

Electroless Nickel and the End-Of-Life Vehicle Directive

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In 2000, the European Union began banning lead, mercury, cadmium, and hexavalent chromium from automobiles with their End-Of-Life Vehicle (ELV) directive. Its purpose was to eliminate hazardous materials from vehicles to make them more easily recycled or recovered. The automotive industry has complied by including the four hazardous metals in their restricted substances standards. Electroless nickel coatings have long been used in the automotive industry because of their protective properties. However, until 2003, neither the automotive industry, nor their suppliers, were aware that most electroless nickel coatings contain amounts of lead and cadmium which exceed the limits set by the ELV directive. This paper will discuss this issue, as well as alternative technologies for lead and cadmium free coatings.

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Electroless nickel coatings have long been used in the automotive industry because of their protective properties. As applied, electroless nickel coatings are uniform, hard, lubricious, solderable, and corrosion resistant. They can be precipitation hardened to very high levels through the use of low temperature treatments, often producing wear resistance equal to that of commercial hard chromium coatings. This combination of properties, together with their economy and ease of application, makes the coating well suited for many severe applications and often allows it to be used in place of more expensive or less readily available alloys.

Properties of Electroless Nickel Coatings

Electroless nickel's structure makes it a unique metal finishing coating. In the as-deposited condition, electroless nickel coatings containing more than 11 percent phosphorus (high phosphorus coatings) are totally amorphous without any crystal structure. Coatings containing less than 4½ percent phosphorus (low phosphorus coatings) are completely crystalline. Between 4½ and 11 percent phosphorus (mid phosphorus coatings), coatings consist of a mixture of crystalline and amorphous phases. The presence of amorphous material gives these coatings many protective and useful properties. Typical properties of electroless nickel coatings are compared in Table 1 to those of electrolytic nickel and chromium.¹

The appearance of electroless nickel coatings is similar to that of metallic nickel, but with a slight yellow cast. The appearance of deposits can vary significantly, depending on the plating solution's formulation, the presence of brightening agents, and the surface finish of the substrate. Typically, low and high phosphorus coatings have a duller finish than the medium phosphorus deposits. Brightened medium phosphorus coatings can be very reflective and shiny.

Perhaps the most widely known property of electroless nickel coatings is their uniform thickness. Because they are applied by chemical reduction, without an external source of electrons, the plating rate and the coating's thickness are the same on any section of the part exposed to fresh plating solution. Grooves, slots, blind holes, and even the inside of tubing can have the same amount of coating as the outside of a part.

The adhesion of electroless nickel coatings to most metals is outstanding. Provided the part has been properly cleaned, an exchange reaction occurs when catalytic metals, like steel and aluminum, are placed into an electroless nickel bath. This reaction allows the coating to establish metallic as well as mechanical bonds with the substrate and to produce shear strengths as high as 400 MPa (60,000 psi).

Electroless nickel coatings have high strength. Except for low phosphorus deposits, the coatings' tensile strength exceeds 700 MPa (100,000 psi). This allows the coating to withstand a considerable amount of abuse without damage. However, because of the amorphous material

they contain, the ductility of electroless nickel coatings is low compared to most other materials. The elongation of most coatings is only about one percent. If a part is deformed or bent after plating, the coating may break.

