Annealing Behavior of Palladium-Nickel Alloy Electrodeposits

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The annealing behavior of palladium-nickel alloy deposits in the range of 85-15 to 70-30 percent Pd-Ni can be utilized to produce a ductile electrodeposit amenable to stamping and forming of electronic contacts. Differential scanning calorimetry, scanning electron microscopy, and transmission electron microscopy have been employed to explain the annealing behavior and reconcile the anomalous observations with the classical binary phase diagram information. The annealed deposit has the desired physical properties characteristic of the Pd-Ni alloy, especially hardness, ductility, and corrosion resistance. Practical application of the annealing process has been demonstrated in the electroplating, annealing and stamping and forming of stripe-on-strip 725 alloy and 910P materials to make electronic contacts and connectors.

Gold has been used extensively as a contact material in the electronics industry. The deregulation of gold in the early 1970s, coupled with the political and economic events of the times, brought about an astronomical increase in its price in the later 1970s and early 1980s. In recent years, gold has traded in the range of $320 to $440 per troy ounce, a factor of 10 higher than its price prior to deregulation. Consequently, *one* of the reasons for substituting gold as a metal finish for electronic applications has been economic.[1,2] Recently, this change has been driven by more stringent technological requirements, however, not readily met by electrodeposited gold.[3-13]

Electroplated palladium has been used extensively for this purpose. Technologically, the material properties (*e.g.* hardness, ductility, thermal stability) of palladium are in many instances superior to hard gold.[3-10, 14-24] For example, the higher hardness of palladium is beneficial for wear resistance,[3] which can be further enhanced by a thin coating of gold[25] and the application of synthetic lubricants.[25,26] The arguments for the use of palladium as a contact material are, in some cases, more valid for palladium-nickel. Alloying with metals such as nickel or cobalt is employed to improve its material properties. For example, suitable amounts of nickel (~20 percent wt) incorporated into palladium have been reported to produce an alloy that is brighter, harder and more ductile,[27] less susceptible to hydrogen embrittlement, less catalytic towards frictional polymerization,[28] and which exhibits lower porosity[6,11-13] and better wear resistance than either hard gold or palladium.[29-31] It is no surprise, then, that palladium-nickel has attracted considerable interest in the electronics industry.[3-7, 32-37]

Alternatively, clad inlay materials have also attracted some attention for connector applications,[38-41] although their use seems to be diminishing. The superior ductility and extensive alloy selection of wrought materials have been touted as considerable advantages in the fabrication of connectors.[40] Clad inlay gold and gold alloys have been said to demonstrate "nobility in hostile environments, stable electrical performance after exposure to elevated temperatures, low porosity and excellent ductility."[40] The ability of clad inlays to be stamped and formed into connectors, is, of course, one of the advantages of these materials.

Clad inlays have a number of disadvantages, however. The material hardness is lower than for electrodeposits; this can be problematic when high wear is desired. Antler[39] states that clad metals tend to wear severely by adhesive wear mechanisms when mated to themselves, and by an abrasive mechanism when mated to either hard gold or gold-flashed palladium. He provides the following guidelines when cladding is to be utilized:

- Clad metals should not be mated to themselves.
- Clad metals can be mated to a hard gold electroplate or hard gold-flashed palladium, provided that the clad metal has the smaller swept area.
- The plated surface should be smooth, free of nodules and other features which may abrade the cladding.

When both contacts are *plated* with the same metal, however, (*e.g.* gold-flashed Pd or gold-flashed Pd-Ni), surface roughness is not an important determinant of wear. The clad inlay industry recognized that this is a serious problem and has investigated the use of thin electrodeposits to enhance the wearability of these materials.

Porosity (or pore corrosion) is also an issue with electronic coating materials. Subsequent degradation of a contact interface, ranging from simple tarnish or discoloration to more serious deterioration, such as increased contact resistance or loss of solderability, can lead to failure during critical operations. These may in turn result in significant reduction in a system's overall performance.

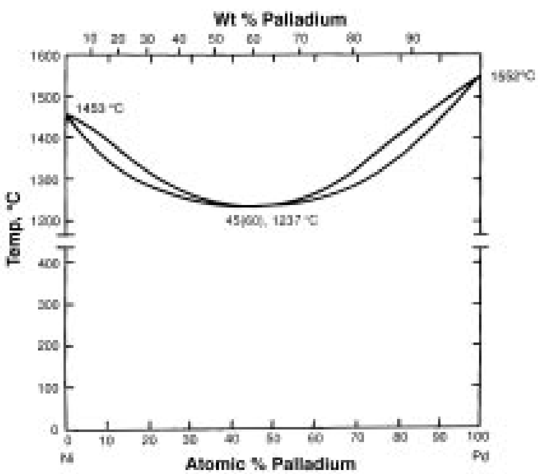

Fig. 1—Phase diagram of Pd-Ni alloys.

