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Tribological characteristics of electroless Ni–P alloys as an alternative to chromium coatings: infometric analysis

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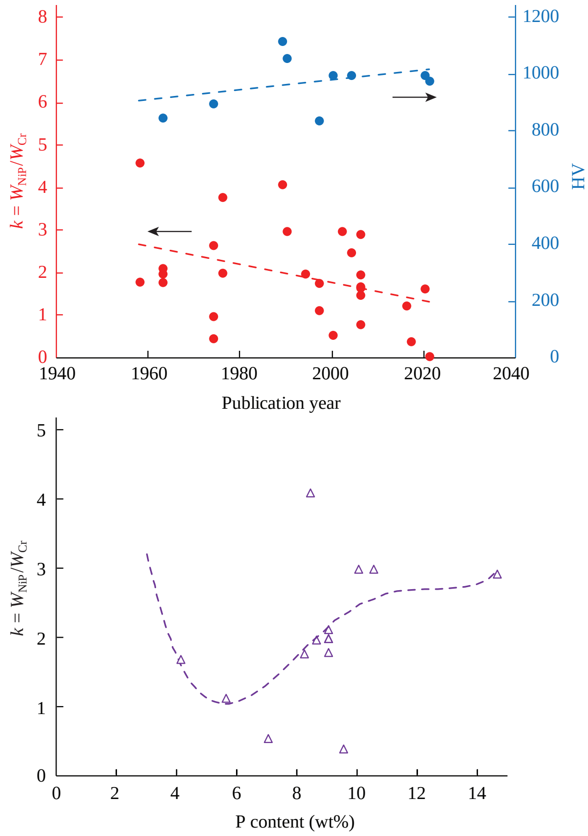
Ni–P coatings, with their high microhardness (HV) and wear resistance, have been proposed as a viable alternative to hard chromium. However, wear testing experiments on electroless Ni–P coatings were conducted under a variety of conditions, including test equipment, load, substrate materials and counterface materials, resulting in unreliable comparative evaluations of experimental results. Therefore, this article presents an analysis of experimental research on Ni–P and Cr coatings, as well as a statistical analysis of the results obtained under similar conditions. The results of simultaneous wear tests on Ni–P and Cr coatings published from 1958 to 2022 were used for evaluation and comparative analysis. The relative wear of Ni–P *versus* hard Cr was calculated from experimental data and analyzed using statistical methods such as Tukey's statistical methods, nonparametric and parametric statistics. This review found that the wear performance of electroless nickel coatings generally does not match that of hard chromium coatings. The results of this analysis indicate that: (i) Ni–P coating can be used as a protective and hardening coating; (ii) the wear resistance of the electroless Ni–P coating, being 1.9 ± 0.4 times less, does not reach the same level of wear resistance as that of hard Cr; (iii) there is a tendency for the wear resistance of Ni–P to gradually approach the wear resistance of Cr; (iv) the dependence of the wear rate on the phosphorus content of the Ni–P alloy indicates that the minimum possible wear rate is achieved at a P content of about 4–7 wt%. The search for effective alternatives to hard chromium plating is ongoing, and this review has identified several areas where effective engineering solutions could be found.



Keywords: alloys, electroless deposition, Ni–P coating, hard chromium, microhardness, nonparametric statistics, relative wear, tribological properties, wear resistance, engineering coatings.

