

Alloys are Promising As Chromium or Cadmium Substitutes

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A Significant amount of work on electrodeposited alloys is available. Browsing the Internet and pointing the browser on alloys can get over 40,000 hits. The commercial database STN¹ gets more than 400,000 hits on the term alloy. One article cannot cover all of the available alloys. This one discusses the alloys that are being used or evaluated as replacements for cadmium and chromium.

More than 5000 years ago man discovered alloys and how combining two or more metals changed the properties of both. This example is bronze and the combination of copper and tin, which produced a metal that was harder than either metal alone. Man is still combining metals to enhance the performance of single metals. One way to improve the corrosion resistance of a metal is to alloy that metal with a more noble metal. To improve the corrosion resistance of zinc, the noble metal is usually Ni, Fe, Sn or Co.

Alloying metals also can change the color of the metal in addition to affecting the corrosion resistance. Some examples of this are Sn/Ni, Sn/Co, Ni/W, Co/W and Cr/Fe. These alloys have a different color than the primary alloying metal. The unique colors of these alloys will be discussed here, along with the physical properties of the alloys, such as color, hardness, wear and corrosion resistance.

Alloy coatings as a replacement for hexavalent chromium

There is currently no one coating that possesses all the attributes needed to replace decorative hexavalent chromium. Some of the alloys evaluated for both rack and barrel plating, possess some of the desired properties, such as color, hardness, stripability, throwing power and waste treatment to meet the customer's

expectation. Each alloy has unique physical properties and these will be discussed.

Plating Process

Commercially viable decorative hexavalent chromium replacements have been around for more than 20 years.² The first process was based on trivalent chromium chemistry. The trivalent chromium species is not as toxic as the hexavalent chromium species. Therefore, industry has and is evaluating plating processes based on trivalent chromium. The main difference between trivalent chromium and the nickel and cobalt alloy as substitute for hexavalent chromium is that the electrodeposit from the trivalent chromium electrolyte is still chromium, whereas the other coatings are not. Initial deposits of chromium from the trivalent chromium electrolyte were darker than the hexavalent chromium electrodeposit, which made acceptance of the color an issue. Over the last few years, research on trivalent chromium electrodeposit has increased and deposits that resemble hexavalent chromium are being produced.^{2,3} Figure 1 shows the color differences of the various chromium electrodeposits.

Of the trivalent chromium electrodeposits, none possesses the blueness of hexavalent chromium. The newer trivalent chromium, however, comes the closest to matching hexavalent chromium electrodeposit. The older trivalent chromium electrodeposits were more yellow- and green-tinted than hexavalent chromium deposits. With proper operation and experimental design optimization of the trivalent chromium electrolyte, it is

possible to obtain deposits that are indistinguishable from hexavalent chromium deposits.⁴

Tin/Nickel Alloys

Nickel and cobalt alloys with tin were first developed more than 45 years ago. They were developed to see whether the hardness and abrasion resistance of tin could be improved by the presence of iron group metals.⁵ Most of this work consisted of Sn/Ni alloys. Electrodeposited Sn/Ni alloy is an example of an alloy with properties that are not the average of the properties of its elements. In hardness, tarnish resistance, and resistance to chemical reagents, it far excels both nickel and tin. Table 1 shows solution composition.

Table 1
Solution Composition
Sn/Ni Alloy

NiCl ₂ · 6H ₂ O	300 g/L
SnCl ₂ · 2H ₂ O	50 g/L
NaF	28 g/L
NH ₄ F	35 g/L

The operating parameters of this solution are quite severe and the electrolyte is very corrosive. Special care is used when operating this

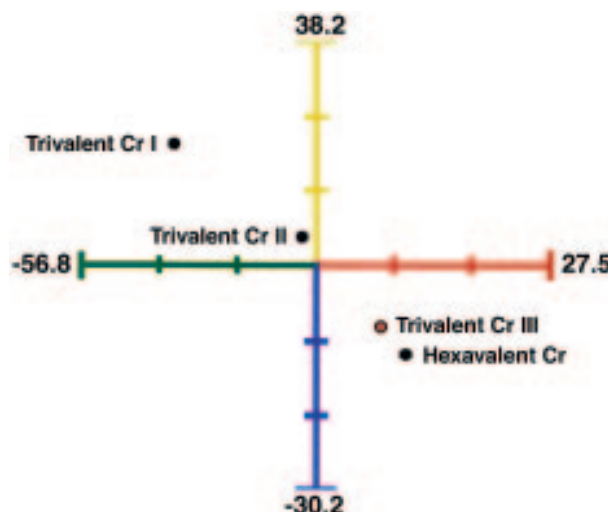


Fig 1—Color differences of chromium deposits.

