The Electrodeposition & Material Properties Of Palladium-Cobalt

By J.A. Abys, G.F. Breck, H.K. Straschil, I. Boguslavsky & G. Holmbom

Palladium and its alloys have become the standard precious-metal finishes for high-reliability, high-durability connectors. While pure palladium is preferred for high-temperature automotive applications, palladium-nickel has become the preferred finish for ambient temperature, high-insertion applications, such as edge card connectors. Palladium-nickel, when plated over nickel, is not without its problems, however. Quality control issues related to the measurement of composition and thickness by simple, non-destructive XRF analysis remain a significant concern. Palladium-cobalt does not suffer from this shortcoming and has been found to out-perform palladium-nickel for high-durability applications.

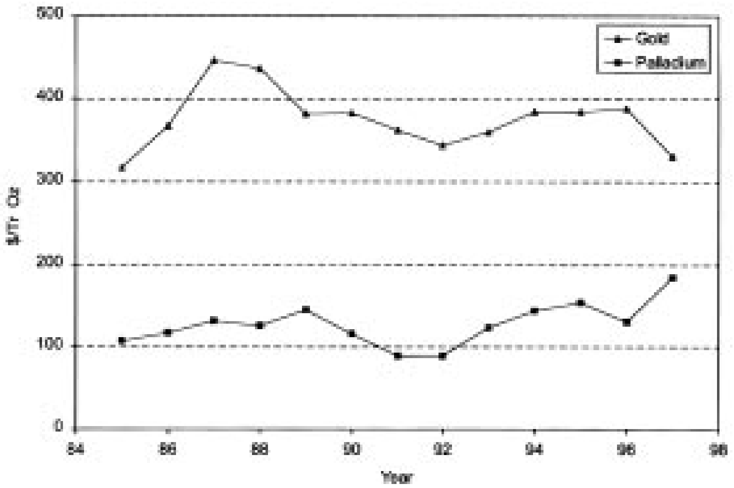

Fig. 1—Gold and Pd price in dollars per Troy oz., 1985-1997.

The technological and economic advantages of substituting palladium (Pd) and Pd alloys for Hard-Gold are generally recognized.[1-3] Historically, the lower price of Pd (Fig. 1), coupled with its lower density (Table 1) resulted in substantial cost reductions. Figure 2 demonstrates that currently there exists a greater than $100 per troy oz. differential for plated palladium-nickel (Pd-Ni) vs. Hard-Gold. Consequently, Pd and its alloys remain competitive finishes for high-reliability applications. More importantly, the increasing use of Pd-based finishes is driven by stringent technological requirements not readily met by Hard-Gold.[3-13]

Technologically, the material properties of Pd are in many instances superior to Hard-Gold.[3-24] For example, the greater hardness of Pd is beneficial for wear resistance,[6] which can be further enhanced by a thin coating of Au[25] and the application of synthetic lubricants.[25,26] The arguments for the use of Pd as a contact material are, in some cases, more valid for Pd-alloys that exhibit superior material properties. For example, suitable amounts of nickel (20 wt pct), co-deposited with Pd, have produced an alloy (Pd-Ni) that is brighter, harder and more ductile,[27] and which exhibits lower porosity[8,9,12] and better wear resistance than Hard-Gold.[7,29] It is no surprise, then, that Pd-Ni attracted considerable interest in the electronics industry.

The disadvantages of Pd-Ni as a contact finish are associated with electrodeposition of this alloy on a nickel sublayer, and the ability to measure composition and thickness in a manufacturing environment by X-ray fluorescence (XRF). Therefore, it is primarily an issue of *quality control*. Moreover, it has been found by Kudrak[10] *et al*. that Pd-Ni is thermally stable only up to ~125 °C, making it unreliable for high-temperature automotive applications. Finally, nickel has been classified as an environmental hazard because of “nickel contact dermatitis.”[30] There was a need, therefore, to develop an alloy that would exhibit the positive attributes of Pd-Ni, yet overcome its deficiencies.

Palladium-cobalt (20 wt pct Co) is such an alloy,[31-35] (Fig. 3). In addition, it possesses greater hardness[31] than Pd-Ni, which implies superior wear durability.[32-34]

This paper describes the development of a high-speed process for electroplating Pd-Co alloys. Throughout this paper the percent metal content will represent a weight percent.

Electrodeposition Chemistry

The objective of this research was to develop a robust plating chemistry that consistently produces the desired material properties over a wide range of operating conditions and bath aging. In this regard, a brief review of Pd-Ni chemistry may be useful prior to discussing Pd-Co.

Background Information

While acidic Pd-Ni electroplating baths have been proposed, most processes appear to be ammoniacal solutions in the pH

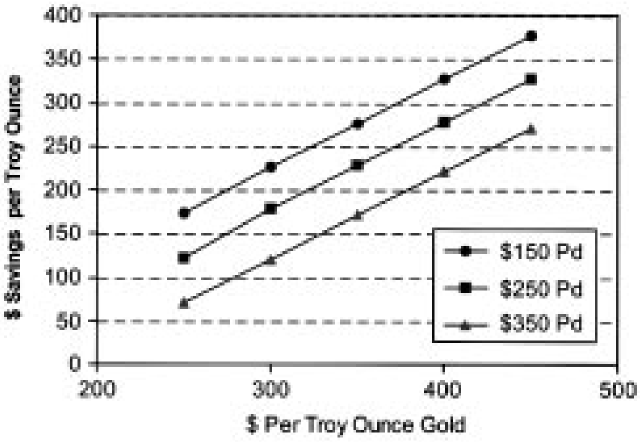

Fig. 2—Cost in dollars per Troy oz., gold vs. Pd-Ni.

