

Corrosion Studies Of Metallic Finishes In Electronics

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Abstract

Substrates like brass, copper, steel, and zinc are generally plated with copper, and nickel. Nickel under-layer provides a diffusion barrier. Depending on the application, coatings of Pd, Pd-Ni, Pd-Co, Au-Co, Sn, and Sn-Pb are applied on the surfaces as a final finish. This investigation assessed the corrosion behavior of some of these finishes in neutral sodium chloride and other selected electrolytes. With the aid of Linear Polarization and Tafel plots, corrosion rates in these mediums were estimated. Potentiodynamic polarization curves predict corrosion behavior under extreme oxidizing conditions. Corrosion characteristics and corrosion products were identified by Scanning Electron Microscopy (SEM), and by X-ray Diffraction methods. The corrosion products formed were compared with those formed under exposure to salt spray and mixed flowing gas. Probable mechanisms were postulated based on corrosion products. Electrochemical techniques are shown to provide a quick predictor of the behavior of electrofinishes in a corrosive environment and may be employed in assessing the quality of a given coating without undergoing long and laborious environmental testing.

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Introduction

Substrates like copper alloys, steel and zinc are often used as building blocks for electronic devices and electrical connectors. These substrates are seldom used in "bare form" due to their susceptibility to corrosion when in service. In order to protect the base materials, viable and economical metallic coatings are applied in order to preserve contact integrity and functionality. Many simulated tests such as mixed flowing gases (MFG), thermal cycling, steam aging, chemical porosity testing, and salt spray testing are used in order to predict electroplate behaviors in certain environments. These tests are carried out in order to derive meaningful correlations with the performance of finished products in service conditions. Sometimes, however there are no short cuts and products need to be exposed to actual environments of real service conditions for extended periods in order to corroborate the results of accelerated corrosion tests. In this work, attempts have been made to utilize electrochemical techniques to derive meaningful information pertaining to the corrosion behaviors of some commonly used finishes in selected electrolytes. Results in sodium chloride spray test and MFG Class II, and III are also recorded for comparative purposes. These initial evaluations can provide useful information in selection of potential surface finishes of electrical components.

Experimental

Copper alloy phosphor bronze 511 circular coupons (1" diameter, 0.015" thick) with total surface area of 0.01 sq. ft were degreased with acetone, methanol and cathodically cleaned in an alkaline cleaner. These samples were activated in 10 % H₂ SO₄ and plated with 50 µin matte nickel in a standard nickel sulfamate bath. Surface coatings (30 µin in thickness) of Pd-Ni, and Pd-Co were then applied using commercially available plating chemistries and alloy compositions controlled nominally 80 wt % Pd -20 wt % Ni or Co. Cobalt- hardened gold was deposited to a plating thickness of 4 µin on selected samples.

Stainless steel 302 coupons of 1"x1" dimensions were also degreased in acetone, and methanol, cathodically electrocleaned in an alkaline cleaner, and activated in dilute HCl. The coupons were then plated with a flash of Watts nickel, followed by nickel deposit of 50 µin from a sulfamate bath and a final finish of hard gold. As an alternate approach to achieve the direct gold deposit on stainless steel, a gold flash was applied from a highly acidic gold (III) bath followed by 30 µin of hard gold deposit. Both finishing schemes provided good adhesion. The

corrosion behavior of these coatings was compared in selected electrolytes.

Corrosion potential and electrochemical polarization curves were obtained using a microprocessor controlled, Potentiostat/Galvanostat apparatus in conjunction with a flat cell equipped with a three-electrode system. All applied potentials were recorded vs. a Ag/AgCl reference electrode. The test solutions were prepared from analytical grade reagents. The electrolytes tested were not de-aerated and all electrochemical tests were carried out in a glass cell at 25 °C as per ASTM methods¹⁻⁴. Cleaned test coupons were mounted in the flat cell (effective exposed area = 1 cm²), immersed in the solution for 15-20 minutes until a steady state corrosion potential (E_{corr}) was obtained. On obtaining the steady state condition polarization was initiated at -250 mV relative to E_{corr}, followed by a forward scan towards the noble direction at a rate of 1 mV/sec. From the knowledge of corrosion currents, and Tafel slopes⁵, the corrosion rates⁶⁻⁸ were calculated from equations (1) and (2).

$$I_{CORR} = \frac{\beta_a \beta_c}{2.3(\beta_a + \beta_c)} \times \frac{\Delta E}{\Delta E} \quad (1)$$

$$Corr. Rate(mpy) = 0.13 \times I_{CORR} \times \frac{Eq. Wt.}{\rho} \quad (2)$$

In equation (1) and (2), where β_a and β_c are Tafel slopes of anodic and cathodic polarization curves and ρ is the alloy density (g/cc).

As a basis for comparison, salt spray testing³ in neutral 5 % sodium chloride was also performed. Exposure times ranged from 24-72 hours depending on the extent of attack.

Test coupons were also subjected to 6 days exposure to Class II and Class III MFG testing. In summary, the electroplated test coupons were subjected to the following tests and their results are discussed in the succeeding sections.