electrolyte. Table 2 shows operating parameters for decorative applications for barrel and rack solution. Tin/nickel coatings can be plated thick. There is an ASTM standard for tin/nickel coatings.⁶

Table 2
Operating Parameters
For Decorative Applications

Time	2-8 min
Current Density	1-30 A/ft ²
pH	<2.5
Cathode Efficiency	100% Anode
Nickel Agitation	mechanical-no air

There is another tin/nickel process that operates at a higher pH than the first tin/nickel. The higher pH process does not use hydrofluoric acid to adjust pH like the acid process does. This process uses a complexor to chelate the nickel so it does not precipitate as NiF₂. The high pH tin/nickel process offers higher current density range and less corrosive electrolyte than the acid tin/nickel electrolyte. Also, there is no difference in alloy composition from the high pH Tin/Nickel versus the acid Tin/Nickel electrolyte. Alloy composition of the Sn/Ni is remarkably stable over a wide current density range, because Sn/Ni is deposited as an intermetallic compound rather than an alloy. Table 3 shows alloy composition over a wide current density range. From this observation, the alloy composition did not vary significantly over these current densities.

Table 3
Alloy Composition

Current Density	%Sn	%Ni
5 A/ft ²	69.8	30.2
10 A/ft ²	70.3	29.7
20 A/ft ²	70.2	29.8
30 A/ft ²	70.5	29.5
40 A/ft ²	70.3	29.7

Besides uniform alloy composition over a wide current density range tin/nickel has exceptional covering power, ability to plate in low current density areas, compared with most acid electrolytes. The only other electrolytes that possess covering power as good as this are cyanide copper or stannate tin electrolyte. Hardness of the tin/nickel, 650 HK100, is between that of bright nickel, 450-550 HK and chromium, 900 HK100. This hardness is especially important for barrel plating large parts. With small parts, the force with which the parts tumble against each other is slight, and other types of alloy coatings can and are used to plate this type of part. When the parts get large, the force against each other is considerable, and a soft coating will scratch. Tin/nickel is hard enough not to scratch, but other tin-based alloys are not and will be discussed later. The corrosion resistance of Sn/Ni is not as good as one would expect from its chemical resistance. Tin/nickel is cathodic to steel but anodic to nickel, like all the alloys that will be discussed, and the corrosion protection is based on nickel thickness. See Figure 3 for a comparison of Tafel plots of the various alloys. All the alternative coatings need to be passivated in a hot

dilute chromic acid solution for optimum corrosion protection. Thin nickel deposits (less than 7 microns) will be porous and corrosion is accelerated because of the porosity of the nickel. Thicker nickel coatings will give better corrosion protection, and duplex nickel coatings will give even better corrosion protection. Color is a critical factor for decorative application. Of the alternatives none quite matches hexavalent chromium's blue-white hue but the new

trivalent chromium and the acid Sn/Co and Co/W are the closest. Figure 2 shows a color plot of the various alloys compared to hexavalent chromium. The Sn/Ni has a slight red hue and Ni/W has a yellow hue compared with all the alternatives. With any number less than 2, the human eye cannot readily perceive any color difference.⁷

Tin/Cobalt Alloys

There are two basic electrolytes for plating tin/cobalt alloys, acid and alkaline. The acid electrolyte is similar to the low pH tin/nickel process. The basic composition for the acid tin/cobalt process is listed in Table 4.⁸

Table 4
Solution Composition
Acid Tin/Cobalt

CoCl ₂ · 6H ₂ O	375 g/L
SnCl ₂	45 g/L
NH ₄ HF ₂	82.5 g/L
NH ₄ Cl	37.5 g/L
pH	<3

The operating parameters are similar to the acid tin/nickel process except insoluble anodes are used and the anode is usually graphite. Composition of the alloy is more varied in the Sn/Co processes than in the Sn/Ni. The hardness of the tin/cobalt is between 300-400 HK100. That is soft compared with chromium and tin/nickel. This softness limits the usefulness of this coating. With a soft deposit and with barrel plating parts there is a tendency for the parts to have a scratched topcoat.

