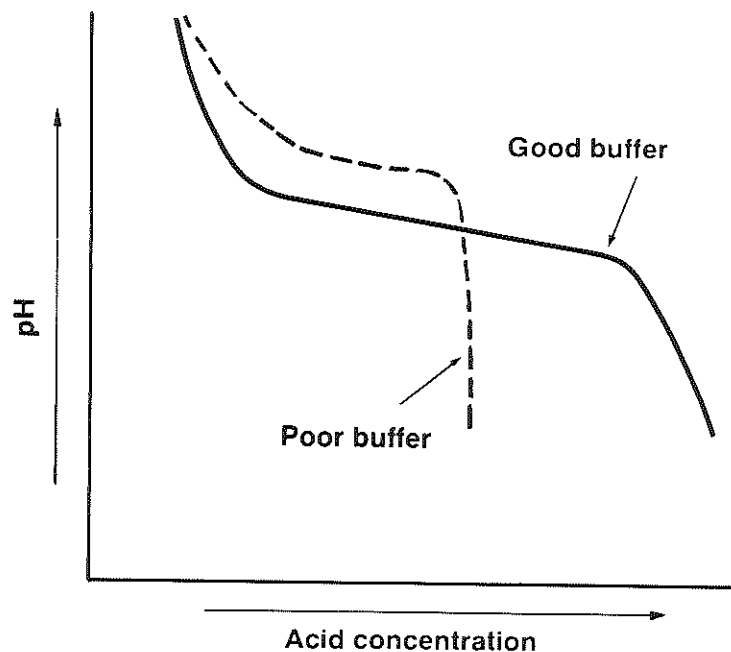


Fig. 2.3—Effect of buffering agents.

The amount that the pH changes as a result of the formation of  $H^+$  is related to the buffer capacity of the complexing agents and certain other materials called *buffers* that are present in the plating bath. In simple terms, a buffer is a substance or mixture of substances that, added to a solution, is capable of neutralizing both acids and bases without appreciably changing the original pH of the solution. A measure of buffer capacity is the amount of acid ( $H^+$ ) required to change the pH by a given amount. The greater the amount of acid required, the better the buffer. The practical buffer capacity,  $P_B$ , of an electroless nickel plating bath can be determined by titrating the bath with a standard acid solution and plotting pH vs. acid concentration. Figure 2.3 is a schematic illustration of data obtained by this technique. The slope of the linear portion of the curve can be expressed mathematically by:

$$|\alpha| = \frac{\Delta pH}{\Delta [H^+]}$$

Table 2.1  
Organic Acids Used as Buffers  
In Electroless Nickel Plating Solutions

Acid	Structure	Dissociation constants at 25° C	
		$K_1$	$K_2$
Acetic	$CH_3CO_2H$	$1.75 \times 10^{-5}$	—
Propionic	$CH_3CH_2CO_2H$	$1.4 \times 10^{-5}$	—
Succinic	$HO_2C(CH_2)_2CO_2H$	$6.6 \times 10^{-5}$	$2.5 \times 10^{-6}$
Glutaric	$HO_2C(CH_2)_3CO_2H$	$4.7 \times 10^{-5}$	$2.9 \times 10^{-6}$
Adipic	$HO_2C(CH_2)_4CO_2H$	$3.7 \times 10^{-5}$	$2.4 \times 10^{-6}$

where  $\Delta pH = pH_{initial} - pH_{final}$  and  $\Delta [H^+] = [H^+]_{initial} - [H^+]_{final}$ .  $P_B$  can then be defined as  $1/\alpha$ . The larger  $P_B$  is, the greater the buffer capacity of the plating bath. This method can be used to compare the buffer capacity of various combinations of complexing agents and buffers.

The most efficient buffers will not prevent the pH from eventually decreasing, therefore it is necessary to monitor the pH and neutralize the excess  $H^+$  being generated in the solution by the addition of ammonium or alkali metal hydroxides or carbonates. The time interval between additions of base solutions to an electroless nickel bath with a large  $P_B$  is less critical than for a bath with a much smaller  $P_B$ . A list of the commonly used buffers is given in Table 2.1. The aliphatic carboxylic acids also function as monodentate ligands (complexing agents); however, their principal action is that of buffer. Additionally, the anions of propionic, succinic, and glutaric acids are said to *exalt* the plating reaction. Exaltants are defined as those compounds that activate the hypophosphite anion and enhance the EN plating rate.