Porosity of inlay materials is generally accepted as low when compared to plated deposits, largely resulting from greater thickness and the nature of the rolling process itself. Porosity of the much thinner precious metal electroplated finishes, palladium-based finishes in particular, has undergone extensive investigation, typically in relation to deposit thickness or plating solution composition and operating parameters. In one study, it was found, for example, that deposits from a cobalt hard-gold solution produced large changes in porosity with respect to changes in current density, while palladium-nickel deposits exhibited little or no change under all conditions of the variables examined.[30]

Many of these studies have demonstrated that the ability of palladium-based electrodeposits to protect underlying base-metal substrates, or underplates, against the effects of corrosion generally goes unchallenged. Accordingly, they continue to enjoy widespread use as electronic contact surface finishes.

Clad inlays are excellent in their ability to be stamped and formed but have demonstrated poor wear and higher costs. The electrodeposits have demonstrated significantly better wear performance at a lower cost but in most instances cannot be stamped and formed into connectors. There is, therefore, a need to develop a plating process that results in highly ductile electrodeposits that can be fabricated into connectors.

We report the annealing behavior of electroplated palladium-nickel alloys (0-30 percent wt) deposited from a proprietary solution. It will be demonstrated that deposits in the nickel range of 15 to 30 weight percent can recrystallize at relatively low temperatures (~380 °C), and that this annealing behavior is connected to the characteristic palladium-nickel binary phase diagram. Moreover, it will be shown that the recrystallized electrodeposit has a higher ductility than the as-plated material, consequently enhancing its potential for "stripe-on-strip" applications.

Background

We have been investigating the electrodeposition of Pd and its alloys since 1979 and have published two papers on palladium-nickel.[4,42] In these papers, we discussed the electroplating and material properties of Pd-Ni alloys and their contact behavior. In one of these papers,[42] we reported but did not elaborate on the observance of exothermic phenomena at ~380 °C, seen in differential scanning calorimetry (DSC) experiments.

An inspection of the Ni-Pd binary phase diagram,[43] Fig. 1, indicates that the melting point can be lowered to ~1237 °C for the 40 wt percent Ni, from 1453 °C for pure Ni and 1552 °C for pure Pd. This diagram is somewhat similar to that of eutectic binary systems, in which the melting point is lowered by alloying. The low temperature exotherm observed at ~380 °C is much lower than the lowest recrystallization temperature for Pd-Ni and this warranted an investigation. The structural and materials properties of Pd-Ni (80/20 percent wt) that had undergone an apparent form of recrystallization (termed "thermal recrystallization") were studied; it was found that the heat-treated material could be stamped and formed, without cracking, into connectors.

Experimental Procedure

Sample Preparation

Films of palladium-nickel alloys were electroplated on oxygen-free, high-conductivity (OFHC) copper foils, using a proprietary solution described elsewhere.[2] Palladium-nickel alloys having the concentration ranges 0, 5, 10, 15, 20, 25 and 30 percent nickel were prepared by varying the concentrations of palladium and nickel in the solution. Thick deposits greater than 25 µm were separated from the OFHC copper substrates by a chemical etching technique that utilized a solution of ammonium hydroxide 30 percent, ammonium

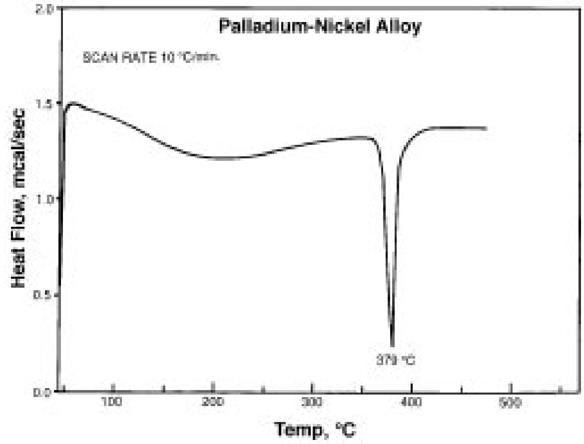

Fig. 2—Differential Scanning Calorimetry, Pd-Ni 80/20 alloy.

chloride and hydrogen peroxide 35 percent. Plated foils were suspended in the solution, which turned dark blue as the copper was etched. The process was repeated until no copper was detected after several hours in the final solution, as determined by atomic absorption spectrometry.