Covering and throwing power is similar to the tin/nickel process. One positive aspect of the acid tin/cobalt process is the color of the coating.

Alkaline tin/cobalt has similar properties to the acid tin/cobalt, except color. Table 5 shows the alloy composition of the alkaline and acid tin/cobalt process.

Table 5
Tin/Cobalt Process

CD(A/ft ²)	Alkaline		Acid	
	%Sn	%Co	%Sn	%Co
40	80	20	58	42
30	72	28	68	32
20	70	30	70	30
10	65	35	72	28
5	65	35	72	28

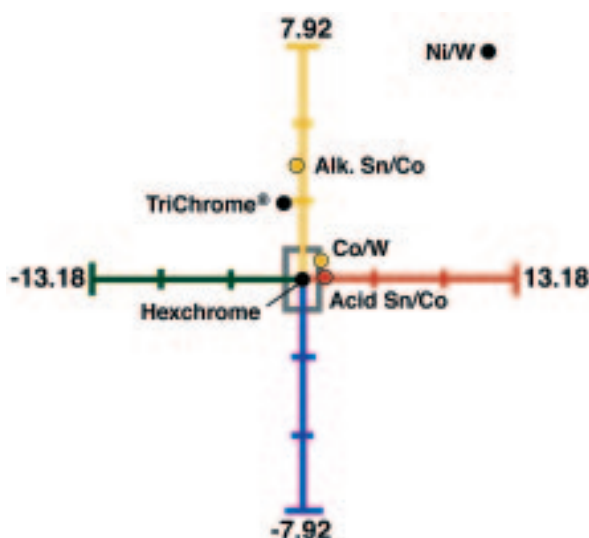


Fig 2—Color plot of alloys compared to hexavalent chromium.

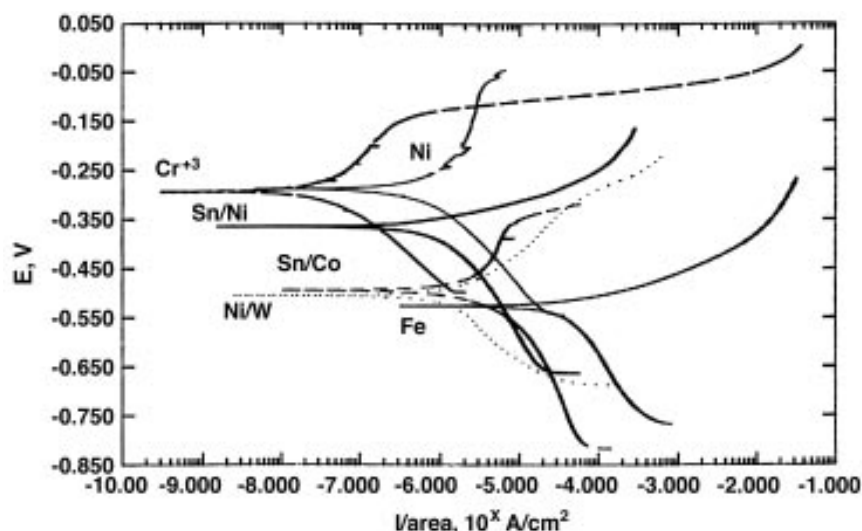


Fig 3—Tafel plots of various alloys.

The alkaline tin/cobalt composition varies more than the acid tin/cobalt and acid tin/nickel over the same current density range. This process is more suitable for barrel application for this reason. The metal concentration is significantly lower in this process than the acid process. Table 6 lists solution chemistry.

Table 6
Solution Composition
Alkaline Tin/Cobalt

Additive	Concentration
CoSO ₄ · 7H ₂ O	3-5 g/L
SnSO ₄	2-4 g/L
Complexor	10-20 g/L
Conductivity Salt	20-30 g/L
Wetter	0-0.2%
pH	6.5-8.5

With low metal concentration, the dragout from the barrel of the alkaline Sn/Co is less than with the high metal concentration process, such as Sn/Ni or acid Sn/Co. This makes the alkaline Sn/Co less stressful on the waste treatment needs, because the

tin-based alloys are not amenable to reclaiming. Tin hydrolyzes to the +4 valance, which is detrimental to all plating processes discussed so far.