1. Electrochemical Tests (E_{corr}, Tafel Plots, and Potentiodynamic Anodic Polarization Plots)
2. Salt Spray Testing
3. MFG Class II and Class III exposures

Results and Discussion

Electrochemical Tests

A. Corrosion in Oxidizing Media

1. Sodium Hypochlorite

Table 1 summarizes the electrochemical corrosion data on indicated finishes in 5% NaOCl 'bleach' solution. Nickel and phosphor bronze corroded considerably. Corrosion rate for gold was the least whereas the Pd, Pd-Ni, Pd-Co corrosion rates ranged between 6-8 mpy. In presence of gold flash, the corrosion potentials tend to shift anodically. The gold flashed Pd-Ni and Pd-Co exhibited lower corrosion rates than Pd-Ni and Pd-Co finishes. Even Pd metal corroded in this environment. Figure 1 compares potentiodynamic response of Pd-Ni/Auf and Pd-Co/Auf in 5 % sodium hypochlorite solution.

TABLE 1

Corrosion behavior in 5% Hypochlorite solution

Substrate	E _{corr.} Vs. Ag/AgCl Volts	I _{corr.} $\mu\text{a}/\text{cm}^2$	Corrosion Rate, mpy
Phosphor Bronze	0.96	84	39
Pd Metal	0.97	17.2	10.6
Au Metal	0.95	1.0	1.3
Ni Plate	0.72	136	58
Pd Plate	0.82	9.8	5.6
Pd-Ni	0.76	13.1	8.1
Pd-Ni/Auf	0.94	3.8	5.5
Pd-Co	0.69	12.6	7.8
Pd-Co/Auf	0.97	2.8	4.1

The application of gold flash on Pd-Ni, and Pd-Co tends to shift E_{corr.} to more anodic values and the role of galvanic corrosion due to coupling of gold with semi-noble finishes of Pd-Ni and Pd-Co should be considered. Table 2 shows results on some selected galvanic couples in 5 % sodium hypochlorite.

TABLE 2

Galvanic Corrosion in 5% Sodium Hypochlorite

Substrate	E _{corr.} Vs. Ag/AgCl Volts	I _{corr.} $\mu\text{a}/\text{cm}^2$	Corrosion Rate, mpy
Pd-Co vs. Au-Co	0.99	3.8	2.4
Pd-Ni vs. Au-Co	0.62	4.0	2.5
Pd vs. Au-Co	0.94	9.0	5.5
Ni vs. Au-Co	0.67	30.0	12.7

Pd-Ni and Pd-Co finishes vs. hard gold exhibited corrosion currents of smaller magnitude which suggests the presence of modest galvanic corrosion. Pd is more prone to hypochlorite attack which is shown by Pd Vs. Au-Co pair. Ni vs. Au-Co combination exhibited the highest corrosion rates. However, in practice, it is imperative to use hard gold coatings as the final flash layer in order to provide low, stable contact resistances with increased wear resistance.

The overall corrosion currents exhibited by a coating is governed by the sum total of both anodic and cathodic currents and also due to the contribution of galvanic currents produced by contact of two dissimilar metals. These coatings are prone to more corrosive attack due to presence of several chemical species, such as Cl₂, Cl₂O, O, NaCl, NaOH, etc. present in a bleach solution. The presence of moist chlorine and OCl⁻ ions tend to enhance the corrosive action⁹. The hypochlorite ions in bleach solution also tend to increase in chloride ions due to the following reaction:



We observe the cumulative effect of these corrodants and in particular chlorine and chloride ions which are highly corrosive in a humid environment. This was confirmed by immersing the coupons in quiescent solutions for 1 hour and results are summarized in Table 3. The tendency to corrode was judged qualitatively in this experiment and expressed in terms of corrosion severity. Rank I indicates the least attack and rank IV denotes the severest attack.

TABLE 3
Immersion Test in Bleach

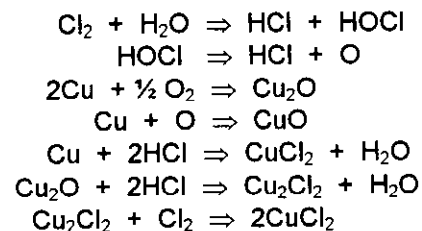
Substrate	General Remarks	Corrosion Severity Ranking
Phosphor Bronze (P.B.)	No visible surface effects	III
P.B./Ni	General attack, green solution black particles	III
P.B./Ni/Pd	Some black particles at edges	I
P.B./Ni/Pd-Ni	Black edges with particles	II
P.B./Ni/Pd-Ni/Auf(4 μ in.)	Dark black solution	IV
P.B./Ni/Pd-Ni/Au-Co (30 μ in.)	Less attack, some attack on edges	I

Another experiment was also performed by exposing hard gold plated coupons of varying thicknesses of 15, 40 and 60 μ inches in 5% sodium hypochlorite solution for six hours at ambient temperature. The surface attack was thickness dependent and was the least for a thick gold coating. Identification of corrosion elements by SEM-X rays indicated presence of Cu, Ni, Cl and Sn suggesting that pore corrosion occurred at thin gold coating and reached the phosphor bronze material. X-ray diffraction indicated presence of CuCl (Nantokite) and Cu₂(OH)₃Cl (Paratacamite).