With few exceptions, it would appear that the ideal operating pH range for an acid Ni-P plating bath would be about 5.0 to 7.0. However, Gutzeit (1) points out that there are two important factors to be considered in the choice of the optimum pH range:

- The solubility of the orthophosphite ( $H_2PO_3^-$ ) produced by the reduction reaction.
- The adhesion of the coating, particularly on ferrous substrates.

The first factor will be discussed in a subsequent section of this chapter. Data obtained from comparative adhesion tests on steel (1) reveal that the adhesion of a specimen plated at pH 4.4 was 60,000 psi, compared to values of less than 30,000 psi for a test specimen plated at pH 6.0. The optimum pH range for acid hypophosphite EN plating baths is usually 4.5 to 5.2. In the majority of EN applications, satisfactory deposits will be obtained if the solution pH is

**Table 2.2**  
**Effect of pH Change on Electroless Nickel Process**

Change	Effect on solution	Effect on deposit
Raise pH	Increased deposition rate; lower phosphite solubility. Decreased stability with resultant plateout.	Decreased P content; shift in stress to tensile direction. Poorer adhesion on steel.
Lower pH	Decreased deposition rate; improved phosphite solubility.	Increased P content; shift in stress to compressive direction. Improved adhesion on steel.

maintained within the stated range. The effects of raising or lowering the pH on the process itself as well as the resultant deposits are discussed in Table 2.2.

The importance of maintaining an essentially constant solution pH during the plating sequence should now be readily apparent; therefore, in the discussions on the remaining bath variables, a constant pH should be inferred unless otherwise stated.

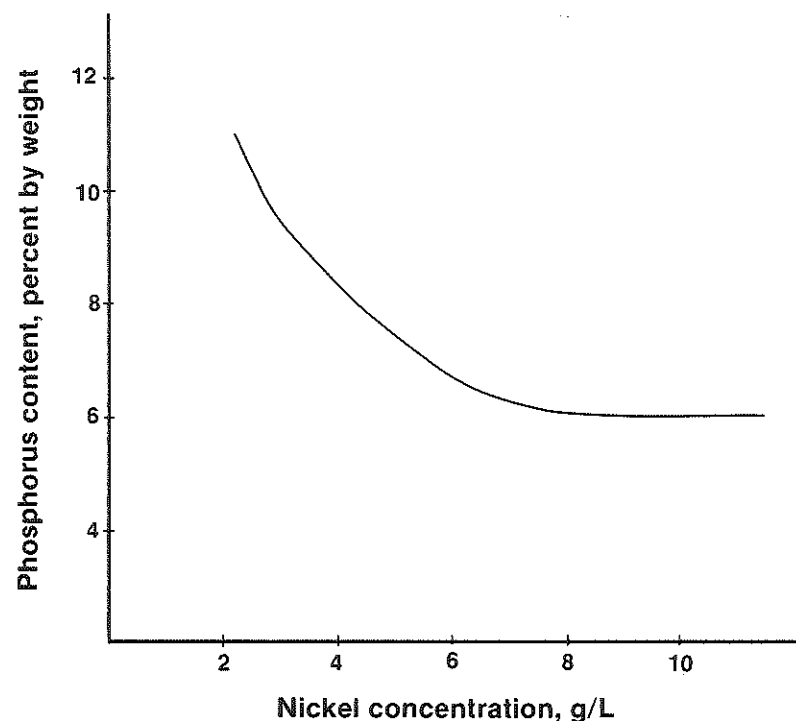
#### The Influence of Nickel and Hypophosphite Ion Concentration

Brenner and Riddell (2) discovered that autocatalytic nickel plating would proceed on an immersed catalytic substrate at temperatures near 90° C in the pH range of 4 to 6. Plating was shown to occur over a wide range of nickel and hypophosphite concentrations—3 to 100 g/L (0.05 to 1.7M) for nickel and 10 to 100 g/L (0.09 to 0.94M) for hypophosphite. Under these conditions, the authors found that nickel deposition would occur on a suitable substrate without the simultaneous random reduction of nickel throughout the plating solution.