Nickel/Tungsten & Cobalt/Tungsten Alloys

There are currently two types of nickel/tungsten alloys. One is 65% nickel and 35% tungsten, and the other is 64.5% nickel, 35% tungsten and <0.5% boron. Both processes are similar in chemistry. Nickel/tungsten alloy electrodeposition has been around for more than 40 years and there are two types of chemistry, acid and alkaline.⁹ The acid type electrolytes only produced alloys with low tungsten levels, 5–10%, whereas the alkaline type process has tungsten levels between 20–65%. The basic requirements for formulation of an alkaline tungsten alloy electrolyte, besides the salts of the metal, are ammonium salts, excess ammonium hydroxide and a complexing agent for the iron group metal. Because

these processes were operated hot, greater than 70 °C, and the pH was about nine, significant amounts of ammonia had to be added to maintain the pH. This is probably why these processes did not gain much commercial acceptance. Over the last few years research on nickel/tungsten has modified the earlier version by lowering the temperature and pH but still keep the tungsten in the 30-40% range. The Co/W process is similar in chemistry to the Ni/W process. Table 7 list the basic chemical composition.

Table 7
Solution Composition,
Co/W Process

Additive	Concentration
Na ₂ WO ₄ · 2H ₂ O	10-75 g/L
NiSO ₄ · 6H ₂ O	4-25 g/L
Organic Complexor	30-80 g/L
NH ₄	10-20 g/L
Brightener	0-0.4%
Wetter	0-0.3%
pH	6-7.5

The metal concentration of the Ni/W and Co/W alloy is less than the tin/nickel and acid tin/cobalt process but more than the alkaline Tin/Cobalt process. The Ni/W and Co/W process can be run for both decorative and functional coatings. For decorative use, the Ni/W possesses properties that make it an ideal replacement, except for one property. Ni/W possesses high hardness, 650±50 HK100, good corrosion protection but the color is yellow when compared to the other alloy replacements. In addition the throwing power of Ni/W is less than the other alloys, but better than chromium. Alloy composition of the Ni/W and Co/W varies slightly versus current density. Table 8 lists the results.

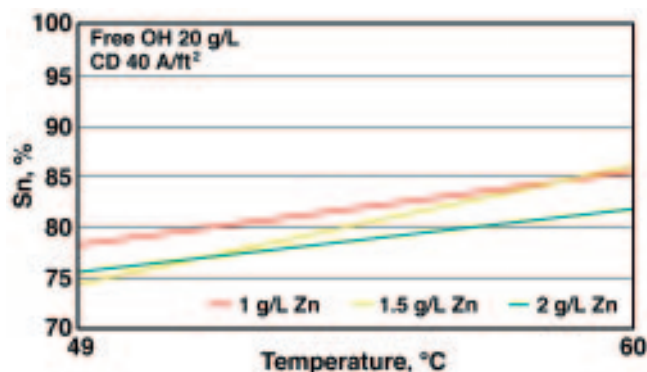


Fig 4—Effect of Zinc and temperature on Sn/Zn alloy.

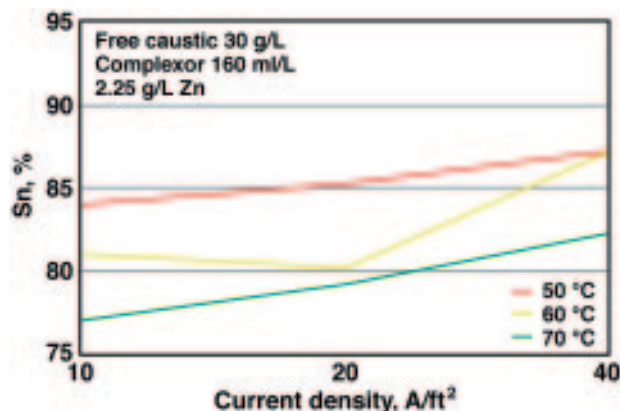


Fig 5—Effect of temperature and current density on Sn/Zn alloy.