Corrosion in Bleach Vapors

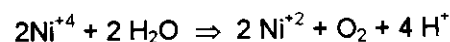
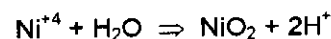
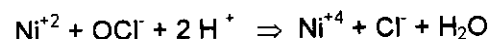
The effect of exposure to bleach vapors for selected finishes was studied. The surface characteristics and the corrosion response of Pd-Ni and Pd-Ni/Auf and Pd-Co and Pd-Co/Auf is shown in figure 2. Gross pore corrosion with mounds of corrosion products was noticed in all finishes of Pd-Ni and Pd-Co. The corrosion appeared to initiate in the pore and then mushroomed outward. The major corrosion products were NiCl₂ and CuCl₂. Presence of gold flash did not alleviate the situation both for Pd-Ni and Pd-Co. Phosphor bronze basis metal and nickel plated coupon showed some pitting though to a

lesser extent (Fig. 3). Bleach vapors emanate chlorine gas and following simplified mechanism based on disproportionation is hypothesized in presence of moist chlorine.



Similar reasoning can be given for nickel corrosion.

Sodium hypochlorite is a strong oxidizing agent and is widely used as a bleaching agent in wood and pulp industry. Metals tend to oxidize in its presence and thus metal ions formed from Ni, Co, Cu catalytically decompose hypochlorite according to the following probable mechanism^{9, 10}.



Nickel is attacked by hypochlorite solutions with local pits. Many metals including noble metals like Gold and Pd are also known to be attacked by moist chlorine vapors¹¹. This has been substantiated by above experiments.

2. Peracetic Acid

The steady state corrosion potentials, E_{corr}, the corrosion current, I_{corr}, and corrosion rates in mils-per-year (mpy) for series of coatings are summarized in Tables 4 and 5 for 0.2% and 5% PAA solutions respectively. As expected, phosphor bronze shows a high corrosion rate in 0.2% PAA. Most finishes exhibited an order of magnitude decrease in corrosion rate relative to bare phosphor bronze. Pd metal as well as Pd plated coupons exhibited high corrosion rates whereas Au metal corroded the least. Essentially, unplated phosphor bronze is severely attacked by PAA but all other noble and semi-noble coatings seem relatively innocuous to PAA attack at 0.2 % levels.

Increase in PAA concentration to 5% showed a significant elevation in corrosion rates of all samples and similar trends were observed as in 0.2%

PAA. Further, the presence of flash gold or thick gold on Pd-Ni; significantly retarded the corrosion (more anodic E_{corr}) in PAA. It is apparent that at both concentrations of PAA (0.2% & 5.0%) Pd-Ni and Pd-Co exhibit some tendency of corrosion. High corrosion rate for Pd plated coupon may be due to micro-cracks.

Table 4
Corrosion Studies in 0.2% Peracetic Acid

Finish	E_{corr} (vs. Ag / AgCl, V)	I_{corr} , $\mu A/cm^2$	Corrosion Rate, mpy
Ni	0.36	0.2	0.08
Pd Metal	0.78	0.08	43.9
Pd/GF	0.63	1.2	0.68
Pd-Ni	0.60	9.9	6.13
Pd-Ni/GF	0.59	0.4	0.22
Pd-Co	0.60	21.4	13.17
Pd-Co/GF	0.68	2.8	1.71
Au-Co	0.64	4.2	6.12
Ph Bronze	0.40	1740.0	792.7
Au Metal	0.91	0.003	1.55
Pd Plated	0.76	0.113	64.6

TABLE 5
Corrosion Studies in 5% Peracetic Acid

Finish	E_{corr} (vs. Ag / AgCl, V)	I_{corr} , $\mu A/cm^2$	Corrosion Rate, mpy
Ni	0.90	0.8	0.33
Pd Metal	0.76	0.72	413.2
Pd/GF	1.00	1.7	2.46
Pd-Ni	1.01	301.9	186.2
Pd-Ni/GF	1.14	2.6	3.8
Pd-Co	1.06	579.1	357.2
Pd-Co/GF	1.15	5.6	8.2
Au-Co	1.11	8.3	12.1
Ph. Bronze	0.68	6.3	2900
Pd Plated	0.83	3.34	1905

B. Studies in 5% Neutral Sodium Chloride

Electrochemical Experiments

Electrochemical corrosion parameters were obtained for indicated finishes in neutral 5% sodium

chloride solutions (Table 6). Stainless steel 302, Ni 200, Pd, Pd-Ni, Pd-Co finishes all showed minimum corrosion rates (< 0.5 mpy) which is remarkably low in a salt laden environment. Phosphor bronze coupons indicated a measurable corrosion rate. The attack on other finishes was minimal. A separate set of these coupons was also subjected to salt spray test which conforms to MIL Std. 883d. As presented below, no significant corrosion was noticed on these coupons except some discolorations or a greenish look at the edges which is due to some bare spots of phosphor bronze. These results are in direct concurrence with our findings from the electro-chemical experiments. These results strongly suggest that potentiodynamic polarization method serves as a powerful predictor for rapid corrosion assessment if a chemical environment can be simulated.

Polarization plots of Ph. Bronze, Pd, Ni, Co, and Au metals in 5 % neutral NaCl are compared in figure 4. Phosphor bronze displayed a hump indicating active behavior followed by a passive region. The anodic curves were not symmetrical in Tafel region in most cases. Palladium metal exhibited active passive behavior.