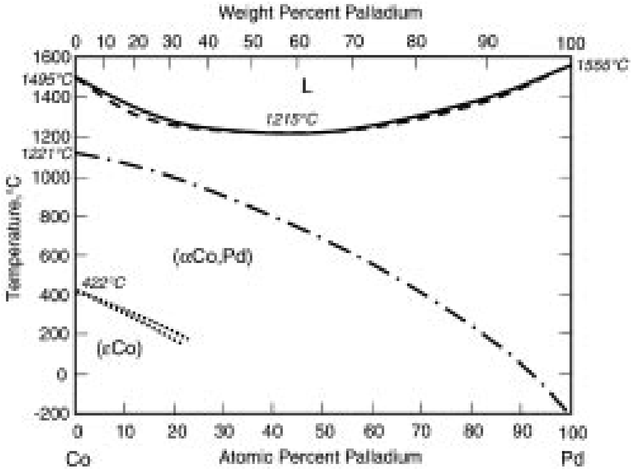

Fig. 3—Co-Pd phase diagram.

range from 7 to 9. Recommended metal sources for Pd are its complexes with ammonia or organic amines; for nickel, the chloride, hydroxide and sulfamate. A Pd-Ni plating bath listed by R.J. Morrissey[29] contains 6 g/L $Pd(NH_3)_2(NO_2)_2$, 3 g/L nickel sulfamate, 90 g/L ammonium, pH 8-9. This bath operates at 20 to 40 °C at current densities between 5 and 10 mA/cm^2 and is suitable for plating an alloy of about 25 percent Ni. Other chemistries use plating temperatures up to 60 °C in the pH range of 7 to 9, metal concentrations up to 25 g/L Pd and 30 g/L Ni, and as many as four proprietary additives.

Such processes exhibit a number of disadvantages in their operation. Perhaps the most troublesome and frequent complaint is the lack of pH stability, which results in undesirable fluctuations in the alloy composition. Another concern is the environmental and occupational hazards connected with ammonia vapors, especially in high-speed operations where the pH, temperature and solution agitation tend to be high, thus favoring the release of ammonia. Additionally, the loss of deposit brightness with bath aging can shorten the useful life of the bath. Last but not least, highly stressed and cracked deposits are also encountered, especially in high-speed operations.

Recently, Boguslavsky *et al.*[37] reported on an environmentally “friendly” process for plating Pd-Ni that overcomes these deficiencies and provides excellent composition control as a function of operating parameters. This particular formulation provided the basis for the development of the new Pd-Co process.

Bath Formulation

The bath make-up depends on the desired alloy composition as well as on the intended operation: Barrel, rack or continuous reel-to-reel plating. Concentration ranges for the chemical constituents for high-speed and low-speed plating are seen in Table 2; intermediate bath compositions can be used for rack operations. This paper will be restricted to an analysis of high-speed deposition.

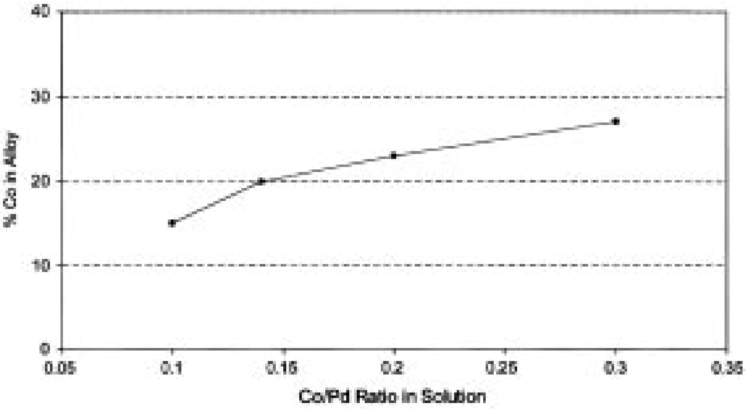

Fig. 4—Wt pct Co in deposit vs. Co-Pd (wt) in solution (High-Speed Process).

Table 1
General Material Properties of Pd-Co

Comparison with Hard-Gold & Pd-Ni

Materials Properties	**Hard Au**	**Pd***	**Pd-Ni* 20 wt %**	**Pd-Co* 20 wt %**
Grain size, Å	200-250	60-2000	50-220	50-150
Density, g/cm^3	17.3	11.7	10.7	10.8
Thermal stability, °C	150	>450	380	395
Hardness, KHN_{25}	140-200	150-500	450-550	590-640
Ductility (E% @ 2.5 µm)	<3	3 to >10	3 to >10	3-7
Average porosity index				
Connector pins (1.5/2.5 µm Ni)	0.5	—	0.11	0.02
Lab coupons (1 µm GFPdX/Cu)	3.5	0.7	0.4	0.3
Wear Properties (load: 100 g)				
Cycles to failure (x 1K)	20	>80**	35	>80
Coeff. fric. @ 10K cycles	0.60	0.31	0.55	0.43
Relative cost (Pd/Au) (a) Gold, $300/Troy oz. (b) Palladium, $280/Troy oz.	1.00	0.63	0.46	0.47

The alloy composition is a function of metal concentrations, pH, current density, temperature, and solution agitation, as discussed below. One of the most critical variables, however, is the relative metal (Co to Pd ratio) concentration of the bath. Within the specified ranges, the percentage of cobalt in the deposit is linearly correlated to the Co/Pd ratio in solution, as shown in Fig. 4. This is important because it indicates that desired alloy(s) in the composition range of 10-30 percent Co are attainable by changing the Co/Pd ratio in solution.