Gutzeit and Krieg (3) observed that to achieve optimum plating conditions, a narrow, more limited concentration range for hypophosphite is required. They also found that the molar ratio of  $\text{Ni}^{2+}/\text{H}_2\text{PO}_2^-$  should be maintained within a limited range of 0.25 to 0.60, with the preferred range being 0.30 to 0.45.

The nickel concentration of commercial acid-type (pH 4 to 6) electroless nickel solutions lies within the range of 4.5 to 11 g/L (0.08 to 0.19M). Simple calculations based on the nickel concentration range and the preferred molar ratio of  $\text{Ni}^{2+}/\text{H}_2\text{PO}_2^-$  yields a molar concentration range of 0.18 to 0.27M for sodium hypophosphite.

The nickel concentration of EN solutions used for most industrial applications is usually  $6.5 \pm 1.0$  g/L (0.09 to 0.13M). When the nickel concentration is equal to or greater than approximately 5 g/L (0.085M), it has little or no effect on the plating rate (4); the plating reaction is said to be zero order with respect to the



**Fig. 2.4—Effect of nickel concentration on Ni-P alloy composition (4).**

nickel ion concentration. The phosphorus content of Ni-P deposits is influenced by the nickel concentration in the plating baths only when the nickel concentration is less than about 0.1M. If the nickel concentration is increased beyond 0.1M (5.8 g/L), the phosphorus content will remain invariant, provided that the hypophosphite concentration is held constant. Figure 2.4 shows the effect of the nickel concentration on Ni-P alloy composition.

Lee (5) reported the influence of the reactants on the phosphorus content of the Ni-P coatings. Generally, increasing the  $\text{H}_2\text{PO}_2^-$  concentration in the plating bath results in an increase in the phosphorus content of the deposit, as shown in Fig. 2.5.

Whereas nickel deposition is first order with respect to the hypophosphite concentration, Lee found a second order dependence on hypophosphite for phosphorus deposition. He derived the following empirical kinetic equation from experimental data:

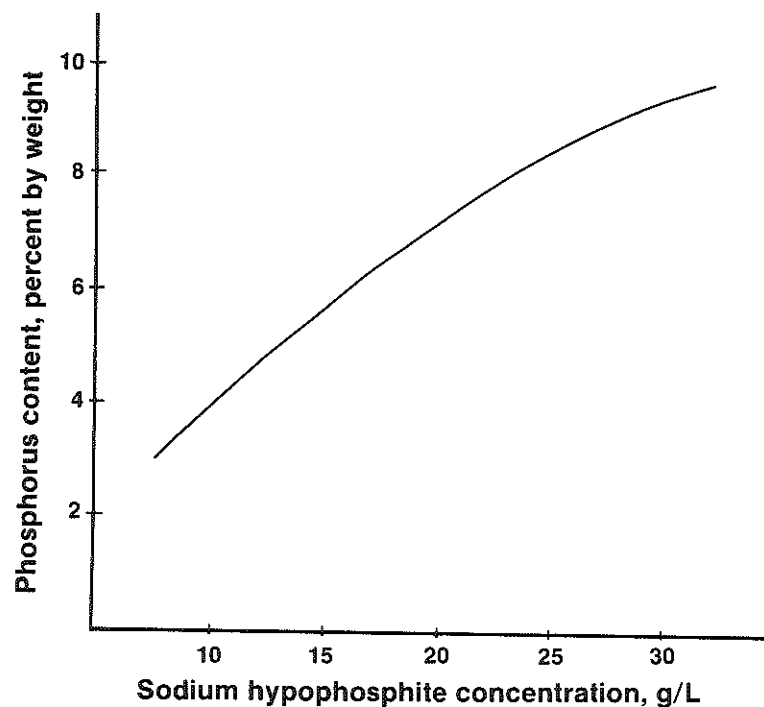


Fig. 2.5—Effect of hypophosphite concentration on Ni-P alloy composition (4).