TABLE 6
Electrochemical Corrosion in neutral 5% NaCl

Substrate	E_{corr} vs. Ag/AgCl	I_{corr} , $\mu A/cm^2$	Corrosion Rate, mpy
Ph.Bronze	-0.10	20.8	9.5
SS302	-0.16	0.4	0.2
SS302/Ni	-0.23	0.2	0.1
SS302/Ni/Au-Co	-0.09	0.1	0.0
Ni 200	0.16	0.9	0.4
P.B./Matte Ni	0.30	0.6	0.3
Ni/Au-Co	0.53	0.3	0.4
Pd	0.45	0.5	0.3
Pd/Auf	0.29	0.5	0.3
Pd-Ni/Auf	0.32	0.4	0.2
Pd-Co	0.45	0.2	0.1
Pd-Co/Auf	0.25	0.1	0.1
Au Metal	0.77	0.2	0.8
Pd Metal	0.59	0.3	0.2

Salt Spray Tests

Salt spray tests for 72 hours were conducted on test coupons plated with Pd, Pd-Ni, Pd-Co of varying alloy composition (80/20, 70/30, 60/40) also these finishes

with gold flash. Nickel plated coupons showed some pits and a green residue. Samples with Pd, Pd/Au, hard gold exhibited some discolorations. However, Pd-Ni and Pd-Co finishes with and without gold flash did not show any visual corrosion except sporadic discolorations. This qualitative behavior is also supported by electrochemical experiments listed above which indicate insignificant corrosion except phosphor bronze. Gross corrosion was noticed for a Pd-Co coupon of 50/50 composition.

These studies show conclusively that Pd-Ni and Pd-Co finishes with and without gold flash were innocuous to salt corrosion as indicated by salt spray and electrochemical tests.

C. Other Electrolytes

Corrosion behavior for Pd-Ni and Pd-Co (80-20) composition in presence of common mineral acids (HCl, HNO₃, H₂SO₄ and their mixtures) and some selected electrolytes is summarized in Table 7. Sulfuric acid solution appeared corrosive for Pd-Ni and Pd-Co finishes in particular. The corrosion rates for Pd-Co seem to be relatively higher than those for Pd-Ni in most electrolytes except NaCl under identical conditions. The coatings tend to dissolve anodically under extreme oxidizing potentials ($\approx 1.0 - 1.5$ volts). However, under normal circumstances, such conditions do not exist except when subjected to potentiodynamic anodic polarization scans.

TABLE 7
Electrochemical Corrosion Behavior of Pd-Ni (80-20) and Pd-Co (80-20) in Selected Electrolytes

Electrolyte	Pd-Ni (80-20)			Pd-Co (80-20)		
	E_{corr} Vs. Ag/Ag Cl Volts	I_{corr} $\mu A/cm^2$	Corrosion Rate (mpy)	E_{corr} Vs. Ag/AgCl Volts	I_{corr} $\mu A/cm^2$	Corrosion Rate (mpy)
1 N H ₂ SO ₄	0.61	0.4	0.3	0.70	5.0	3.5
1 N HCl	0.48	0.6	0.4	0.30	4.1	2.5
1 N HNO ₃	0.61	0.3	0.2	0.75	2.6	1.6
1 N H ₂ SO ₃	0.43	3.6	2.2	0.38	5.5	3.4
1 N HCl/HNO ₃ / H ₂ SO ₄	0.54	0.4	0.2	0.66	2.0	1.2
1 M NaOH	0.32	7.3	4.5	0.39	1.8	1.1
1 M NaCl	0.44	0.4	0.2	0.24	0.2	0.1
5% NaCl	0.48	0.4	0.2	0.45	0.2	0.1
1 M (NH ₄) ₂ SO ₄	0.58	0.3	0.2	0.51	3.3	2.0

A few typical plots for some of these electrolytes are shown in figures 5-7. Ammonium sulfate tends to

hydrolyze yielding sulfuric acid and probably is the cause for higher corrosion rate in case of Pd-Co. Pd-Ni exhibited active passive behavior with a transpassive region in 1N H₂SO₄ whereas Pd-Co indicated higher corrosion current followed by passive region. Pd-Ni coating corroded faster in NaOH.

D. Mixed Flowing Gases

Some of these finishes were exposed to a mixture of gases class II and class III for six days. The conditions for class II and III MFG chamber are listed in table 8.

Table 8
Mixed Flowing Gas Conditions

Parameter	Class II	Class III
Cl ₂	10 ppb	20 ppb
H ₂ S	10 ppb	100 ppb
NO ₂	200 ppb	200 ppb
RH, %	70	75
T, °C	30	30