Introduction

Environmental considerations are increasingly changing the electroplating industry. Current environmental regulations often require replacing the existing electroplating process with more environmentally friendly technologies.[1,2] The development of cleaner electroplating technologies is a priority supported by environmental legislation in many countries.[3,4] Environmental regulations generally recognize chromic acid, used for hard chromium plating, as a highly toxic and carcinogenic chemical. The US Environmental Protection Agency and EU environmental agencies list chromic acid as a 'high priority' toxicant.[5] Chromium electroplating from chromic acid solutions is widely used in industry.[6,7] Currently, it is one of the most important electroplating technologies. The use of chromates requires special waste disposal processes, expensive personal respiratory protection devices and exhaust systems to control process emissions.[8] For these reasons, alternative technologies, substitutes and new designs continue to attract great interest.[9] Unfortunately, few coatings can completely replace conventional hard chromium with its high microhardness (HV), low coefficient of friction and excellent resistance to wear and corrosion. To date, alternatives to chromium coatings have been explored, such as composite coatings and coatings from solutions of trivalent chromium.[10] Several binary (Ni–W, Ni–P and Co–W),[9] ternary (Ni–Co–P,[11] Ni–Cu–P,[12] Ni–W–P[13] and others) and quaternary alloys have been proposed as replacements for conventional hard chromium coatings.[14,15] Nickel alloy coatings can be considered as an alternative to hard chromium, since the microhardness of Ni–P alloys after heat treatment at 400 °C is almost as high or higher than that of conventional hard chromium.

The technology for producing nickel–phosphorus (Ni–P) coatings was proposed in 1946.[16,17] These coatings, as well as Ni–P alloy composites, are of interest due to their high microhardness, acceptable wear resistance and corrosion resistance.[18–20] Some of the well-studied coatings are already used for various purposes[9,21,22] and are reported to demonstrate high wear resistance.[23,24] However, their brittleness and insufficient integrity after heat treatment limit their use as a replacement for hard chromium under heavy loads with the occurrence of significant through cracking.[15,25,26]

The increased wear resistance of such coatings is usually associated with their high microhardness and sufficient ductility.[27] The abrasion resistance of all Ni–P coatings is also directly related to phosphorus content, heat treatment and adhesion to the metal substrate.[28] Higher phosphorus content and increased microhardness after heat treatment improve abrasion resistance.[29] It has been established that with increasing phosphorus content in the coating, the heat treatment temperature should be about 600 °C to reduce wear.[30]

The abrasion test methods specified in ISO 4527 depend on the specific application of electroless Ni–P coatings. Studies of the wear resistance of Ni–P coatings and composites have established that after heat treatment at 400 °C for 1 h, the samples have higher microhardness and better wear resistance.[31,32] Under high load and dry friction conditions, nickel coatings with 10 wt% P showed significant transfer of iron on the worn surface. No direct relationship was observed between wear rate and microhardness.[33]

Some reviews[9,27,34–37] are devoted to the study and optimization of electroless nickel plating technology aimed at increasing the wear resistance of Ni–P coatings. However, results from wear testing of coatings are difficult to generalize due to the complexity and diversity of testing machines, different loads and environmental conditions during wear testing, and the variety of substrates and counterface materials used. The wear patterns of nickel–phosphorus coatings and their comparison with conventional chromium wear-resistant coatings have not been sufficiently studied to realize their potential.

In this review, we aim to generalize the results of wear tests on Ni–P and chromium alloy coatings and perform a statistical analysis of the results obtained under identical conditions.

1. Data analysis

In the sampling phase, we analyzed research papers on the wear resistance of Ni–P and conventional hard chromium coatings published between 1958 and 2021 and selected those suitable for subsequent analysis.

The next step was to determine the object of research. Because tribological test conditions, including test procedure, counterpart material and shape, loads, humidity, duration, wear metrics, *etc.*, are different, published results were not directly comparable. To account for different test conditions and create a unified scale for wear rates W expressed in a variety of units (mg, mg per 10^3 cycles, µm, $mm^3\ min^{-1}$, $mm^3\ N^{-1}\ m^{-1}$, *etc.*), we calculated the relative wear rate k of Ni–P alloy coatings compared to conventional hard chromium (Table 1):

$$k = W_{NiP}/W_{Cr}.$$

This parameter indicates whether the wear of the alloy coating is greater or less than the wear of the chrome coating when tested under identical conditions. This approach allowed us to combine data reported by many researchers from around the world.