$$\frac{dp}{dt} = K[\text{H}_2\text{PO}_2^-]^{1.91} [\text{H}^+]^{0.25} \quad [2]$$

Equation 2 indicates that phosphorus deposition is acid-catalyzed (increase phosphorus content by increasing  $\text{H}^+$ ). On the other hand, experimental data for nickel deposition leads to an empirical rate equation of the form:

$$\frac{d\text{Ni}^0}{dt} = K \frac{[\text{H}_2\text{PO}_2^-]}{[\text{H}^+]^\beta} \quad [3]$$

where  $\beta$  is the order of the reaction with respect to  $\text{H}^+$  concentration. Hence Eq. 3 shows that nickel reduction is base-catalyzed (increase nickel deposition by decreasing  $\text{H}^+$ ).

In addition to determining the effects of individual reactants on the electroless nickel plating process, it is equally instructive to ascertain the role their interdependence plays on the plating reaction. This is usually done by observing how the relationship expressed by the molar ratio  $\text{H}^+/\text{H}_2\text{PO}_2^-$  affects the plating

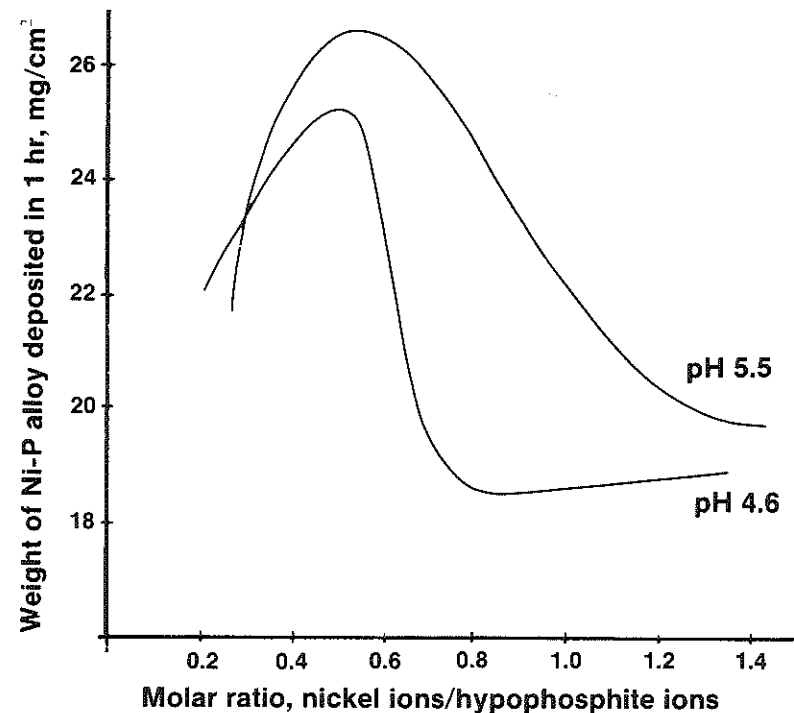


Fig. 2.6—Rate of deposition from acetate baths as a function of  $\text{Ni}^{2+}/\text{H}_2\text{PO}_2^-$  molar ratio at two different pH values (1).

rate. Figure 2.6, taken from Gutzeit (1), illustrates the effect of the molar ratio on the plating rate (constant  $\text{H}_2\text{PO}_2^-$ , varying  $\text{Ni}^{2+}$ ). The results given in Fig. 2.6 were obtained from an uncomplexed acetate bath, and it in no way infers that all electroless nickel solutions, especially those containing chelating agents, will yield equivalent results.