These corrosive gases tend to react with the exposed metal. The corrosion products may be due to reaction of the environment and the surface finish or reaction with the underlayer base material which is exposed during the porosity tests. It must be noted that porosity evaluations was not the main objective of this study. In general, pore corrosion was noticed in most of these samples and the extent of corrosion seemed to be dependent upon coating thickness and its intrinsic porosity. Typical pictures depict the pore corrosion behavior for Pd-Ni and Pd-Co (80-20) in figures 9,10. These figures indicate gross pore corrosion and general attack increased immensely in Pd-Co (50-50) composition (figure 8). Class III appeared to be fairly more aggressive than Class II in which comparatively less corrosion was noticed. A thin discolored film without any pits was noticed on finishes after 6 days exposure to class II. This was evident from these studies that no coating is pore-less and suggested standard methods should be followed for assessing the porosity of coatings. Noble metals and semi-noble alloys are applied as viable coatings to offer resistance to corrodants. Ideally, a noble metal plating should be pore free but intrinsic porosity is always present on platings of normal thickness. The gas tests are better than liquid immersions. The gases Cl₂, NO₂, H₂S or SO₂ in presence of water vapors 'humidity' may produce HCl, HNO₃ and H₂SO₄ in situ at the pore site due to high permeability of gases in the pores and start galvanic corrosion

reactions. The mechanism is explicable based on electrochemical principles but it seems difficult to simulate this system in liquid solutions. Thus a semi-noble plating with porous texture can contain concentrated surface solutions which form a path of high electrical conductivity between the finish and the base metal. This creates an electrolytic cell with oxygen or other pollutants being reduced at the noble surface and base metal or underplate being oxidized. The porous noble finish acts as large cathode and the corrosion attack enhances at small anodic areas leading to formation of corrosion products in pores and their exudation on to surface forming mounds, halos, and films. Thus, simulation for corroding media, their components concentration and relative corrosion rate are difficult to re-enact for practical correlations with MFG tests.

Conclusions

1. Potentiodynamic polarization plots can be employed for prediction of estimated corrosion rates from the knowledge of corrosion currents and Tafel slopes in simulated electrically conducting aqueous solutions. The results of various finishes such as Pd, Pd-Ni, Pd-Co and their counterparts with gold flash showed negligible corrosion. This was supported by the salt spray data.
2. Studies in presence of oxidizing agents, sodium hypochlorite and peracetic acid, indicated that bleach solution was extremely corrosive to the coatings possible due to available chlorine and nascent oxygen. Peracetic acid showed increased corrosion at 5% levels than 0.2 %.
3. Mixed flowing gases class III was more corrosive than class II and pore corrosion was indicated in most of these coatings. Limitations of the electrochemical method were discussed with special reference to simulation and correlation of these methods.

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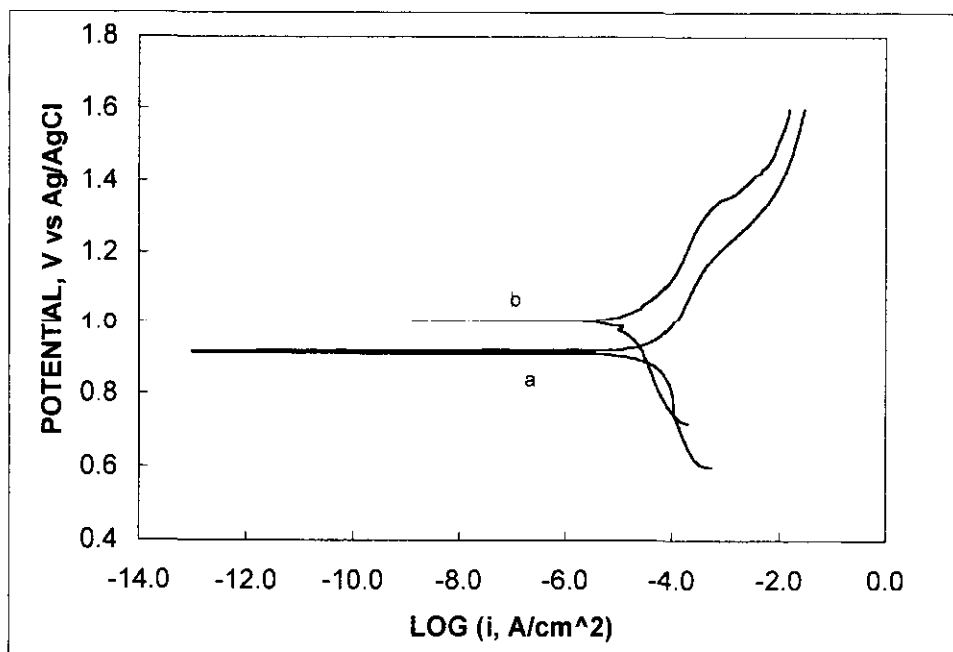
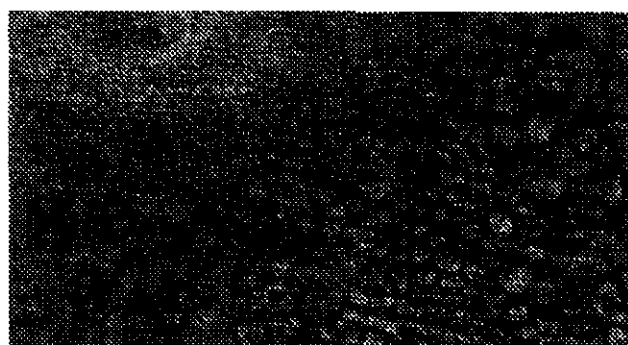
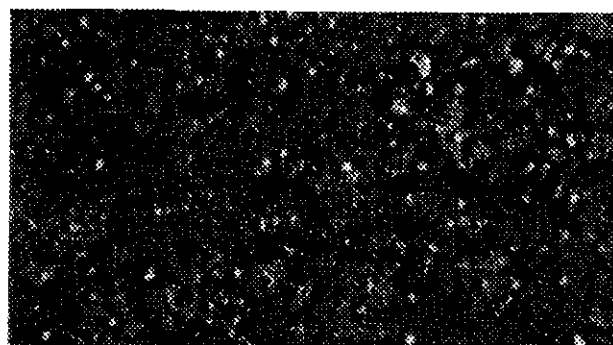