For our analysis, we applied a set of tools that included Tukey's exploratory data analysis,[38] nonparametric statistics,[39] specifically the G sign test,[10,40] and parametric statistics.[41]

We then performed the G sign test and an estimation of its observed value. The G sign test determines whether there are too many 'atypical' shifts in the sample ($k < 1$) such that a shift in the 'typical' direction ($k > 1$) is considered statistically significant. Zero shifts ($k = 1$) are excluded and the sample size is reduced. G_{emp} is the number of observed 'atypical' shifts. The smaller this

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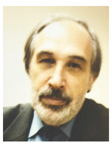

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Table 1 Summary of published data on the wear resistance of electroless Ni–P coatings (W_{NiP}), which are not comparable due to varying testing conditions, into a unified scale of relative wear rates ($k = W_{NiP}/W_{Cr}$) by correlating them with the wear resistance of hard Cr coatings (W_{Cr}) measured under the same conditions.

Entry	Source	Publication year	Wear test method	Tribometer model	Counterface		Wear unit	Cr coating wear (W_{Cr})	Ni–P coating			
					Material	HRC			P content (wt%)	HV	Wear (W_{NiP})	Relative wear (k)
1	Ref. 42	1958	Pin-on-disc	AE-5	Steel grade 30KhGSA (analog: 30HGS)	32	mg	17.5			31.6	1.81[a]
2	Ref. 42	1958	Pin-on-disc	AE-5	Steel grade 30KhGSA (analog: 30HGS)	32	mg	18			83	4.61[a]
3	Ref. 43	1963	Reciprocal	77 MT-1	Steel grade 30KhGSA (analog: 30HGS)	32	mg	20	9	850	40	2.0
4	Ref. 43	1963	Reciprocal	77 MT-1	Steel grade 30KhGSA (analog: 30HGS)	32	mg	2.0	9		3.6	1.80[a]
5	Ref. 43	1963		MI	Grey cast iron		mg	0.76	9		1.62	2.13[a]
6	Ref. 44	1974		Falex machine	V block, no plating	20–24	mg	6.2		900	3.0	0.48
7	Ref. 44	1974		Falex machine	Pin, no plating	60	mg	0.5			0.5	1.0
8	Ref. 44	1974	Taber		SiC (CS-10)		mg per 10^3 cycles	3			8	2.67
9	Ref. 45	1976	Taber		SiC (CS-10)		mg per 10^3 cycles	2			7.6	3.8
10	Ref. 45	1976	Taber		CS-17		mg per 10^3 cycles	8.4			17	2.02
11	Ref. 46	1989	Pin-on-flat		Steel grade 080M40				8.4	1120		6.2
12	Ref. 46	1989		Falex machine	Steel grade 080M40				8.4			**32**
13	Ref. 46	1989	Scratch test		Diamond		µm		8.4			**46**
14	Ref. 46	1989	Taber		SiC (CS-10)		mg per 10^3 cycles		8.4			4.1
15	Ref. 47	1990	Taber		SiC (CS-10)		mg per 10^3 cycles	2	10	1060	6	3.0
16	Ref. 48	1994	Taber		SiC (CS-10)		mg per 10^3 cycles	2	9		4	2.0
17	Ref. 49	1997		Crossed cylinder wear tester	Steel	47	µm min^{-1}	0.7	5.6–8.2	675–840	0.8–1.25	1.14–1.78[b]
18	Ref. 50	2000		MM-200 wear tester	Steel bonded WC hard alloy	63	$mm^3\ min^{-1}$	3.8×10^{-5}	7	1000	2.1×10^{-5}	0.56[a]
19	Ref. 51	2002	Taber		SiC (CS-10)		mg per 10^3 cycles	2–3	10–11		2–9	3.0
20	Ref. 52	2004	Taber		SiC (CS-10)		mg per 10^3 cycles	1–4.7		1000	11.6	2.5
21	Ref. 53	2006	Ball-on-disc	UMT-2MT	Si_3N_4 ceramic ball		$mm^3\ N^{-1}\ m^{-1}$	1.5×10^{-5}	[c]		2.25×10^{-5}	1.5[a,b]
22	Ref. 53	2006	Ball-on-disc	UMT-2MT	Si_3N_4 ceramic ball		$mm^3\ N^{-1}\ m^{-1}$	9.6×10^{-6}	[c]		1.6×10^{-5}	1.67[a,b]
23	Ref. 54	2006	Ball-on-disc	UMT-2MT	Si_3N_4 ceramic ball		$mm^3\ N^{-1}\ m^{-1}$	4.3×10^{-5}	14.6		1.26×10^{-4}	2.93
24	Ref. 54	2006	Ball-on-disc	UMT-2MT	Si_3N_4 ceramic ball		$mm^3\ N^{-1}\ m^{-1}$	4.3×10^{-5}	8.6		8.5×10^{-5}	1.98
25	Ref. 54	2006	Ball-on-disc	UMT-2MT	Si_3N_4 ceramic ball		$mm^3\ N^{-1}\ m^{-1}$	4.3×10^{-5}	4.1		7.3×10^{-5}	1.70
26	Ref. 54	2006	Ball-on-disc	UMT-2MT	Si_3N_4 ceramic ball		$mm^3\ N^{-1}\ m^{-1}$	4.3×10^{-5}	[c]		3.5×10^{-5}	0.81
27	Ref. 55	2013	Taber		SiC (CS-10)		mg per 10^3 cycles	3			18	6.0
28	Ref. 56	2016		MFT-5000 wear tester	Stainless steel		µm	2.8			3.5	1.25
29	Ref. 57	2017	Scratch test		Al_2O_3 ball		µm	404	9–10		164	0.41
30	Ref. 9	2020			Steel grade AISI 52100		$mm^3\ N^{-1}\ m^{-1}$	2.0×10^{-5}		1000	3.3×10^{-5}	1.65[b]
31	Ref. 58	2021		Plint TE66 wear tester	Steel grade AISI 52100/ Gerdau VC10 tool steel		$g\ N^{-1}\ m^{-1}$	1.03×10^{-7}		980	6×10^{-9}	0.06[d]