### The Phosphite Anion

The phosphite anion is generated by the oxidation of hypophosphite during the plating reaction (see Eq. 1). For each nickel ion reduced to metal, approximately three phosphite ions are produced; or, for each gram of  $\text{Ni}^{2+}$  reduced, approximately four grams of phosphite are formed. For continuous operation of the plating bath, the depleted hypophosphite (as well as nickel) must be replenished. Hence, phosphite steadily accumulates in the plating bath. With time, phosphite becomes an important solution component. As the phosphite concentration increases, it will begin to compete with the complexing agent for nickel ions. This usually occurs when the phosphite concentration reaches 30 g/L. Nickel phosphite is relatively insoluble and exhibits the inverse solubility

**Table 11.4**  
**Wear of Steel Pins in V Blocks<sup>a</sup>**

Coating on pin <sup>c</sup>	Coating on V block <sup>b</sup>	Pin wear, mg	Block wear, mg
EN	None	3.7	0.7
EN + WC	None	0.1	4.4
EN + TiC	None	0.4	2.3
EN + SiC	None	0.1	2.6
EN + B <sub>2</sub> C	None	0.2	0.7
None	EN	0.1	0.4
None	EN + SiC	0.3	0.7
None	EN + TiC	0.2	0.8
None	EN + B <sub>2</sub> C	0.8	0.3
None	EN + WC	2.0	0.3

<sup>a</sup>Data for wear with an applied load of 90 kg for 60 min followed by a load of 182 kg for 40 min, using a Falex lubricant tester.

<sup>b</sup>Coated steel pins and blocks were heat treated 16 hr at 288° C.

## FRICITION COEFFICIENT

It has been observed (13) that electroless composites can yield a lower friction coefficient than the same coating without particulate matter. Table 11.5 presents the results obtained for a friction coefficient utilizing an apparatus with a rotating steel ring contacting plated steel blocks (13). In recent years, the incorporation of PTFE into electroless nickel deposits attracted commercial interest, which then led investigators to an exploration of such properties.

The incorporation of PTFE into the electroless nickel composite serves several functions:

- Dry lubrication
- Improved wear resistance
- Improved release properties
- Repellency of contaminants such as water and oil

Most applications employ coating thicknesses of approximately 0.25 to 0.5 mil, with a preferred underlayer of electroless nickel. The presence of the underlying electroless nickel is believed to provide improved corrosion resistance. Typical electroless nickel-PTFE composite coatings incorporate PTFE in the range of 25 percent by volume, with focus on deposits having 18 to

**Table 11.5**  
**Friction Coefficients and Wear Data For Electroless Nickel Composites**

Coating on block	Coefficients of friction <sup>a</sup>				Block wear, mg	Ring wear, mg
	Static		Kinetic			
	initial	final	initial	final		
None	0.233	0.123	0.140	0.123	3.8	+0.3
EN	0.193	0.133	0.165	0.128	9.0	0.6
EN <sup>b</sup>	0.180	0.117	0.178	0.120	2.3	0.5
EN + B <sub>2</sub> C	0.177	0.142	0.113	0.103	2.0	0.8
EN + SiC	0.180	0.137	0.133	0.128	1.0	1.0
EN + WC	0.167	0.160	0.133	0.133	3.2	3.1
Teflon	0.213	0.140	0.160	0.113	2.8	0.9

<sup>a</sup>Friction coefficients calculated from friction forces measured on a friction and wear machine consisting of a steel ring rotating at 72 rpm on a line contacted against a coated block.

<sup>b</sup>Heat treated at 400° C for 1 hour.

25 vol. percent. Contrary to the inclusion of wear resistant particles (e.g., silicon carbide and diamond), electroless nickel-PTFE composite coatings appear to be limited to particles of 1 μm or smaller.

With the growing commercial interest in electroless nickel-PTFE composite coatings, various investigators have explored the wear properties of such composites utilizing accelerated testing procedures, which may or may not reflect actual field conditions.

Using a rotating ring apparatus, Tulsi (16) investigated the friction coefficients for electroless nickel and composites with PTFE. Table 11.6 summarizes these observations, which suggest that the lowest coefficient of friction is attained when both the pin and the ring are coated with an electroless nickel-PTFE composite coating. Ebdon (17) reported that an electroless nickel-PTFE composite exhibited a friction coefficient of 0.10 in contact with an SAE 300 series stainless steel and lasted much longer (8,000,000 cycles) on pneumatic cylinders than did hard anodized aluminum (10 to 33,000 cycles). After 3,000 cycles, the friction coefficient increased to 0.19; the friction coefficient of the hard anodized aluminum was 0.7.