Table 1
Typical Properties of Electroless Nickel, Nickel and Chromium Coatings

Property	Ni-3P	Ni-8P	Ni-11P	Nickel	Chromium
Composition	3-4%P, balance Ni	6-9%P, balance Ni	11-12%P, balance Ni	100% Ni	100% Cr
Structure	Micro crystalline	Mixed crystalline and amorphous	Amorphous	Crystalline	Crystalline
Internal stress	-10 MPa	+40 MPa	-20 MPa	+140 MPa	+200 MPa
Final Melting Point	1275°C	1000°C	880°C	1450°C	1890°C
Density	8.6 g/cm ³	8.1 g/cm ³	7.8 g/cm ³	8.9 g/cm ³	7.1 g/cm ³
Coefficient of Thermal Expansion	12.4 µm/m°C	13 µm/m°C	12 µm/m°C	14 µm/m°C	8.4 µm/m°C
Electrical Resistivity	30 µΩ-cm	75 µΩ-cm	100 µΩ-cm	8 µΩ-cm	40µΩ-cm
Thermal Conductivity	0.6 W/cm°K	0.05 W/cm°K	0.08 W/cm°K	0.95 W/cm°K	0.67 W/cm°K
Specific Heat	0.25 cal/g°K	ND	0.11 cal/g°K	0.11 cal/g°K	0.11 cal/g°K
Magnetic Coercivity	130 Oe	1.4 Oe	0	70 Oe	ND
Tensile Strength	300 MPa	900 MPa	800 MPa	400 MPa	120 MPa
Ductility	0.7 %	0.7 %	1.5 %	28 %	0.1 %
Modulus of Elasticity	130 GPa	110 GPa	170 GPa	180 GPa	120 GPa
Hardness, as deposited	700 HV ₁₀₀	600 HV ₁₀₀	530 HV ₁₀₀	170 HV ₁₀₀	1000 HV ₁₀₀
Hardness, heat treated	960 HV ₁₀₀	1000 HV ₁₀₀	1050 HV ₁₀₀	NA	NA
Coefficient of Friction, lubricated	ND	0.38	0.45	ND	0.43
Taber Wear Index, as deposited	11 mg/ 1000 cycles	16 mg/ 1000 cycles	19 mg/ 1000 cycles	25 mg/ 1000 cycles	2 mg/ 1000 cycles
Taber Wear Index, heat treated	9 mg/ 1000 cycles	12 mg/ 1000 cycles	12 mg/ 1000 cycles	NA	NA
Corrosion Protection, salt fog resistance	24 hours	96 hours	1000 hours	24 hours	<24 hours

ND = Not Determined NA = Not Applicable
Salt fog resistance is based on testing standard flat panels

Hardness and the resulting wear resistance of electroless nickel coatings are very important properties for many applications. Before heat treatment, the microhardness of medium and high phosphorus coatings is between 500 and 600 HV₁₀₀. Low phosphorus coatings can have hardness values as high as 650 to 700 HV₁₀₀. These are very approximately equal to 50 and 56 HRC respectively and are equal to the hardness of many alloy or tool steels.

Heat treatment will cause electroless nickel coatings to precipitation harden and can produce hardness values as high as 1100 HV₁₀₀. This is approximately equal to 72 HRC and similar to the hardness of commercial hard chromium coatings.

Because of their high hardness and natural lubricity, electroless nickel coatings have excellent resistance to both adhesive and abrasive wear. Wear resistance can be improved further by heat treatment. For many applications, the wear resistance of electroless nickel after heat treatment is equal to that of hard chromium deposits.

Electroless nickel coatings have excellent frictional characteristics. Their phosphorus content provides a natural lubricity, which helps to reduce friction, heat buildup and wear and to overcome galling. The coefficient of friction of electroless nickel is about the same as that of chromium, and a significant improvement over steel, stainless steel and aluminum. The frictional characteristics of a surface can be further improved by the use of composite coatings containing PTFE particles.

Electroless nickel coatings provide corrosion protection by forming a barrier between the corroding environment and the substrate. They are not sacrificial like cadmium or zinc deposits. However, because they are completely or partially amorphous, and free of grain boundaries and porosity, the corrosion protections provided by electroless nickel coatings is excellent and in most environments superior to that of other coatings. Generally, high phosphorus coatings provide the highest level of protection.

Applications of Electroless Nickel Coatings

Electroless nickel coatings have been used by the automotive industry since they were developed in the 1950's. In the 1990's, automotive applications were 15 percent of the total electroless nickel market in North America.² While there have been many different reasons for their use, the most common applications have been for wear or corrosion prevention. Typical applications of electroless nickel coatings in the automotive industry are listed in Table 2.^{3 4}

One long time automotive application of electroless nickel has been protecting the pinion shaft of differential assemblies from wear. The shaft holds a gear that could score it under high loads. The shaft is coated with 12 microns of medium phosphorus electroless nickel and then

heat treated to maximize the deposit's hardness. Over the years, other coatings (including hard chromium and salt bath surface hardening) have been tried with this part, but none have been found to be as successful as electroless nickel.

Another common application for avoiding wear is the cast iron slip yoke in the drive assembly. Without lubrication, the yoke and its mating spline would gall, resulting in noise and potential failure. An eight micron thick coating of medium phosphorus electroless nickel has used for fifteen years to prevent this problem.