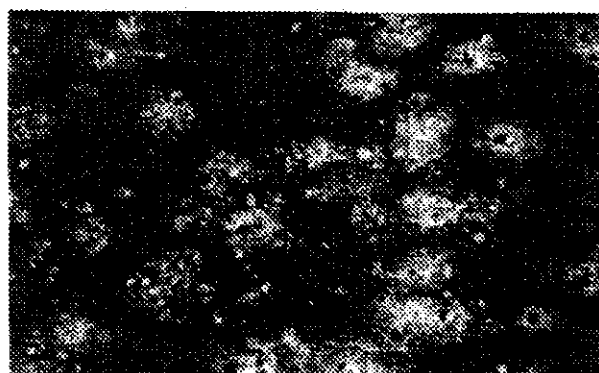
Figure 1 Potentiodynamic polarization plot in 5% sodium hypochlorite solution, (a) Pd-Ni/Auf (b) Pd-Co/Auf.



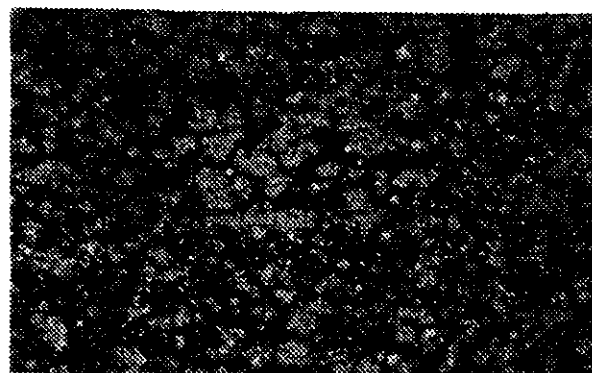
A. Pd-Co



B. Pd-Co/ Au flash

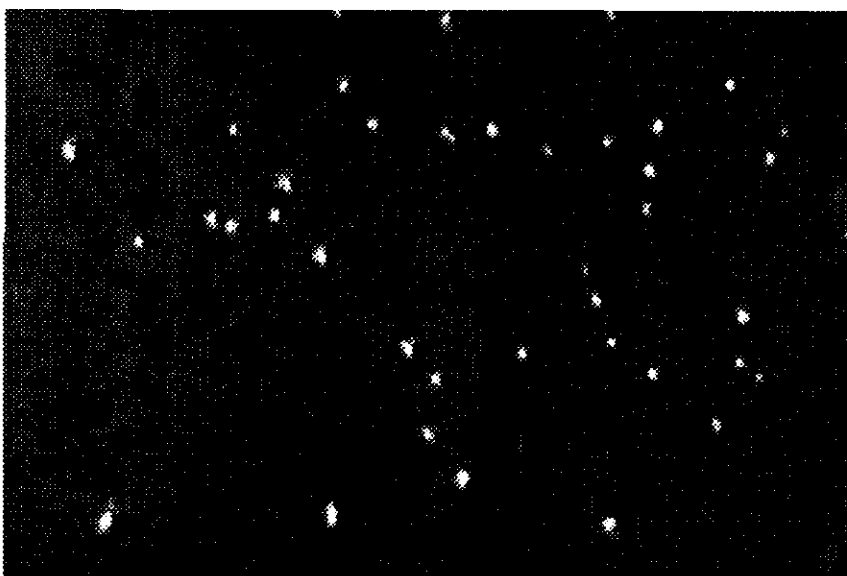


C. Pd-Ni



D. Pd-Ni/ Au flash

Figure 2 After exposure to bleach vapor for 7 days



E. Phos Bronze



F. ~ Ni Plated Phos Bronze

Figure 3 After exposure to bleach vapor for 7 days

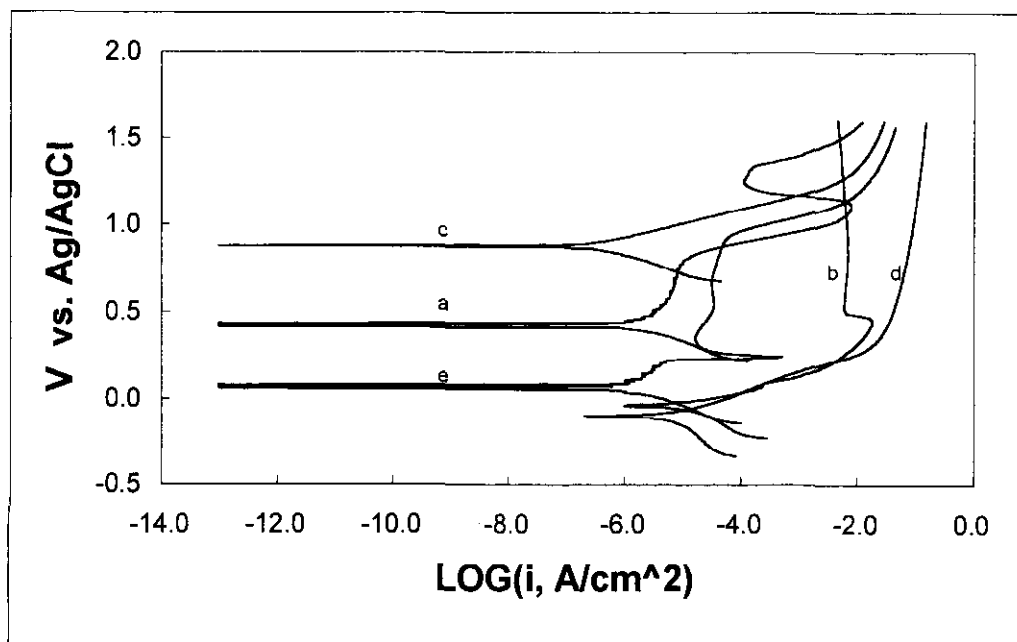


Figure 4 Potentiodynamic polarization plots of (a) Pd, (b) P.B., (c) Ni, (d) Co, and (e) Au in 5% neutral sodium chloride.