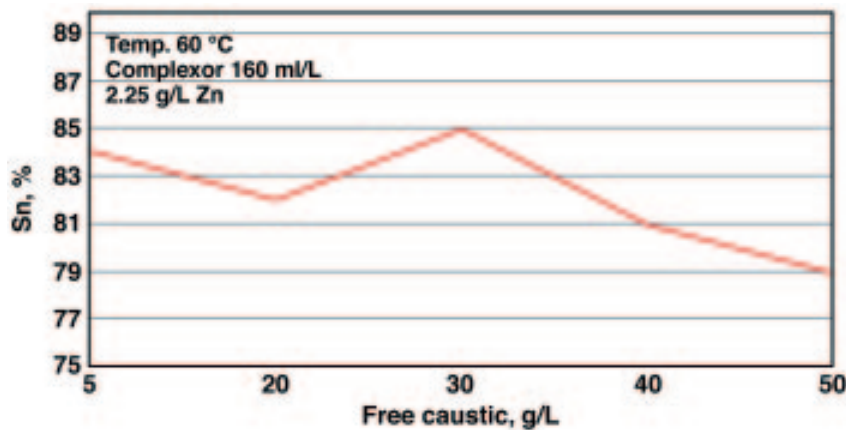


Fig 6—Effect of free caustic on Sn/Zn alloy.

CD (A/ft ²)	%Ni	%W	%Co	%Ni
40	85	15	57	43
30	66	34	57	43
20	71	29	57	43
10	63	37	59	41
5	60	40	62	38

The operating parameters of the Ni/W and Co/W in rack application are:

Time 1-4 min
 CD(A/ft²) 5-50
 Temperature 120-140 °F
 Cathode Efficiency 25-35%
 Anodes Stainless Steel

The corrosion properties of Ni/W are similar to the other alloys evaluated. Figure 3 shows Tafel plots of the various alloys. Table 9 lists the $E_{(corr)}$ or open circuit potential of the various alloys. From this information one can determine how the material acts, either anodic or cathodic to the

substrate, depending on its potential. All the alloys are more noble to Fe but are sacrificial to Ni. In decorative applications the substrate is usually Ni-plated. One does not obtain the corrosion protection of chromium unless alloys are passivated in a hot dilute chromic acid solution.

Alloy	$E_{(corr)}$ vs SCE
Sn/Ni	0.360 V
Sn/Co	-0.490 V
Ni/W	-0.510 V
Fe	-0.560 V
Ni	-0.300V
Cr ³⁺	-0.295 V

Also, when looking for a replacement for chromium, wear is another property that needs to be evaluated. Just because a coating has good hardness does not necessarily mean good wear. Table 10 list results of the dropping sand test with 20 lb of sand.

Table 10
Results , Dropping Sand Test

Coating	Weight loss (mg)
Sn/Ni	9.0
Acid Sn/Co	2.0
Alk. Sn/Co	2.7
Ni/W	1.8
Co/W	2.6
Cr ³⁺	2.1
Cr ⁶⁺	0.2

None of the coatings was better than hexavalent chromium.

Summary of
 Decorative Substitute
 An assortment of various coatings both alloys and single metal is available as a replacement for hexavalent chromium.

Each process possesses its own unique property and should be evaluated in the environment for which it is replacing hexavalent chromium.

Alloy Coatings as a Replacement for Cadmium
 There is currently no one coating that possesses all the attributes needed to replace cadmium. Cadmium not only possesses excellent corrosion properties, but also low torque tension and solderability, whereas zinc alone only possesses excellent corrosion properties. A significant amount of work has been done and is still being done now to improve the properties of Zn. Most of this work consists of alloying Zn with an Fe-group metal, Ni, Co or Fe or with Sn. In addition, there is work being done to improve the properties of Zn by applying either an organic or inorganic topcoat to seal the Zn and improve the corrosion and torque tension properties of Zn. Only the alloying metals with Zn will be discussed here. Alloying zinc with a more noble metal, such as Ni, Fe, Co or Sn, increases the corrosion resistance significantly when compared to straight Zn. All of these alloys can be plated out of either an acid or alkaline electrolyte, except Zn/Fe, which is usually plated out of an alkaline electrolyte. Depending on the electrolyte, the Zn alloy has approximately the same composition from either the acid or alkaline electrolyte. All alloys have excellent corrosion properties when chromated, but the Sn/Zn alloy has good corrosion properties unchromated. Because the purpose of