Table 2
Typical Applications of Electroless Nickel Coatings

Application	Substrate	Composition	Thickness, μm	Reason for Use
Wheels	Aluminum	M, H	10	CR, U
Carburetor components	Zinc	L, M	15	CR
Fuel injectors	Steel	M, H	12	CR, WR
Windshield wiper ball studs	Steel	H	12	U, WR, CR
Differential pinion shafts	Steel	M	12 h	WR
Drive assembly slip yoke	Cast iron	M	8	WR
Disc brake pistons	Steel	M	13 h	WR, U, CR
Disc brake caliper bolts	Steel	M, H	12	CR, U
Disc brake bleed valves	Steel	H	8	CR, U
Hydraulic clutch sleeve liners	Steel	PT	10	L, U, WR
Transmission thrust washers	Steel	M, B	12 h	WR
Synchromesh gears	Brass	M, H	30	WR
Viscous coupling discs	Steel	M	12 h	WR
Lock and steering components	Powder metal	L, M	13	WR, L
Water pump components	Steel	PT	10	L, CR
Fuel pump electric motor parts	Steel	H	8	CR
Tubing and hose couplings	Steel	M, H	5	CR, U
Decorative assemblies	Plastic	L	0.3	M

Composition: H = 10 - 12%P; M = 6 - 9%P; L = 3 - 5%P; PT = PTFE composite; B = 3 - 5%B

Thickness: h = heat treated to maximize hardness and wear resistance

Reason for Use: CR = corrosion resistance; L = lubricity; M = metallization; S = solderability;
WR = wear resistance; U = uniformity

In Brazil, ethanol based fuels confirmed the superior corrosion resistance of electroless nickel coatings. If unprotected, zinc carburetor components and aluminum fuel injection systems would be rapidly attacked by the alcohol solutions. For more than fifteen years, these components have been protected against corrosion by electroless nickel coatings. Originally, zinc carburetors were plated with low phosphorus coatings from alkaline baths, and then with a copper strike followed by seven microns of a medium phosphorus deposit. Aluminum fuel systems were plated with

an alkaline electroless nickel strike with an over-coat of seven microns of a medium phosphorus coating. Medium phosphorus coatings are also used for steel fuel components.

Another common application for corrosion protection is with components that must be disassembled periodically for maintenance. These components include brake parts, couplings and connectors for air conditioning tubing, and hose nuts and connectors. These are typically plated with five to 7 microns of medium or high phosphorus electroless nickel. Without the coating, these components could seize from the rusting produced by atmospheric or road splash exposures.

Another large use for electroless nickel is the metallization layer under nickel and chromium on decorative plastics, such as grills, lamp bezels, and wheel covers. Electroless nickel replaced electroless copper for decorative plastics because it provides more consistent adhesion.

Electroless Nickel Plating Baths

Electroless nickel solutions are blends of different chemicals, each performing an important function to control the chemical reaction. Electroless nickel solutions typically contain the following:

- < A source of nickel ions,
- < A reducing agent,
- < Energy as heat,
- < Complexing agents,
- < Buffering agents,
- < Inhibitors or stabilizers, and
- < Brighteners.

The characteristics of an electroless nickel bath and its deposit are determined by the way these components are combined.

Nickel sulfate is usually used as the source of nickel ions in electroless nickel solutions. The reducing agent supplies electrons to plate nickel onto the substrate. Sodium hypophosphite is used as the reducing agent in most electroless nickel solutions and produce deposits that are nickel-phosphorus alloys.²

Because electroless nickel coatings are applied by chemical reduction, rather than by electroplating, their plating rate is relatively low. To increase their rate, the solutions are heated to as high a temperature as possible without causing them to decompose. The operating temperature of most baths is about 90°C (190°F).

Complexing agents are carboxylic acids like lactic, malic and glycine. They control the

amount of free nickel available for reaction, so that plating occurs only on the catalytic surface. Complexing agents also retard the precipitation of nickel phosphite. These organic acids also buffer the plating solution and help prevent its pH from decreasing too rapidly as hydrogen ions are produced. This also helps keep the plating rate high.

Electroless nickel plating must be controlled so that deposition occurs at a predictable rate and only on the substrate to be plated. To accomplish this, inhibitors or stabilizers are added to the bath. Without stabilizers, an electroless nickel plating solution would decompose after only a few hours of operation. Stabilizers function by absorbing on any colloidal particles present in the solution, preventing them from plating. Stabilizers also prevent plating on the tank and fixtures, reduce roughness and increase smoothness.