Operating Conditions

Temperature & pH, Effect on Alloy Composition

The ability to electrodeposit Pd-Co alloys of desired composition (10 to 30 percent Co) from a single chemistry by modifying the Co/Pd ratio in solution is an important feature; however, just as critical is the ability to un-

Table 2
Concentration Ranges for Chemicals Used

	High Speed		Low Speed	
Components	**Typical**	**Range**	**Typical**	**Range**
Pd as metal, g/L	40	35-45	4	3-4
Co as metal, g/L	8	6-10	1.5	1.25-1.75
Conducting salt, g/L	40	30-35	40	40-60
Surfactant, mL/L	20	15-25	5	3-20
Brightener, mL/L	10	5-20	5	5-10

Table 3
Plating Conditions Used

Plating conditions	**Typical**	**Range**
Temp, °C	55	45-60
pH	7.5	7.0-8.0
Current density, A/ft^2	300	100-700
Agitation, m/sec	2	1.5-3
Anode:cathode ratio	1:1	15-25
Plating rate, µin./sec; 100 A/ft^2	10	5-20

derstand how variations in the operating parameters affect the alloy composition.

The influence of temperature on the Co content can be seen in Fig. 5, where a slight dependence is observed. At 300 mA/cm^2, the Co varies from ~16 percent at 45 °C to 22 percent at 65 °C. Likewise, at 500 mA/cm^2, the Co varies from ~18 to 23 percent when the temperature is varied from 45 to 65 °C. Because temperature is relatively easy to maintain within ±2 °C, the slight variation over a 20-degree change poses no quality control issues and is very useful in varying the composition of the alloy (if desired) without changing the chemical make-up.

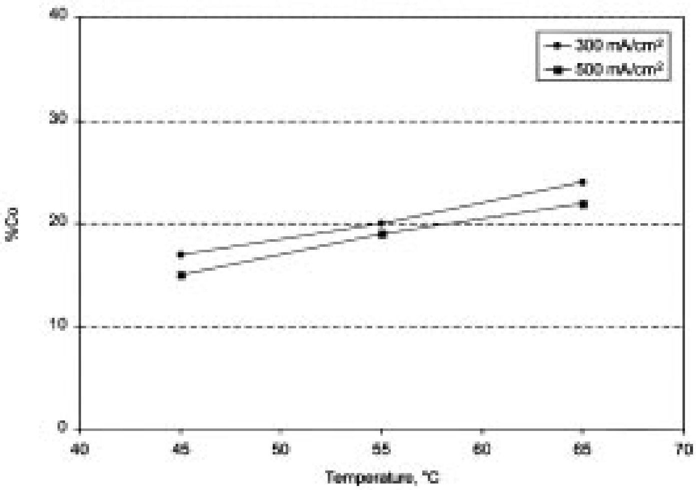

Fig. 5—Wt pct Co in deposit vs. temperature (High-Speed Process @ 300 mA/cm^2 and 500 mA/cm^2.

Unlike the typical Pd-Ni system where pH is a major factor in controlling composition, pH does not play a very significant role in determining the Co content in Pd-Co. This can be seen in Fig. 6 where a very slight increase in Co content is observed as the pH is raised.

Current Density, Effect on Alloy Composition

Current densities for the high-speed process can vary from less than 50 mA/cm^2 to more than 500 mA/cm^2, depending on the specific application. One of the concerns is the stability of the composition as a function of current density because a single bath composition is expected to plate various products that may require operation of the plating process at different current densities. Moreover, the current density across a plated part may vary considerably, yet the composition of the alloy must be maintained to assure reliability.

Figure 7 shows the alloy composition at current densities ranging from 50 to 700 mA/cm^2. As can be seen, the Co content varies from 19 to 28 percent.

Solution Agitation, Effect on Alloy Composition

Solution agitation is a parameter that must be considered in plating alloys such as Pd-Co. If the reduction potential for the two metals is significantly different, the limiting current density that prevails for each individual metal will affect the composition of the deposit. Only if the plating potentials of both metals nearly coincide, can the alloy composition be relatively insensitive to changes in agitation. This has been achieved as illustrated in Fig. 8,

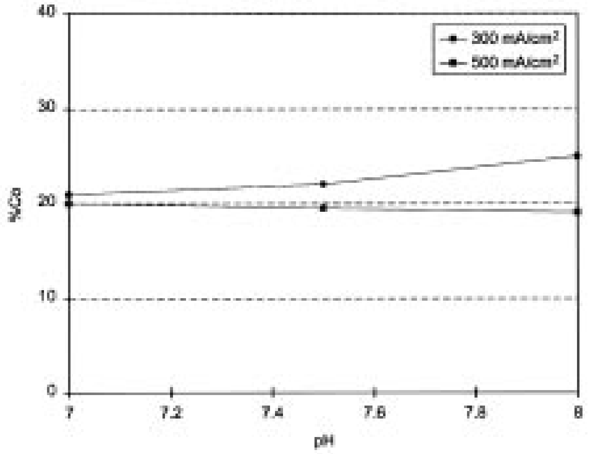

Fig. 6—Wt pct Co in deposit vs. pH (High-Speed Process @ 300 mA/cm^2 and 500 mA/cm^2.