[a] Testing with lubricant. [b] Coating obtained by electrodeposition. [c] Graded Ni–P coating. [d] After treatment at 290 °C for 10 h.

value, the more likely it is that a shift in the 'typical' direction is statistically significant.

To determine trends in the relative wear of Ni–P alloy coatings, we used regression analysis.

2. Results

The dataset was collected from a variety of published articles from Brazil, China, India, Russia, UK and USA (see Table 1).

The k values selected for further analysis (see Table 1) indicate significant variation in the data (max: 46, min: 0.06) with possible minor and extreme outliers. We used Tukey's exploratory data analysis[38] to assess outliers: numbers that deviate from the median (Me) by more than three interquartile ranges (IQR) should be considered extreme outliers; numbers that deviate from Me by more than 1.5 IQR [lower whisker (LW) and upper whisker (UW)], but less than 3 IQR, should be considered minor outliers.

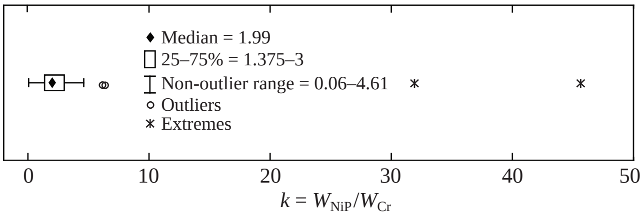

Figure 1 Box-and-whisker plot of the relative wear values (see Table 1).