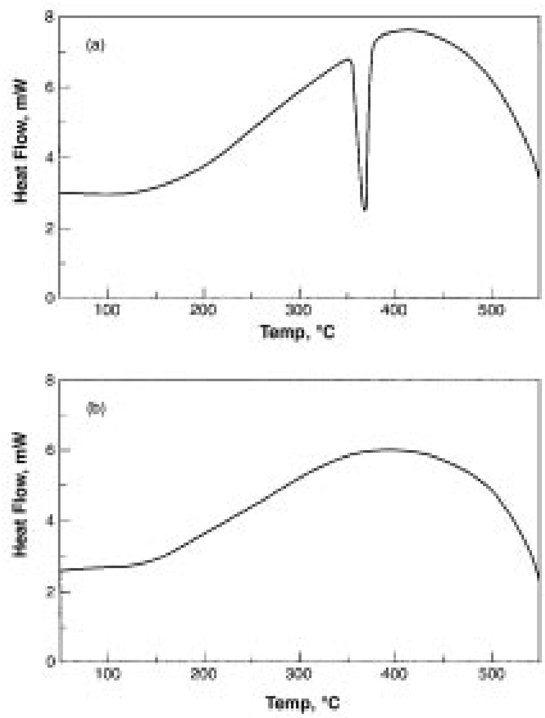

Fig. 3—Differential Scanning Calorimetry (Pd-Ni 80/20 wt %): (a) first sweep; (b) second sweep.

Differential Scanning Calorimetry

For differential scanning calorimetry (DSC) measurements, the samples, weighing 5 to 10 mg, were first put into a graphite container and heated to 550 °C under a nitrogen atmosphere. The rate of heating was 10 °C/min. (This technique is ideal for characterization of physical changes in a material caused by heating. In addition, the onset temperature and magnitude of any exothermic or endothermic phenomena associated with a thermally-induced physical change can be quantitatively determined.)

Transmission Electron Microscopy

TEM analysis was done using an electron microscope operated at 300 KV. *In-situ* heating experiments inside the TEM were conducted from room temperature (20 °C) up to 550 °C. TEM is ideal for determining the grain size of fine-grained deposits such as palladium-nickel, organic occlusions in the grain boundaries and uniformity of alloy composition. The ability to heat the sample *in-situ* offers an unparalleled opportunity to visually observe and confirm physical phenomena in real time.

Microstructure, Hardness, Porosity and Ductility

Selected 40- to 60-µin. (0.040 to 0.06 mil) electrodeposits over OFHC copper were also tested for general morphology and microstructure, hardness, porosity and ductility before and after thermal annealing. The microstructure of the samples was investigated, using scanning electron microscopy of metallographic cross-sections to determine the grain growth of the electrodeposits. Knoop hardness was measured by a hardness tester, using a diamond indenter at 50 g load. Porosity to copper was determined using the sulfurous acid vapor test (ASTM Method B-799-882).

Ductility was measured as specified in the ASTM B-489-85 bend test. This method involves bending plated samples over a series of mandrels of varying diameter, followed by inspection for cracks at a magnification of 10X. For the purposes of this test, samples of 40-µin.-thick 80-20 wt percent Pd-Ni alloy were plated on 0.5, 2.0, 5.0 and 10 mil* thick OFHC copper and bent around a series of mandrels ranging from 20 to 111 mil. This enabled estimation of the percent elongation over discrete ranges, using a pass/fail criterion. Samples that passed the 10X magnification inspection were also examined at 3000X using scanning electron microscopy to ensure that they were crack-free. The elongation test table is shown below:

Foil Thickness	Mandrel Diameter	Percent Elongation
0.5 mil	20 mil	3.0
2.0	40	5.1
5.0	70	6.9
10.0	111	8.4
5.0	50	9.3
10.0	85	10.7
2.0	20	10.7
5.0	40	11.4
10.0	70	12.7
10.0	50	16.9
10.0	40	20.2
5.0	20	20.5
10.0	20	33.2

* 1 mil ≈25 µm; 1000 micro-in. ≈25 µm

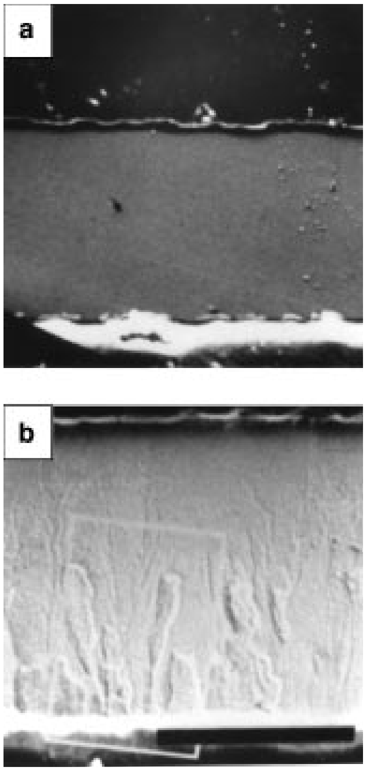

Fig. 4—Scanning Electron Micrographs: (a) as-plated; (b) heat-treated.