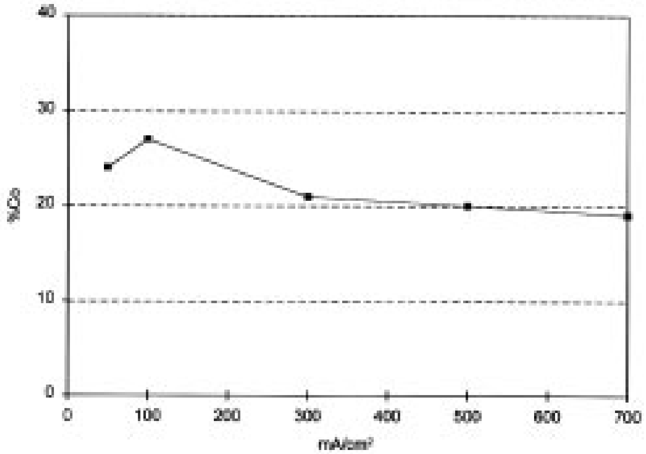

Fig. 7—Wt pct Co in deposit vs. current density (High-Speed Process).

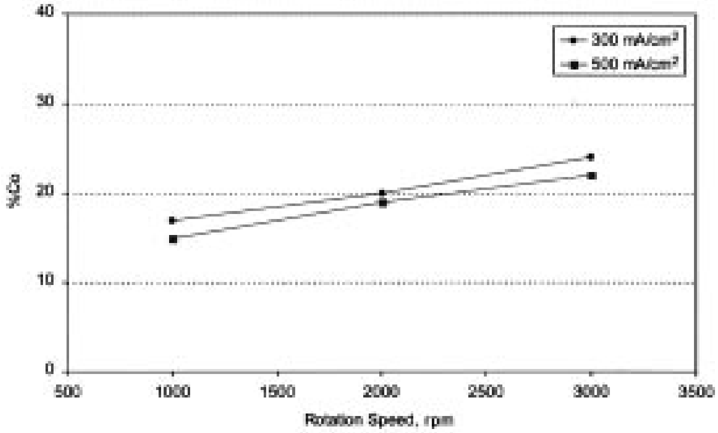

Fig. 8—Wt pct Co in deposit vs. rotation speed (High-Speed Process @ 300 mA/cm^2 and 500 mA/cm^2.

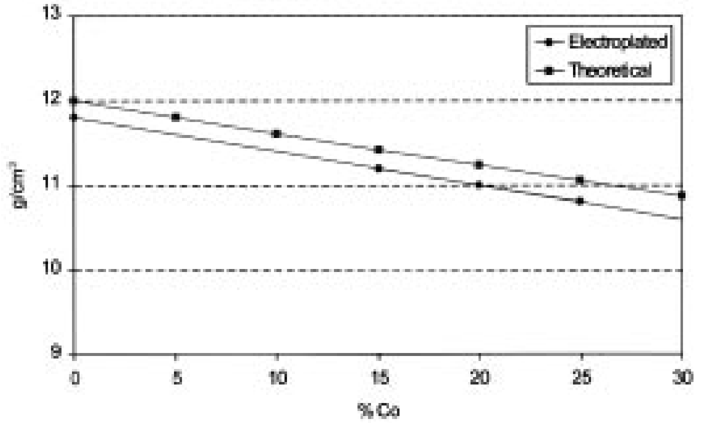

Fig. 9—Density of electroplated Pd-Co @ 0-30 wt pct Co content.

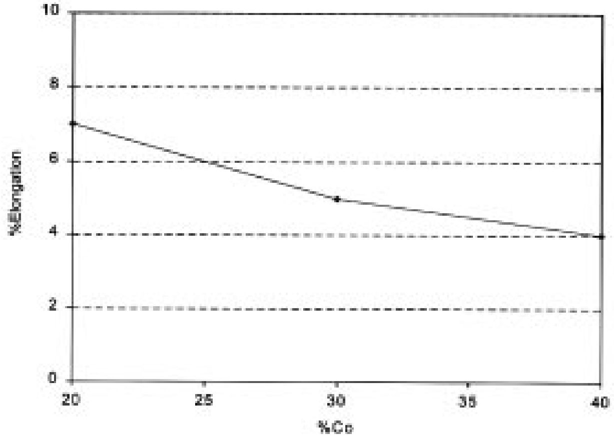

Fig. 10—Ductility of electroplated Pd-Co @ 20-40 wt pct Co.

Fig. 11—Electroformed 80/20 Pd-Co @ 20-40 wt pct Co.

where the Co content is relatively constant as a function of solution agitation and current density.

Materials Properties & Process Performance

Materials Properties

The ultimate objective of any electroplating process is to produce deposits with desired physical properties that remain constant as a function of bath usage or aging. All samples for material properties and aging studies were plated under standard conditions described in Table 3 to a thickness of 2.5 µm; alloy composition was 80/20 Pd-Co. Table 1 describes some of the general material properties of Pd-Co and compares them to Hard-Gold and Pd-Ni.