The following parameters are defined for the dataset in question (Figure 1):

$$Me = 1.99,\ Q1 = 1.313,\ Q3 = 3,\ IQR = 1.687,$$

$$LW_{extr} = Me - 3IQR = -3.07,\ UW_{extr} = Me + 3IQR = 7.05,$$

$$LW = Me - 1.5IQR = -0.54 \text{ and } UW = Me + 1.5IQR = 4.52.$$

Two values in Table 1, 32 (entry 12) and 46 (entry 13), are significantly beyond the UW_{extr} boundary. Therefore, these values as extreme outliers were excluded from the dataset before analysis. The values of 6 (entry 27) and 6.2 (entry 11) in Table 1 slightly exceed the UW boundary, so we did not exclude them from the dataset. The median of the new dataset decreased slightly to 1.90.

Because the new dataset is small ($n = 30$), we used the Shapiro–Wilk test[41] to assess the normality of the distribution for its highest power. We proposed two hypotheses.

The null hypothesis $\mathbf{H_0}$ assumes that the experimental data are drawn from a normally distributed population.

The alternative hypothesis $\mathbf{H_1}$ states that the experimental data are not normally distributed.

We obtained observed test values of $W = 0.89882$ and $p = 0.00786$. At a significance level of 0.05 ($p < 0.05$), the null hypothesis is rejected. Consequently, the distribution of the dataset is not normal and only nonparametric statistics are applicable.

The statistical confidence of the decrease in wear resistance was estimated using the nonparametric *G* sign test. It shows the general direction of change of a given variable (wear resistance) in the dataset.[10]

The dataset ($n = 30$) includes 5 cases of increased wear resistance ($k < 1$, positive shift), 1 case of unchanged wear resistance ($k = 1$, zero shift) and 24 cases of decreased wear resistance ($k > 1$, negative shift). This means that the decrease in wear resistance is a 'typical' shift. For our dataset $G_{emp} = 5$.

We formulated the following hypotheses:

$\mathbf{H_0}$: The predominance of negative shift is accidental.

$\mathbf{H_1}$: The predominance of negative shift is not accidental.

Table 2 Results of data processing after excluding outliers.

Entry	Statistical parameter of the relative wear coefficient (*k*)	Value
1	Mean	1.94
2	Standard deviation	1.106
3	Confidence limit	0.41
4	Confidence (−95%)	1.53
5	Confidence (+95%)	2.35
6	Median	1.80
7	Interquartile range (IQR)	1.39
8	Median absolute deviation (MAD)	0.68
9	First quartile (Q1)	1.20
10	Third quartile (Q3)	2.59

For our dataset ($n = 30$), G_{crit} is 9 for a significance level of $p \leqslant 0.05$ and 7 for $p \leqslant 0.01$.[40] Since $G_{emp} < G_{crit}$, hypothesis $\mathbf{H_0}$ was rejected and hypothesis $\mathbf{H_1}$ was accepted. The decrease in wear resistance when replacing chromium coatings with Ni–P alloy coatings is statistically significant for all significance levels considered. Based on the median value *k* of the dataset, we can state that the wear of Ni–P alloy coatings is 1.9 times greater than that of chromium coatings.

To apply parametric statistics, it is advisable to exclude *k* values of 6 and 6.2, since they are above the UW boundary. After excluding these values, the observed Shapiro–Wilk test ($W = 0.96045$ and $p = 0.35724$) indicates that the distribution of the dataset is normal with a significance level of 0.05 ($p > 0.05$) (Figure 2).

Consequently, the distribution after excluding all four outliers ($n = 28$) is normal and parametric statistics can be applied. Table 2 lists the results of data processing.

3. Discussion

Our results (see Table 2) show that the average wear of Ni–P alloy coatings is 1.9 ± 0.4 times higher than that of hard chromium coatings.