Lead was the first and the most commonly used stabilizer in electroless nickel baths. It became universally used in all types of baths because it was very effective. Today, lead is usually used as one of a combination of complimentary stabilizers. Other common stabilizers include sulfur compounds, oxy anions (such as iodates), and some unsaturated acids (such as maleic).

Electroless nickel coatings typically have a semi-bright or slightly matte appearance, like stainless steel. However, for some applications, a bright and shiny appearance, like decorative chromium, has been desired. Applicators often wrongfully equated brightness with quality. For these users, brightening agents were added to the electroless nickel bath. Two different classes of brighteners are usually used together. Cadmium, used with a sulfur compound like thiourea, acts as a leveling or smoothing agent. Cadmium also provides luster and color.

Cadmium also became universally used as a brightener because it also was very effective. One disadvantage of cadmium brightened coatings is that they tend to stain more after plating or from environmental exposure than do non brightened deposits. They also are less corrosion resistant.

End-of-Life Vehicle Directive

In 2000, the European Union began banning lead, mercury, cadmium, and hexavalent chromium from automobiles with their End-Of-Life Vehicle (ELV) directive.⁵ Their purpose was to eliminate hazardous materials from vehicles so as to make them more easily recycled or recovered. They estimated that between eight and nine million tonnes of waste were generated each year from automobiles in Europe.

The directive states “Member states shall ensure that materials and components of vehicles put on the market after 1 July 2003 do not contain lead, mercury, cadmium or hexavalent chromium....” In its Annex II, the directive exempted certain materials (like lead in batteries and free-machining metals) that could not be easily replaced with a non-hazardous equivalent.

Annex II was revised in 2002 to narrow some of the exemptions and to limit others.⁶ The automotive industry has complied with the directive by including the four hazardous metals in their restricted substances standards.^{7 8 9}

Neither the automotive industry, nor their vendors and applicators, were aware that most electroless nickel coatings contain significant amounts of lead and cadmium. While an electroless nickel plating solution may only contain one or two mg/L of lead as a stabilizer or cadmium as a brightener, these additives deposit at a faster rate than does nickel. As a result, they are concentrated in the deposit, and may reach levels as high as 0.1 percent by weight (1000 ppm) each.

The end-of-life vehicle directive does not provide an exemption for lead and cadmium in electroless nickel coatings. It does provide an exemption for trace contaminants up to 0.1 percent for lead and 0.01 percent for cadmium, “provided these substances are not intentionally introduced.” With electroless nickel solutions, of course, lead and sometimes cadmium are intentionally introduced. Accordingly, they are not allowed in electroless nickel coatings used for automobiles.

The automobile industry’s USCAR’s Vehicle Recycling Partnership became aware of this situation in late 2003. After discussions with suppliers of electroless nickel solutions, the Partnership established a goal to change their supply to lead free and cadmium free (LFCF) coatings. So far, General Motors has announced that lead and cadmium must be eliminated from the coatings on their vehicles within 18 months.¹⁰ Ford has also prohibited lead and cadmium from the coatings on their vehicles, but has not established a timetable for its elimination.¹¹ DaimlerChrysler has stated that all parts that they now receive must be lead and cadmium free.¹²

The electrical and electronic industries have established a similar program where they have banned lead, cadmium, mercury and hexavalent chromium, as well as a number of organic compounds, from the materials used for their parts.¹³ Similarly, the food industry has recently banned the intentional additions of lead to the materials used to manufacture equipment to handle or process foods.¹⁴ The European Union has also addressed hazardous materials in the electrical and electronic industries with their Restrictions on The Use of Certain Hazardous Substances (RoHS) directive.¹⁵ This directive will ban the use of materials containing lead, mercury, cadmium, hexavalent chromium, and polybrominated fire retardants after July 1, 2006. Accordingly, it has and will become more and more difficult for job shop platers to use conventional electroless nickel solutions containing lead and cadmium.