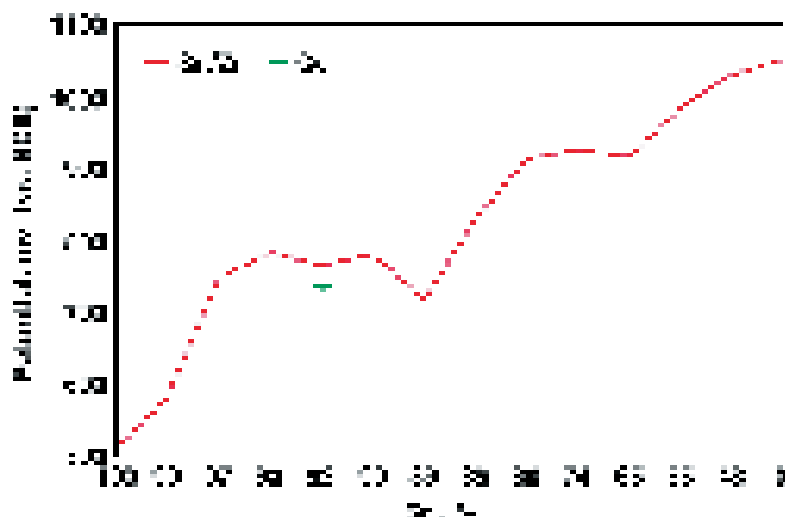


Fig 7—Static potential of Sn/Zn alloy.

this work is to evaluate a coating that possesses properties similar to cadmium, the high Zn alloys (Zn/Ni, Zn/Fe and Zn/Co) only posses the excellent corrosion properties, but not the lubricity or solderability characteristics. There is a significant amount of data about the high Zn alloys in the literature, and we will focus on the alkaline Sn/Zn process.^{10,11}

Introduction to Tin/Zinc

Combining the barrier protection offered by tin with the galvanic protection of zinc, without the bulky corrosion products associated with pure zinc or high percentage zinc alloy deposits, tin/zinc electrodeposits containing 20-30% zinc offer out-standing corrosion protection for steel and other substrates. Since the 1940s, tin/zinc alloys have been plated from cyanide electrolytes, it is not surprising that a number of applications for this alloy have been known for many years.¹² Since the mid-1960s, however, the use of tin/zinc has declined considerably, and now the coating is not used extensively. Although this is in part attributable to the unpopularity of cyanide plating solutions, it is more probable that cyanide tin/zinc plating solutions are very difficult to operate, requiring significant control and service.

Plating Process

In general, alloy plating processes are harder to control than single metal plating processes. This newly developed tin/zinc process follows the same tendencies, however, statistical experimental design techniques employed during process development have made this alloy process easier to control than most others. The electroplating solution is a mixture of potassium stannate, potassium hydroxide, potassium zincate, complexing agent and grain refiner.

Tables 11 and 12 give typical electrolyte composition and operating parameters required to give a deposit containing 20-25% zinc, with the balance tin. Operating parameters, such as solution composition, temperature and current density, can each affect composition of the deposit. The concentrations of tin and zinc in the solution have a direct effect on the composition of the deposit.

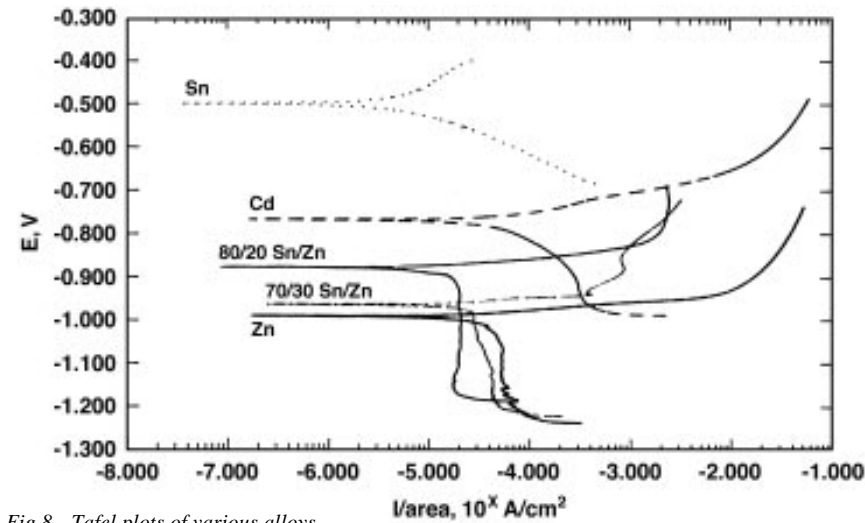


Fig 8—Tafel plots of various alloys.