Density

Density measurements indirectly provide information about the composition (*i.e.*, level of impurities) and physical structure (*i.e.*, void content) of electrodeposits.[38] Usually, the density of electrodeposits is lower than the metallurgical materials because of the presence of voids and impurities. Density measurements also provide information about the phase composition of electrodeposits. For example, Brenner *et al*.[39] reported a straight-line relationship between density and the phosphorus content of electrodeposited Ni-P alloys.

Density can be measured by various methods[38] and in this study a pycnometric method was used. As shown in Fig. 9, the density of the deposited alloys is ~2 percent lower than the theoretical density and varies linearly with the Co content. The theoretical density was calculated based on the wt pct of Co in the alloy and the individual densities of Pd and Co in the following manner:

$$d_{\text{theoretical}} = 100/(8.32 + 0.0292\ \%\text{Co})\ \text{g/cm}^3$$

Ductility

Ductility was measured by the ASTM B 489-85 bend test. This method involves bending samples plated on a thin Cu substrate through a series of angles over mandrels of varying diameter, or over dies of varying radii, utilizing a bending machine described elsewhere.[32] Inspection for cracks is conducted at 10X magnification. In our specific case, the angles were 90° and 180° and the diameter of the mandrels varied from 20 to 132 mil.

Ductility was measured on 12.5-µm-thick copper foils on which 1.25 µm 80/20 Pd-Co deposits were electroplated. Fig. 10 shows the ductility of Pd-Co as a function of percent Co in the deposit. Of particular interest is a photograph of a "free-standing" electroformed Pd-Co foil plated to 29 µm (Fig. 11).

Hardness

The Knoop hardness was measured by a Tukon hardness tester, using a diamond indenter at 50-g load. The samples were 25 µm in thickness. Figure 12 shows the variation in hardness vs. the Co content in the deposit. Figure 13 shows the variation in hardness as a function of bath aging for Pd-Co, Pd-Ni, Pd and

Hard-Gold. It can be seen that the hardness of the Pd-Co is in the range of 590-640 KHN, which is ~20 percent higher than Pd-Ni and ~40 percent higher than Pd, and remains fairly constant with bath aging. This property is important for wear resistance.

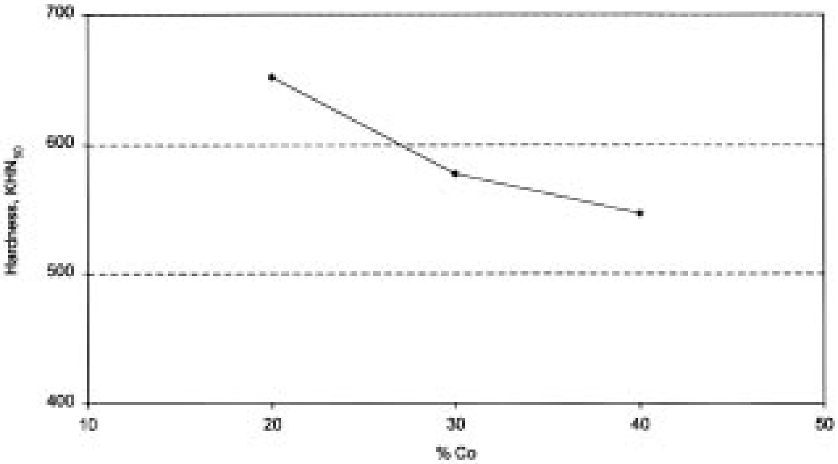

Fig. 12—Hardness of electroplated Pd-Co at 20-40 wt pct Co.

Process Performance—Bath Aging

A plating process is not ready for field trials until the chemistry has been "aged" and the process proves robust to chemical, electrochemical and thermal degradation. Various properties were investigated, such as alloy composition, hardness, brightness and plating rate as a function of aging.

As discussed in the previous section, composition is of utmost importance because it determines the material properties of the deposit. Figure 14 shows the alloy composition as a function of current densities at zero and one bath turnover. Figure 15 looks at two specific current densities, 100 and 300 mA/cm^2, up to three bath turnovers. It is clear that the alloy composition is not affected by the aging of the bath.

Plating rate is also very significant since assurance is desired that the plated thickness remains constant as the bath ages. Figure 16 shows that the plating rate (μm/sec/100 mA/cm^2) at two current densities remains constant as a function of aging.

Process Performance—Field Trials

A plating process is essentially a laboratory curiosity, considered "manufacturable" only after successful field trials. The field trials are intended to assess the ability of the process to produce the desired deposit characteristics, its "manufacturability," and its cost of operation.

The field trial consisted of plating two types of raw material on a "production" machine. It was designed to assess the operating windows as follows:

- Thickness & composition vs. current density & aging
- Appearance vs. current density & aging
- Adhesion vs. aging
- Porosity vs. aging
- Solution stability & control

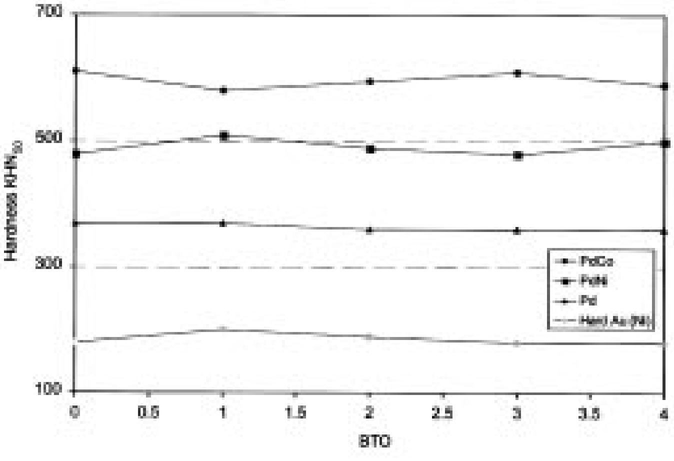

Fig. 13—Hardness of electroplated 80/20 Pd-Co as a function of bath aging vs. Pd-Ni, Pd and Hard-Gold (Nickel).