Lead Free, Cadmium Free Coatings

Lead and cadmium became almost universally used in electroless nickel baths because they were very effective as a stabilizer and brightener. Replacing them with more environmentally friendly

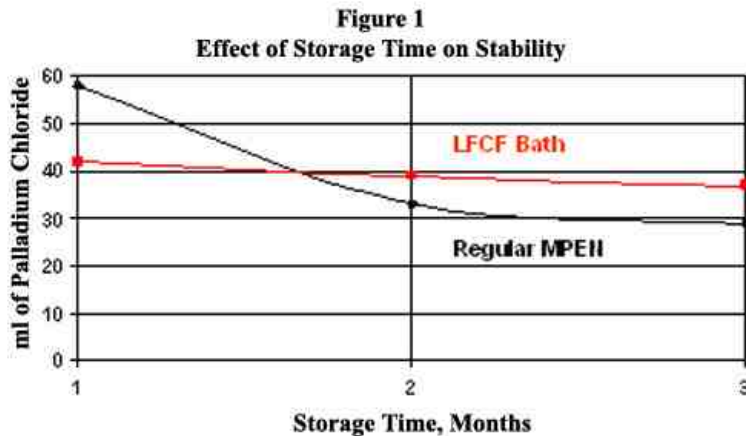
additives, must be done carefully. The replacement must produce coatings with equal protective properties, while not detrimentally affecting the baths' operation and performance. If the new bath is difficult to operate, is unstable, or slower than conventional solutions, it can reduce the applicator's productivity and he will be reluctant to use it. Of most concern is the stability of electroless nickel solutions without lead as a stabilizer. A secondary concern is the appearance of the coating without cadmium.

The appearance of electroless nickel coatings for automotive applications should not normally be an issue. Automotive applications are functional and not decorative. Most parts cannot even be seen. A bright appearance should not be required. However, there is the occasional exception, where a high degree of brightness is required in order to maintain smoothness and a low friction surface.¹⁶ For these applications, solutions with non-cadmium brighteners may be needed.

Many of the proprietary suppliers of electroless nickel solutions have been working on this problem since 2002 and appear to have solved most of these issues. They have been able to create non-lead based stabilizer packages that provide equal operational stability. Some suppliers have touted baths with non-cadmium brightener packages. In presentations to USCAR on May 10, 2004, the different proprietary electroless nickel suppliers summarized the status of their LFCF solutions.¹⁷ The properties of these new LFCF electroless nickel baths and their deposits are described in the following paragraphs and are based on these presentations.

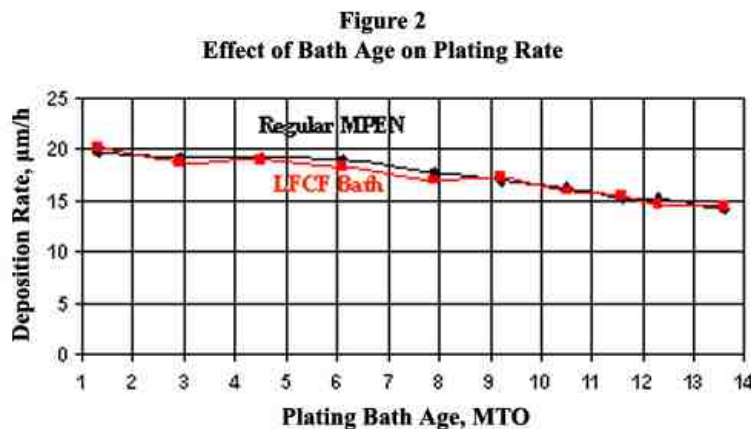
Stability. The stability of LFCF baths is equal to that of traditional formulations. Historically, the stability of electroless nickel baths has been measured by progressively adding small amounts of a dilute palladium chloride solution to the hot electroless nickel bath until it decomposed. With this test, it took 2.4 times as much of the palladium solution to decompose one LFCF medium phosphorus bath as it did for a conventional bath. With high phosphorus type baths, the stability of the LFCF and the regular bath was about the same.¹⁸

Another measure of stability is how long an electroless nickel solution can be boiled before it decomposes. In this test, the LFCF bath survived seven hours of boiling, while the regular mid phosphorus formulation decomposed after only four hours. The LFCF bath was almost twice as stable as the regular bath.¹⁹



One supplier reported the stability of their solution as a function of the time that it was stored in a drum. Their intent was to determine whether the new lead-free stabilizer package degraded or lost effectiveness over time. The results of these tests are shown in Figure 1.¹⁹ While initially, the lead free solution was less stable than the conventional bath, it maintained its stability better over time.

Bath Life. Another important characteristic of the plating bath is bath life. Not only must the bath perform properly when new, but also after it has been used. If the deposition rate declines or the coating's appearance changes, the bath might have to be replaced early, reducing the applicator's productivity. Fortunately, this has not been a problem. The new LFCF baths maintain their plating rate and their deposits' properties as well as and sometimes better than conventional formulations.