Table 11 Solution Composition, Tin/Zinc		
Material	Rack (g/L)	Barrel (g/L)
Tin Metal	53-68	45-55
Zinc Metal	0.8-2.5	1.5-2.75
Free KOH	15-60	30-60
Complexor	220-350 ml/L	220-350 ml/L
Grain Refiner	1-2 ml/L	1-2 ml/L

Table 12 Operating Parameters, Tin/Zinc		
Parameter	Rack	Barrel
Temperature	49-55 °C	45-50 °C
Cathode CD	0.5-3.3 A/dm ²	0.2-1.0A/dm ²
Voltage	4-8 Volts	
Filtration	Continuous	
Anodes	Stainless Steel, 304 or 316	
Tank	Polypropylene or polyethylene	
Heaters	Stainless steel or Teflon	

An increase in either concentration is reflected in an increase in the percentage of that metal in the deposit. This is shown in Figure 4,

which also shows the effect of temperature on alloy composition. At lower temperature, less tin and more zinc is deposited inconsequential of the amount of zinc in solution. At higher temperatures, more tin is deposited. Figure 5 shows a more detailed effect of temperature and current density on the composition of the alloy. At higher temperature and current density, the tin concentration in the deposit increases.

Unlike the cyanide process where free caustic has a significant effect on the zinc content in the alloy, the non-cyanide alkaline process does not have that problem. Figure 6 shows the effect of free caustic on the composition of the alloy with the temperature, complexor and zinc content held constant at 60 °C, 300 ml/L and 2.25, g/L respectively. Very little change in the alloy composition over this range of free caustic is observed. There is a trend that, with higher free caustic, more zinc in the deposit is observed.

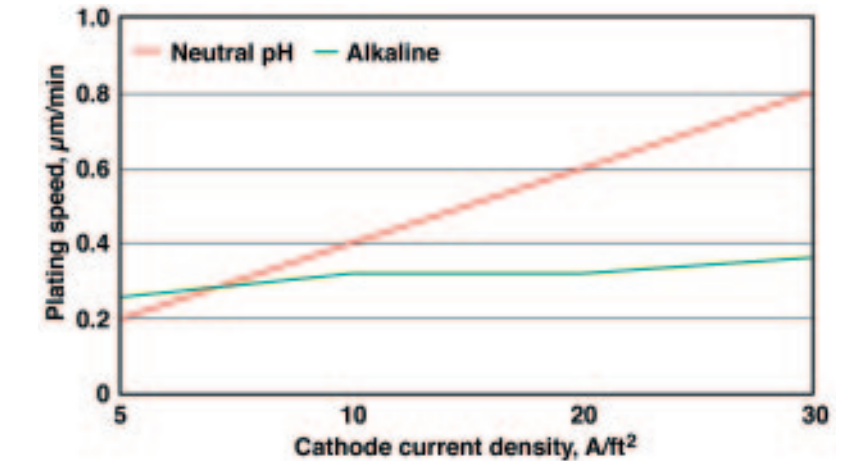


Fig 9—Plating rate of various Sn/Zn alloys.

Table 13
Corrosion Results, Tin/Zinc Process

Sample	White Corrosion	First sign of rust	% Rust
8μ unchromated	48 hr	840	0.5% after 900 hr
8μ chromated	1200 hr	1400	0.5% after 1800 hr

This process has a minimum of 15 g/L of free caustic and preferably 30 g/L to prevent the hydrolysis of tin.

Results from the process development studies described above show that the most significant factors in controlling the alloy composition are concentration of zinc in solution and temperature. Maintaining close control of these two variables, while keeping the other variables in the proper operating window, can ensure proper alloy composition.

Properties of the Coating

Tin/zinc deposits obtained from this process are compact, fine-grained and essentially pore-free. The deposit has a semibright satin finish.

There is significant quantity of corrosion data on tin/zinc coatings. This data is based mainly on the cyanide/stannate chemistry and consists of accelerated test, industrial air SO₂ - CO₂, humidity and neutral salt spray test, and outdoor exposure to different environments.¹³⁻¹⁶

Because there is a large amount of data from these prior works and the data is relevant to rating the corrosion resistance of various coatings compared to tin/zinc alloys, a short summary is needed.

In outdoor exposure, performance of the tin/zinc alloy is environment-dependent. For instance, the tin/zinc alloy performed better than zinc in the jungle and tin at the coast. However, tin/zinc was inferior in the city environment, compared to tin and zinc.

When cross comparison of environmental and accelerated tests are made, different results are observed. Data is available in the accelerated corrosion test comparing cadmium to tin, zinc and tin/zinc.

In the humidity test, zinc has the best results, but in outdoor testing the tin has better results. The tin/zinc coating was the second-best-rated coating in this test.