The continuous reel-to-reel plating machine was configured for a typical connector finish as follows:

- Pay-off
- Alkaline clean
- Acid activate
- Ni plate
- Pd-Co plate
- Au flash
- Dry
- Take-up

The Pd-Co plating "station" consisted of two controlled-depth cells, positioned in series and fed with the Pd-Co chemistry from one reservoir. The use of two cells in series provided the capability to age the solution at a high rate, such as would be experienced in a continuous "around-the-clock" manufacturing environment. Line speeds used were from 1 to 7 m/min.

Two types of raw material were utilized. One was brass stock, 1.9 cm wide by 0.02 cm thick. This material provided maximum surface area for metal deposition, along with the required flat surface for accurate thickness and composition determinations. "Backplane" pins, specifically 1-Type, Body-Carried Contacts (Fig. 17), were also plated. These contacts are commonly used in switching systems and have very stringent specification criteria. These pins were chosen because their configuration was typical of many connector components and because of a long history at Lucent Tech-

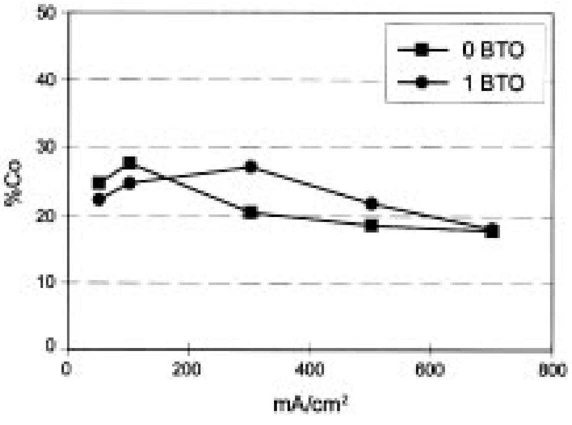

Fig. 14—Alloy composition of electroplated Pd-Co as a function of current density and bath age.

nologies where the surface finish changed from Hard-Gold to Gold Flash Palladium (GFPd) to the present GFPd-Ni. Therefore, they provide an excellent baseline for assessing the quality of Pd-Co.

As previously discussed, maintaining composition is essential in providing a plated alloy that can meet specifications. One of the critical factors in a manufacturing environment is whether the alloy composition can be maintained as a function of "distance" along the plated item, in our case a connector pin. Figure 18 demonstrates that the Co varies from ~13 percent at the tip of the pin to ~18 percent at 0.76 cm from the tip of the pin, defined as the "plate line." Obviously, the difference in composition results from the differences in current density and solution agitation experienced by the separate areas of the pin. Nevertheless, the variation in Co content is acceptable and remains stable as a function of bath aging. Similarly, Fig. 19 illustrates the thickness variation along the pin, which varies from 1.7 µm at the tip to 1.5 µm at the "plate-line."

Porosity of the pins was measured by the sulfurous acid vapor method outlined in ASTM B 799-88. This test exposes the plated part to sulfur dioxide and, subsequently, hydrogen sulfide vapors. The pores were counted using a porosity index method described elsewhere by Kudrak.[8] In this case, the number and size of all pore sites visible at 10X magnification were measured. Figure 20 charts the average porosity of 1.25-µm Pd-Co and Hard-Gold plated on 1.25 µm of Ni. It clearly reveals that the Pd-Co is slightly better than the Hard-Gold and maintains this property as a function of aging. This substantiated laboratory tests on plated coupons, as shown in Table 1.

The results of the field trial indicate that the Pd-Co plating process is a robust manufacturing technology. The Co content can be varied from 10 to 30 percent of composition and maintained ±5 percent of the desired composition, usually 80/20 Pd-Co. The deposited Pd-Co alloys are bright in appearance, have excellent adhesion, and show relatively low porosity. Overall, the process was easy to control and maintain as the bath aged.

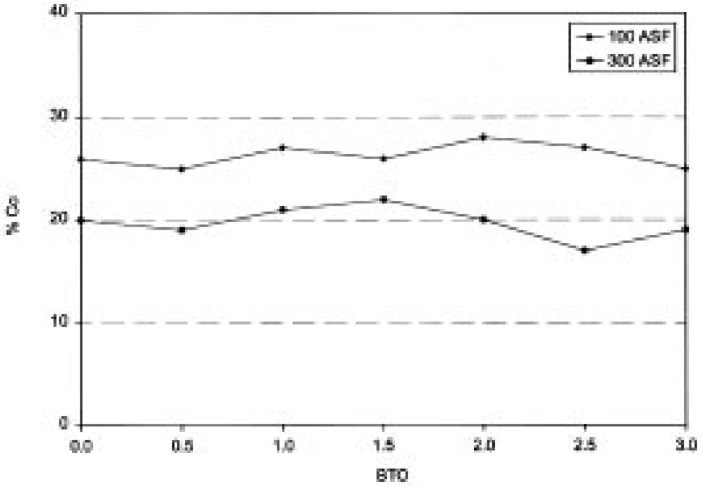

Fig. 15—Alloy composition of electroplated Pd-Co as a function of bath aging.