Plating Rate. An adequate plating rate is a critical characteristic of a bath. It determines the applicator's productivity. If the rate of a bath is low, the applicator will be reluctant to use it. Fortunately, the deposition rate of the LFCF baths is very similar to that of conventional

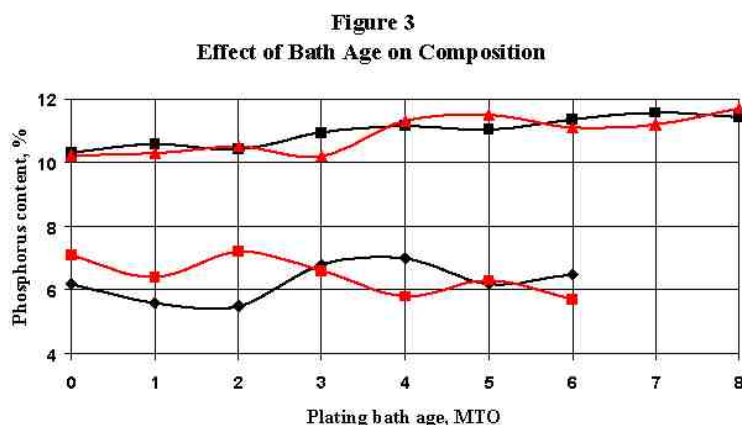
formulations as shown by Figure 2 below.²⁰

In this graph, the age or usage of the bath is shown in terms of a metal turnover (MTO), which is the deposition of the original nickel content of the bath. This is the traditional way that bath age is described. One MTO is also sometimes called a cycle or one hundred percent.

Bath Loading. Bath loading is the ratio of total exposed substrate surface area to the volume of the bath. With many baths, if the loading is too low, coating defects may result. With others if loading is too high, the bath may decompose. Bath loading results from the relative concentration of heavy metal and sulfur-based stabilizers on the parts' surface. Bath loading is mostly a concern with medium phosphorus baths, where loading usually must be maintained between 0.6 and 1.8 dm²/L (0.25 and 0.75 ft²/gal). High phosphorus baths do not contain sulfur compounds or large quantities of heavy metals and usually are not subject to bath loading limits.

Most suppliers reported that their new stabilizer packages allowed their medium phosphorus baths to be operated at lower bath loadings than had their previous formulations. One supplier reported a lower loading limit of 0.4 dm²/L (0.15 ft²/gal) without coating defects; another reported a limit of 0.02 dm²/L (0.01 ft²/gal).^{21 22 23} The upper loading limit of some baths was also shown to have increased. Two suppliers reported values of at least 1 ft²/gal.^{20 22}

Composition. Conventional medium phosphorus coatings typically contain 0.05 to 0.08 percent lead and 0.05 to 0.10 percent cadmium. Conventional high phosphorus coatings typically contain 0.02 to 0.04 percent lead and no cadmium. With the new LFCF baths, the lead and cadmium contents are not detectable by atomic absorption spectrophotometry or inductively coupled plasma spectrophotometry.^{18 19 23} With square wave cyclic voltammetry, one deposit was analyzed to contain less than 0.0001 percent lead and less than 0.00005 percent cadmium.²¹



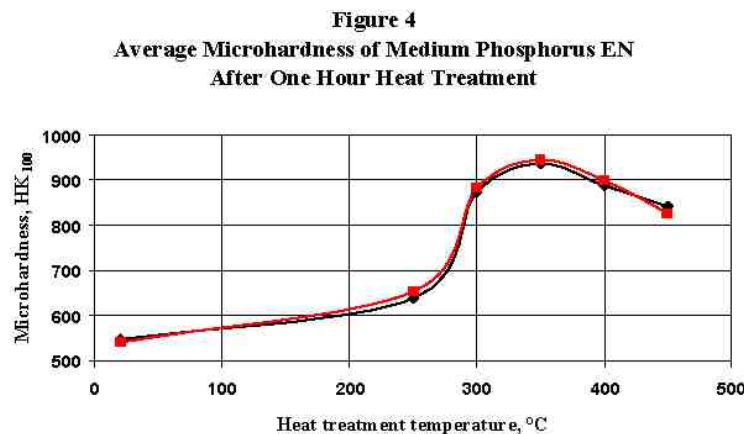
The phosphorus content of many electroless nickel coatings increase as their baths age. The coatings from LFCF baths behave the same way. Figure 3 shows the effect of bath age on the

phosphorus content of a conventional and LFCF version of both medium and high phosphorus electroless nickel coatings.¹⁸ There is no significant difference between the conventional and LFCF coatings.