So far, the reported (14,16-18) results for composites containing Teflon reveal that only particles in the range of 1 μm and less are incorporated, and that the plating rate is 8 to 9 μm/hr.



**Table 11.6**  
Friction Coefficient and Wear Data  
For Electroless Nickel-PTFE Composite

Coating on pin	Coating on ring	Coefficient of friction	Relative wear rate <sup>a</sup>
EN	Cr steel	0.6 to 0.7	35
EN + PTFE	Cr steel	0.2 to 0.3	40
EN + PTFE	EN + PTFE	0.1 to 0.2	1
EN + PTFE <sup>b</sup>	Cr steel	0.2 to 0.5	20
EN + PTFE <sup>b</sup>	EN + PTFE	0.1 to 0.7	2

<sup>a</sup>Wear determined in a test machine consisting of a pin and a rotating ring.

<sup>b</sup>Heated 4 hr at 400° C.

Table 11.7 (19) documents the friction coefficient for miscellaneous coatings, of which boron nitride appears to yield the lowest coefficient of friction, especially with increased loads employed in the friction machine. At this point, however, little if any commercial interest has been focused on boron nitride. Kim (20) describes the codeposition of carbon fluoride (CF<sub>x</sub>) along with electroless nickel for the purpose of attaining self-lubricating coatings to temperatures as high as 500° C. The success of the Kim process appears to rely upon the inclusion of non-ionic and cationic surfactants where the latter is at a significantly lower concentration. The presence of the cationic surfactant tends to increase the volume percent codeposited. Preferred non-ionic surfactants are those having an HLB value in the range of 10 to 20.

In pursuing the deposition of composites containing PTFE or carbon fluoride, filtration appears to be required in the removal of large agglomerates of particles larger than 50 μm. In addition, the number of metal turnovers is significantly less than that commercially obtained by conventional electroless nickel deposition and the inclusion of hard particles (e.g., silicon carbide and diamond).

## SURFACE FINISHING

The degree of surface roughness is dependent upon various parameters such as particle size, concentration of loading, coating thickness, particle size distribution, and smoothness of the starting substrate. Inclusion of particulate matter within the electroless metal matrices tends to increase the surface roughness, especially in the case of hard particles. Since it is important to consider the ultimate application of the plated article, surface roughness must be a factor. In certain circumstances, a surface finishing operation may be

**Table 11.7**  
Friction Coefficients  
For Miscellaneous Composites

Coating	Load, kg/cm <sup>2</sup>	Friction coefficient
TFE	0.1	0.12
Ni-P-BN	0.1	0.13
Ni-P-SiC	0.1	0.15
Ni-P	0.1	0.18
Cr	0.1	0.25
Ni-P-BN	0.3	0.09
TFE	0.3	0.13
Ni-P-SiC	0.3	0.14
Ni-P	0.3	0.16
Cr	0.3	0.40
Ni-P-BN	0.5	0.08
TFE	0.5	0.13
Ni-P-SiC	0.5	0.14
Ni-P	0.5	0.15
Cr	0.5	150.00

required to modify the as-plated roughness, thereby also controlling the frictional properties of the coating.

Table 11.8 shows the surface roughness of composite coatings containing polycrystalline diamond of different nominal sizes in a nickel-phosphorus matrix. It also shows the final roughness attainable with various newly-developed smoothing techniques.

Composite coatings, especially those containing wear resistant particles, are difficult to smooth. Newly developed methods afford a simplicity and economy of the smoothing operation not previously available. One recent development (21) relies upon the deposition of a second metallic layer that covers all exposed particulate matter, followed by the removal of a portion of the secondary layer, while still covering the exposed particles. In so doing, a smoothness level can be attained in a much shorter period of time (e.g., 30 sec vs 480 sec). Table 11.9 (21) documents the ease of smoothing with the application of the overcoat layer.

The use of an overcoat layer on top of the composite layer has been found beneficial in commercial applications. In the deposition of such a layer free of any particulate matter, smoothing of parts may be carried out more readily without expending significant energy and designing specialized tools for surface polishing hard-to-reach areas.

In another method, Spencer (22) discovered that the addition of comparatively small particles along with larger sizes of the same particulate matter provides a