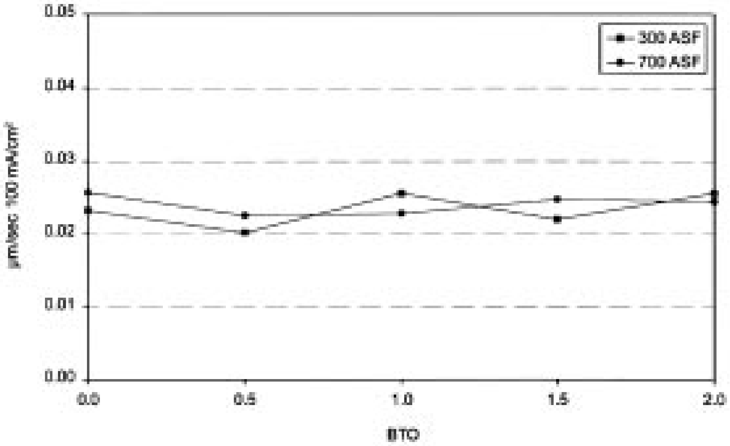

Fig. 16—Plating rate of electroplated Pd-Co in µm/sec/100 mA/cm².

Wear Resistance of Gold-Flashed Pd-Co

Gold-flashed palladium (GFPd) and GFPd-Ni have been extensively studied as contact materials. We recently reported[32-34] data on the wear and contact resistance of GFPd-Co plated to 1 µm thickness on 2.5-µm-thick nickel deposited on a highly polished (20 nm rms) OFHC Cu disk. The sliding wear testing was conducted using the "rider-flat" system described elsewhere.[32] The "rider" was a rivet with a 1.75-mm radius and the "flat" was a 25-mm diameter disk, loaded to 100 g with a reciprocal movement of 14 mm in amplitude at a rate of 30 strokes/min (0.5 Hz). The frictional force was measured by a strain gauge, and the contact resistance was simultaneously evaluated by driving a 25-mA current through the system while measuring

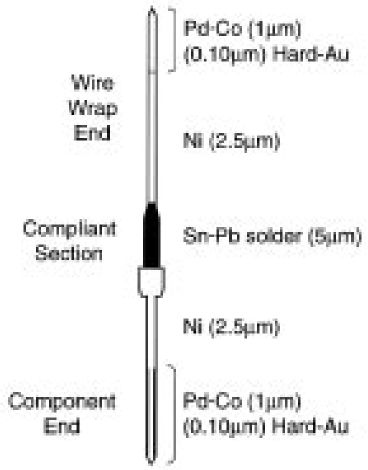

Fig. 17—A schematic and optical photograph of 1B backplane pin. (Lucent FASTECH® Interconnection System).

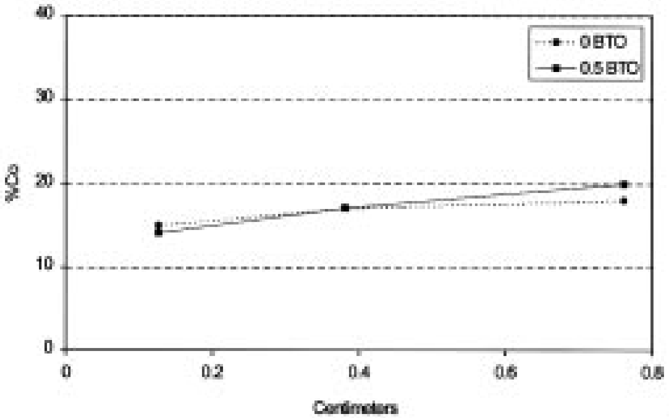

Fig. 18—Alloy composition as a function of distance from the tip of the plated pin.

the voltage drop. The frictional force and contact resistance were measured 20 times each wear cycle.

The sliding wear resistance of GFPd-Ni, GFPd-Co and Hard-Gold are shown in Fig. 21. It can be seen that GFPd-Co outperforms both Pd-Ni and Hard-Gold, maintaining a relatively low contact resistance at more than 80,000 wear cycles.

Summary

The electrodeposition process described in this paper produces mechanically stable, specular Pd-Co films at current densities from less than 50 mA/cm^2 to greater than 700 mA/cm^2. It can produce alloys of 10 to 30 percent Co content. In addition, any desired composition (*e.g.*, 20% Co) can be maintained within ±5 percent over a wide range of operating conditions and bath aging. The material properties of the Pd-Co deposits compare favorably with Hard-Gold and Pd-Ni. As can be seen, the Pd-Co alloy is much harder than Hard-Gold or Pd-Ni and exhibits superior durability. Overall, it has been proven to be an excellent material for contact finish applications.[35]

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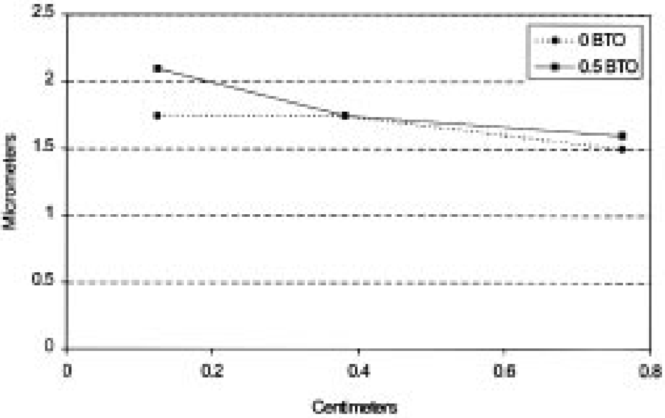

Fig. 19—Thickness of Pd-Co as a function of distance from the tip of the plated Pin.