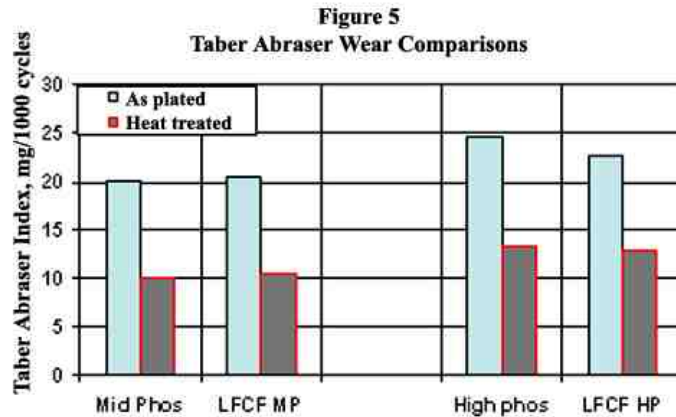
Appearance. Appearance is the one property with major differences between the coatings provided by different suppliers. A few suppliers have developed non-cadmium brighteners for their medium phosphorus baths, while others have decided that automotive and electronic applications are functional and that brighteners are not required. Whether a coating was brightened, however, did not seem to have any effect on the coating's other properties.

Hardness and Wear Resistance. Hardness and resulting wear resistance are the two most widely used properties of electroless nickel coatings for automotive applications. Tests have shown that the hardness of LFCF coatings is identical to those applied from conventional baths. This is confirmed by Figure 4, which compares the hardness of LFCF and conventional medium phosphorus coatings.²³

Similar tests showed that the microhardness of conventional and LFCF high phosphorus coatings also to be the same.²³

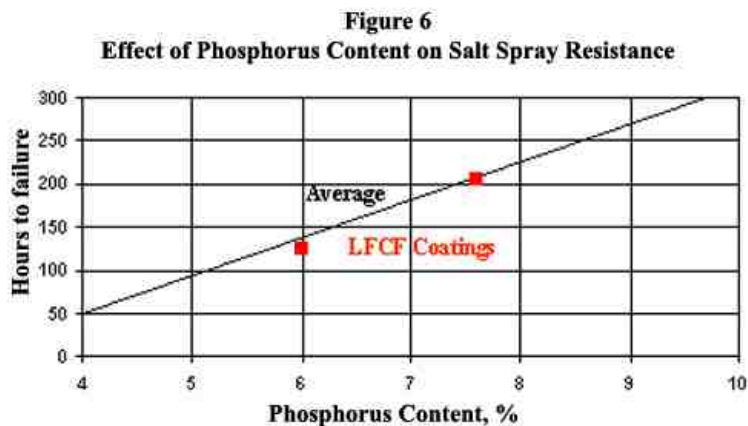


Similarly, the wear resistance of LFCF coatings has been found to be the same as that of coatings applied from conventional baths. Figure 5 compares the Taber Abraser Wear Index of both medium and high phosphorus electroless nickel coatings.²³



The hardness and wear resistance of LFCF and conventional coatings should be similar since the coatings' basic structure is unchanged. It is the relative amounts of crystalline and amorphous phases present that determines hardness and wear resistance.²⁴ These phases and structure are a function of the coating's phosphorus content. Additives like lead and cadmium have little effect on structure.

Corrosion Resistance. The phosphorus content and structure of electroless nickel coatings also establishes their corrosion resistance.²⁴ The straight line on Figure 6 is the average resistance to neutral salt fog for medium phosphorus coatings.²⁵ The two data points at six and 7.6 percent phosphorus are for LFCF coatings.²¹ Their corrosion resistance is equal to the previously used coatings.



Conclusion

Lead and cadmium can be eliminated from electroless nickel plating solutions without degrading the plating baths' operation, life, or performance. The resulting coatings from LFCF baths have equal resistive properties to those from previously used baths with lead and cadmium. Accordingly, there is no technical reason why LFCF coatings cannot be substituted for all of the electroless nickel coatings used by any industry.

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