To assess the evolution of the wear resistance of coatings over the years (1958–2021), we identified a relationship between relative wear and the year of publication of test results (Figure 3, curve *1*) and estimated the pairwise correlation coefficient (*r*) as −0.3865. Despite the significant scattering of data ($n = 28$, df = 26), there is a fairly reliable correlation for $p = 0.0422$ ($1 - p = 0.9578$) and a trend for the *k* value to decrease to 1.

The microhardness of coatings tested for wear resistance (Figure 3, curve *2*) increased slightly from 850 HV in 1963 to 1000 HV in 2021. This partially contributed to the reduction in wear.[59–61] The microhardness values of chromium and Ni–P alloy coatings can be considered suitable for the application

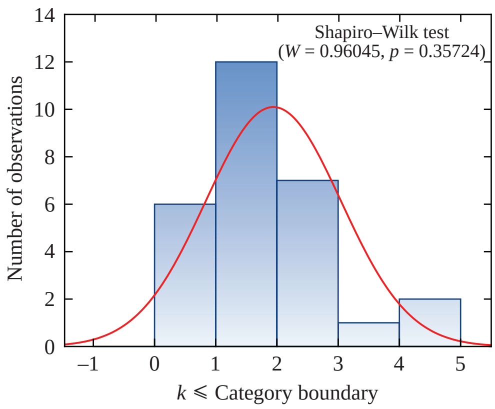

Figure 2 Normal distribution of the relative wear coefficient *k* after eliminating outliers.

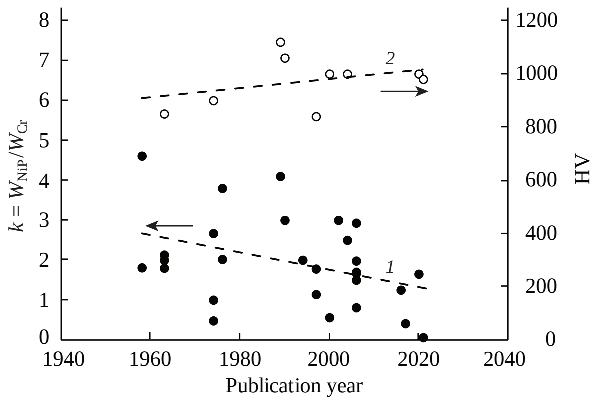

Figure 3 Trends in (*1*) relative wear and (*2*) HV of tested coatings depending on year of publication.

of Ni–P alloys as protective and hardening coatings to replace hard chromium where parts are subject to mechanical loads rather than friction. The wear resistance values of coatings measured in different years show (see Figure 3) that the average relative wear is steadily decreasing. This indicates the ongoing search for solutions to improve the wear resistance of electroless coatings to the level of hard chromium. For example, Sahoo[62] found that the concentration of nickel ions in the electroless plating bath significantly affects the friction characteristics of the Ni–P coating. Under optimal conditions, the coefficient of friction is reduced by approximately 60%. Sahoo *et al.*[63] also showed that nickel ion concentration and solution temperature are the most important process variables that control the coefficient of friction and wear rate of the coating.

We analyzed papers that reported minimum wear rates,[44,54,57,58] but were unable to determine the exact composition of the solution. For electroless Ni–P plating baths, the authors either used commercially available products, for example, NICHEM–ATOTECH,[57] or did not specify the composition. Only one paper[50] indicates the composition of the solution, including 20 g dm^{-3} $NiSO_4 \cdot 6H_2O$, 10 g dm^{-3} $AcONa \cdot 3H_2O$, 10 g dm^{-3} $Na_3C_6H_5O_7 \cdot 2H_2O$, 15–20 g dm^{-3} $NaH_2PO_2 \cdot H_2O$, surface activator and stabilizer. Without information on the composition of the solutions, we were unable to assess the effect of the process variables on the wear rate of the coating.

The scattering of data presented in Figure 3 (curve *1*) may be due to various levels of phosphorus content in the coatings. Researchers report values ranging from 4.1 to 14.6 wt% (see Table 1). Graphic analysis of the dependence of wear rate on phosphorus content in the alloy indicates the minimum possible wear value for alloys with a P content of about 4–7 wt%. Some authors, whose results for various reasons are not included in Table 1 and Figure 4, also note minimal wear in this range of compositions (Table 3).