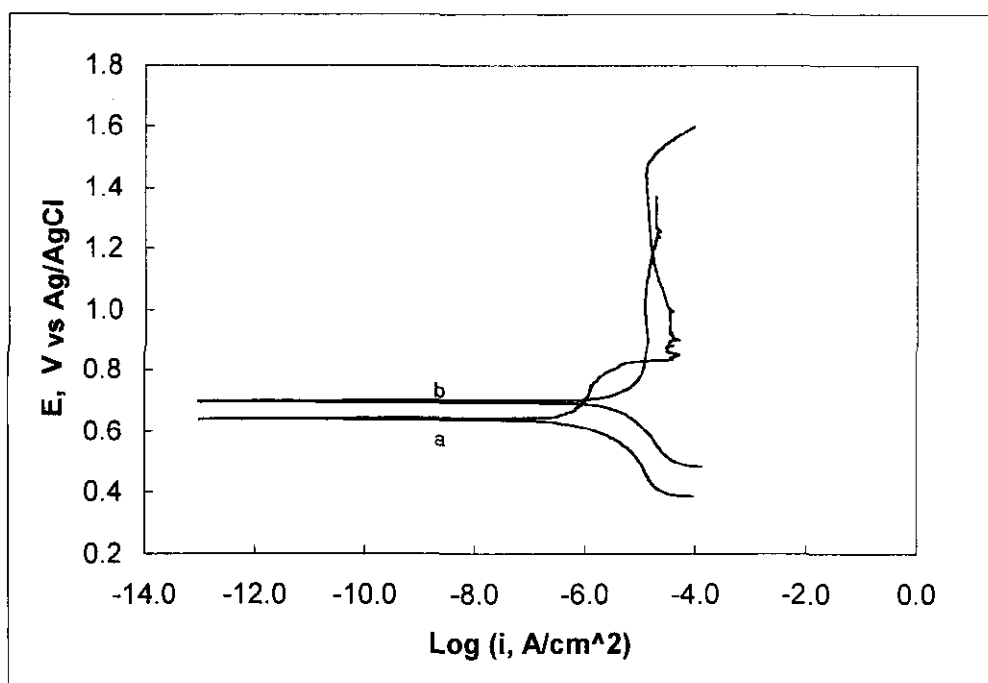


Figure 5 Potentiodynamic polarization plots in 1N H₂SO₄. (a) Pd-Ni and (b) Pd-Co

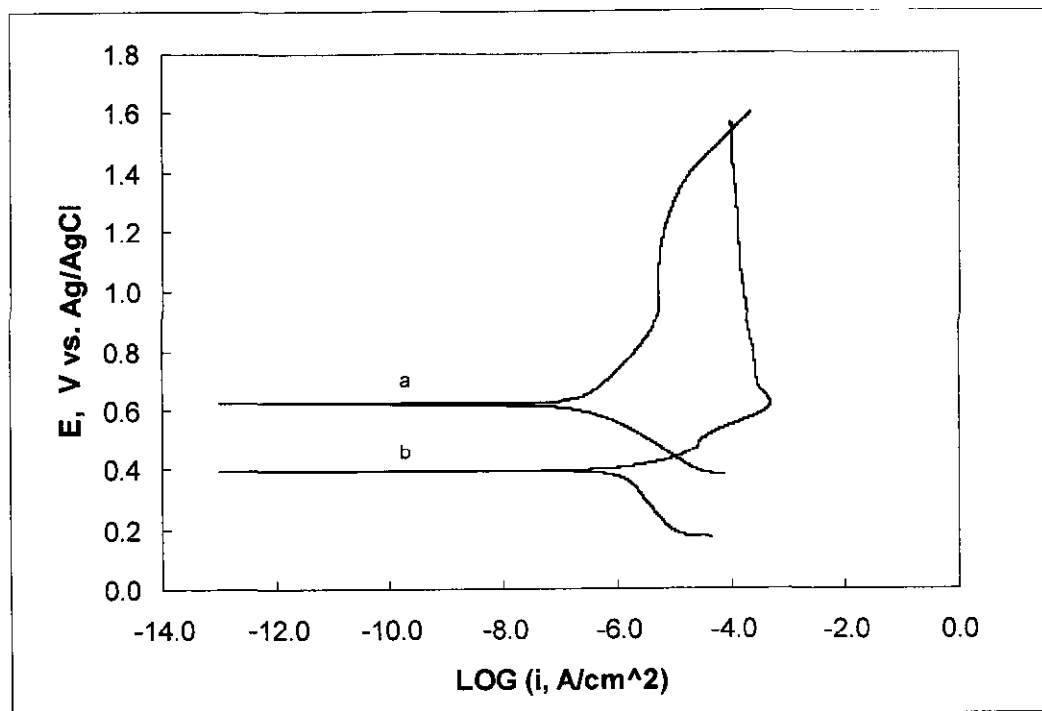


Figure 6 Potentiodynamic polarization plots of (a) Pd-Ni and (b) Pd-Co in 1M $(\text{NH}_4)_2\text{SO}_4$.