In neutral salt spray test, unchromated zinc performed the worst. In outdoor

coast tests, however, zinc was the best coating. Cadmium was the best coating in the neutral salt spray test, followed by tin/zinc. In the industrial environment test, the tin/zinc coating was the best compared to tin, zinc and cadmium. But in the

outdoor exposure in the city environment, tin/zinc was the inferior coating.

In summary, there is no direct correlation between the accelerated corrosion test and outdoor corrosion test. The best way to test a new coating is to do a side-by-side comparison in the environment in which the coating will be used.

Salt spray corrosion tests were performed (ASTM B-117)¹⁷ on unpassivated and passivated 8 micron thick coatings from the new non-cyanide tin/zinc alloy. The alloy composition was 80% tin/ 20% zinc, measured by XRF. These results show only a 0.3μ thickness difference and a 3% alloy difference over the whole panel.

Corrosion results of the unchromated and chromated panels are listed in Table 13. This shows chromated sample offers better corrosion protection than the unchromated samples in this test environment. Other reports also indicate this conclusion to.¹⁸

The corrosion results obtained from the non-cyanide electrolyte were comparable to the cyanide/stannate based tin/zinc alloy.

Further work evaluated the static potential and Tafel plots of tin/zinc alloys compared to tin, zinc and cadmium. One reference showed the static potential of various tin/zinc alloys in 2% NaCl solution, but most neutral salt spray corrosion work is done in 5% solution.¹⁹ The potentials also should be measured in 5% NaCl solution. Figure 7 shows the static potential of various tin/zinc alloys in

5% NaCl solution with two distinct potential regions. The first region, between 89-97% tin, is around -750 mv (SCE), which is close to the cadmium potential -730 mv (SCE). The second region is where the alloy is between 65-80% tin, is about -900 mv (SCE), which is approximately 150 mv less than pure zinc 1050 mv (SCE).

Tafel plots run in a 5% static NaCl solution on various alloys and their single metals (Figure 8) show some similar potential to the static potential of the alloys. The tin had a static potential of about -500 mv (SCE) and the Tafel plot showed about the same potential. The 80/20 tin/zinc alloy had about the same potential as the cadmium deposit. The static potential for the 80/20 tin/zinc alloy was -900 (SCE) whereas the potential from the Tafel scan was -800 mv (SCE). The 70/30 tin/zinc alloy had about the same potential as the pure zinc deposit from the Tafel scan, -960 mv (SCE). The static potential for the 70/30 tin/zinc alloy, -920 mv (SCE), however, was much lower than the potential for pure zinc, -1050 mv (SCE).

Table 14 describes the corrosion rates of the various materials in a static 5% NaCl solution. The 80/20 tin/zinc alloy had the lowest corrosion rate and current, followed by tin, than the 70/30 tin/zinc alloy.

Another property of the tin/zinc alloy is its low torque tension, which differentiates tin/zinc alloys from the other zinc and zinc alloys, Zn/Ni, Zn/Co and Zn/Fe.¹⁹ Also, cadmium is solderable, but the zinc and high zinc alloy need a more active flux to activate the surface, whereas the tin/zinc alloy can be activated by a mild flux.

The above work describes an alkaline Sn/Zn process. There is a neutral-to-slightly-acid Sn/Zn process that deposits an alloy composition similar to the alkaline process. Figure 9 shows a comparison of the plating rates of the two different processes.

Table 14
Corrosion Rates in a Static 5% NaCl Solution

Metal	E _(corr) (vs SCE)	I _(corr) ((A/cm ²))	Corrosion Rate (mpy)
Cd	-0.7671	57.17	95.59
Sn	-0.5001	8.867	17.0
Zn	-0.9894	75.18	88.3
80/20 Sn/Zn	-0.8082	6.78	13.0
70/30 Sn/Zn	-0.9656	27.23	57.1

From this work we see the neutral pH Sn/Zn process plates faster in the high current density area, whereas the alkaline process plates more uniform over the entire current density range.²⁰ This result is similar to comparing an acid Zn to an alkaline Zn process.

Summary—Cadmium Substitute

This work describes a tin/zinc alloy as a potential replacement for cadmium electrodeposits. Tin/zinc alloy offers good corrosion protection, solderability and a lubricious coating, which are properties of cadmium. Of all the zinc and high zinc alloys commercially available only the tin/zinc electrodeposit processes most of the attributes of cadmium.

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