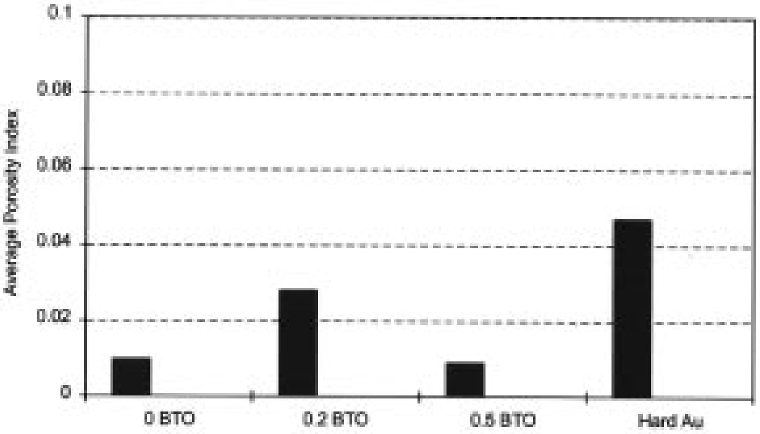

Fig. 20—Porosity index of Au-flashed Pd-Co as a function of bath aging for 1.2-μm-thick deposits.

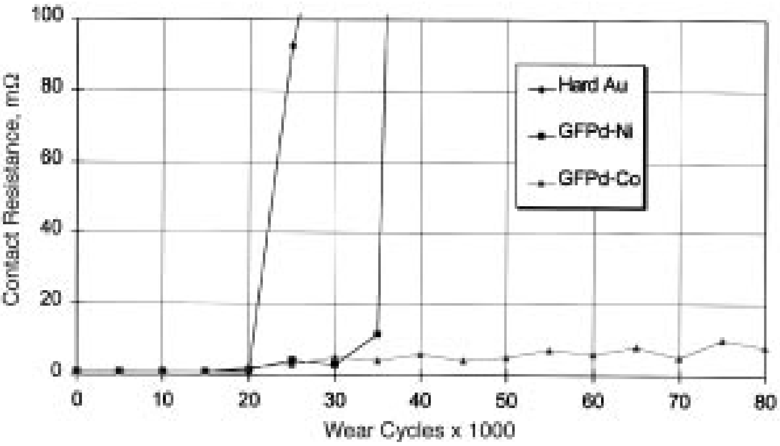

Fig. 21—Sliding wear contact resistance for Hard-Gold, GFPd-Ni and GFPd-Co.

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About the Authors

Dr. Joseph A. Abys is president of the Electroplating Chemicals & Services New Venture organization of Lucent Technologies (Bell Laboratories), 600 Mountain Ave., Murray Hill, NJ 07974-0636. He received a PhD in physical inorganic chemistry from Brown University and has been working in the field of electrodeposition for 19 years, with concentration on plating of palladium and its alloys. Recently, the organization's efforts have expanded to include tin, tin-lead, nickel and rhodium. Dr. Abys has authored three book chapters and more than 50 technical publications. In addition, he has been granted more than 35 patents in the U.S. and abroad. He was the recipient of the 1997 Lucent Technologies Patent Recognition Award and was inducted into the New Jersey Inventors Hall of Fame in February, 1998.*

Gregory F. Breck is a technical service engineer (MTS) with the Electroplating Chemicals & Services New Venture organization of Lucent Technologies (Bell Laboratories), Murray Hill, NJ. Prior to joining Lucent Technologies, he was a project manager in the New Products Introduction Group at AT&T Microelectronics. His experience covers more than 20 years of barrel, rack and reel-to-reel plating. He holds a BS in chemical engineering from the University of Kansas and has written many scientific publications.

Dr. H.K. Straschil was a member of the technical staff of Lucent Technologies Electroplating Chemicals & Services business unit, Murray Hill, NJ. He was involved in research and process development of palladium, its alloys, and gold-tin alloys. He received his Doctor of Technical Science degree (inorganic and physical chemistry) from the Technical University, Vienna, Austria, and taught General Chemistry, Physics and related courses at several colleges in New Jersey. **Editor's note:** With regret, we report that Dr. Straschil passed away, March 18, 1998.

Dr. Irina Boguslavsky is an R&D project leader, Plating & New Materials, at Reliability Electrochemical Laboratory of Molex Inc. Her responsibilities include optimization of existing plating processes and developing and testing new connector finishes. She holds an MS in chemistry from Moscow State University and a PhD in electrochemistry from the Institute of Technology, St. Petersburg. She has written more than 15 technical papers and made presentations at international symposia. She holds three patents in the USSR.

Dr. Göran Holmbom is a project manager for surface treatment at CSM Materialteknik AB in Sweden. He holds a PhD in physics from Linköping University, Sweden. His experience includes materials characterization of electrodeposits, as well as R&D electroplating for microelectronics, such as surface treatment for wire bonding and soldering. Another important area of his experience is pulse plating for composition-modulated alloy plating, mainly for engineering applications.

** To whom correspondence should be addressed.*

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