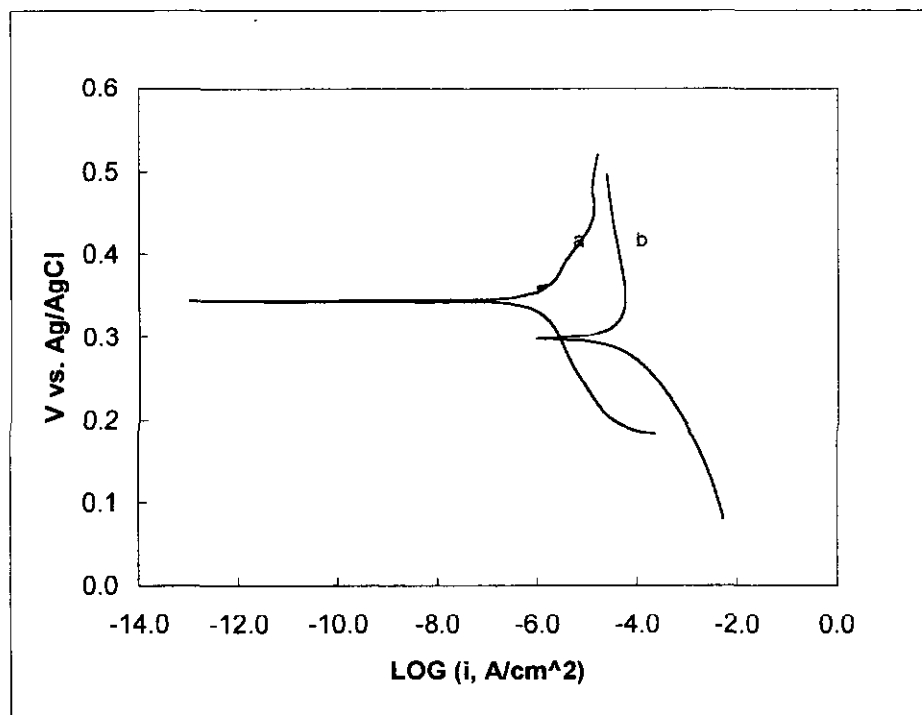
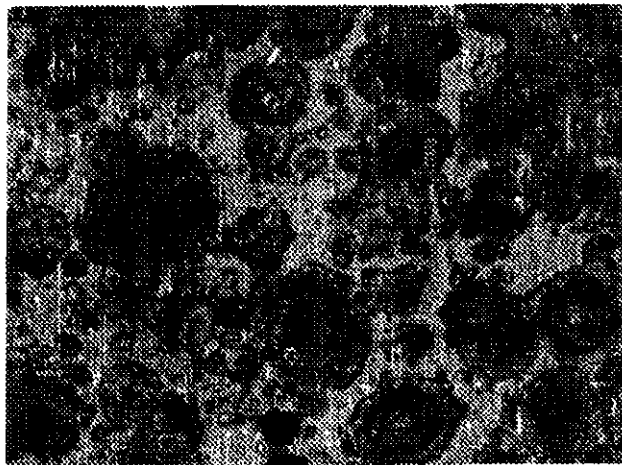


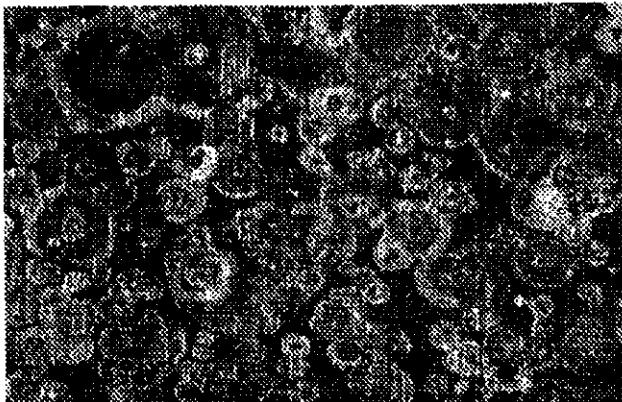
Figure 7 Tafel plots in 1N H_2SO_3 solution. (a) Pd-Ni and (b) Pd-Co



Pd-Ni (80-20)



Pd-Co (80-20)



Pd-Co (50-50)

Figure 8 Class III MFG exposure for 6 days