In most cases, Ni–P coatings were tested after heat treatment at 400 °C. In the cited work,[30] the authors found that for coatings with a phosphorus content of 10–12 wt% heat treatment at 600 °C is more effective in terms of wear reduction than heat treatment at 400 °C.

This result requires further research into the effect of phosphorus content on wear resistance. The combined effect of heat treatment temperature and phosphorus content needs to be studied because different phosphorus contents may be required to achieve maximum wear resistance under specific heat treatment conditions.

Conditions for plating (solution composition, pH and temperature) and heat treatment of electroless coatings, their composition and structure, which together may provide consistently high wear protection, have yet to be clearly defined. Undoubtedly, further optimization of wear-resistant electroless coating technology will define suitable materials and conditions to create a viable alternative to hard chromium coatings.

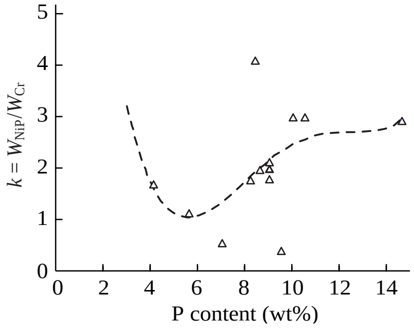

Figure 4 Relative wear of Ni–P coatings as a function of P content.

Table 3 Phosphorus content in Ni–P coatings with minimum wear rates.

Entry	Source	Publication year	P content (wt%)							
			1	2	3	4	5	6	7	8
1	Ref. 61	1982					+			
2	Ref. 64	1996			+	+	+			
3	Ref. 65	1998				+				
4	Ref. 66	2000			+	+	+			
5	Ref. 67	2002			+	+				
6	Ref. 54	2006				+				
7	Ref. 68	2008				+	+			
8	Ref. 69	2020		+	+	+				

Conclusion and future developments

This review revealed that the wear performance of electroless nickel coatings generally does not match that of hard chromium coatings. The results show that, on average, the wear rate of Ni–P alloy coatings is 1.9 ± 0.4 times higher than that of hard chromium coatings. The dependence of wear on the phosphorus content in the Ni–P alloy indicates that the minimum possible wear is observed in alloys with a P content of about 4–7 wt%. We found a sufficiently reliable correlation ($p = 0.0422$, $1 - p = 0.9578$) in the evolution of relative wear (k), with k tending to 1 or less. Due to the lack of information on the composition of solutions for electroless coating deposition in studies reporting minimal relative wear, we were unable to identify the effects of process variables. The search for effective alternatives to hard chromium plating is far from over. Further research will bring us closer to solving this important problem.

The review identified several areas where efficient engineering solutions could be found.

First, further study is needed on the dependence of the wear rate of Ni–P coatings compared to chromium coatings on electroless plating process variables, such as the qualitative and quantitative composition of the solution and deposition conditions, and on the optimal annealing temperature for a particular coating composition.

Secondly, the effect of the third component in the Ni–P alloy on the wear resistance of electroless coatings requires additional research. The third component can be either hard metals, such as tungsten or molybdenum, or relatively soft metals, such as copper or silver, that can act as friction lubricants.

Thirdly, a promising alternative to hard chromium plating is electroless composite coatings containing dispersed particles of hard and/or soft materials. Intensive research into electroless composite coatings has not provided clear answers to numerous questions about the influence of phosphorus content, as well as the nature, content and degree of dispersion of particles on the microhardness, wear resistance and other mechanical properties of such coatings.

Fourth, the effectiveness of alternative coatings under certain wear conditions has not yet been determined. Such research may lead to at least a partial replacement of hard chromium plating in industry.

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