Results

Differential Scanning Calorimetry

As part of a "general analysis" of electrodeposits plated from proprietary solutions developed in this laboratory, DSCs are utilized to determine bulk thermal behavior. Figure 2 shows a DSC curve for electroplated Pd-Ni (20 wt percent) with a sharp exothermic peak at 379 °C. The heat associated with this event is ~3.6 cal/g. No such peak was observed for pure electroplated Pd and Pd-Ni (~10 wt percent) upon heating to 550 °C. Moreover, a second sample (Fig. 3a) demonstrates this behavior and upon re-heating this same sample through the identical thermal excursion, no peak is observed (Fig. 3b). This indicates that the phenomenon is related to the "structure" of the electroplate, which is obviously deposited in a non-equilibrium state. It was of interest to investigate the structural and material property changes associated with this phenomenon.

Microstructural Analysis

Figures 4a and 4b are scanning electron micrographs of the cross-sectioned, as-plated and heat-treated Pd-Ni alloy. The cross section of the as-plated film is featureless and is apparently very fine-grained, so as to be difficult to resolve by SEM. Alternatively, the heat-treated sample is large-grained, showing a growth that originates from the substrate side and grows toward the surface. The dramatic change in the grain structure appears to be driven by recrystallization and grain growth processes.

A more detailed analysis was conducted via transmission electron microscopy (TEM). Figures 5a and 5b present the bright- and dark-field TEM micrographs, showing the fine-grained structures of the as-plated films. Bright spots seen in the dark-field image yield the size and shape of a selected group of similarly-oriented grains. As seen in Fig. 5b, the grain shape is highly irregular and the grain size is very small, ranging from 50 to 200 Å. These grains did not show any preferred orientation. This small grain size can be compared with the average grain size of other electrodeposits, such as 350 Å for cobalt-hardened gold[44] and 50 Å for Pd deposits.[45] From the grain size, therefore, the as-plated

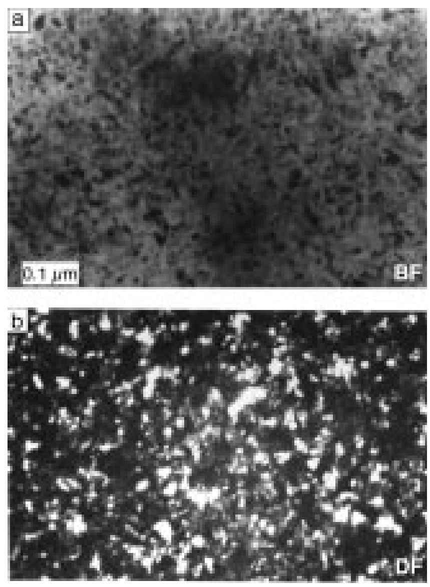

Fig. 5—Transmission Electron Micrographs, Pd-Ni: (a) as-plated; (b) heat-treated.

Pd-Ni 80-20 deposit is expected to be hard and relatively brittle.

The annealed film (Fig. 6) contained large grains and several dislocations, as marked by the symbol D. The grain, which was imaged under strongly-diffracting conditions, shows a mottled background, indicating uniform distribution of small localized strains in the film. Figure 7 shows a large recrystallized grain surrounded by a few smaller recrystallized grains and is seen to contain “twins” marked by the symbol T. These twins, which are limited primarily by coherent boundaries, are most likely to be twin-oriented to the matrix in the as-plated state and not annihilated by the sweeping of high-angle grain boundaries during recrystallization. The presence of twins behind the migrating grain boundary is direct evidence that the observed process is promoted primarily by grain boundary diffusion, not by bulk volume diffusion. This argument will be presented in more detail in a future publication.

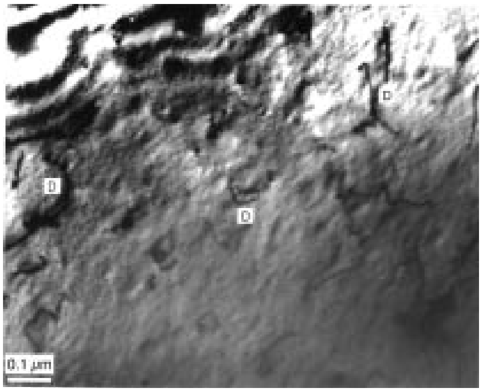

Fig. 6—Transmission Electron Micrographs: Heat-treated sample; D shows areas of dislocations.

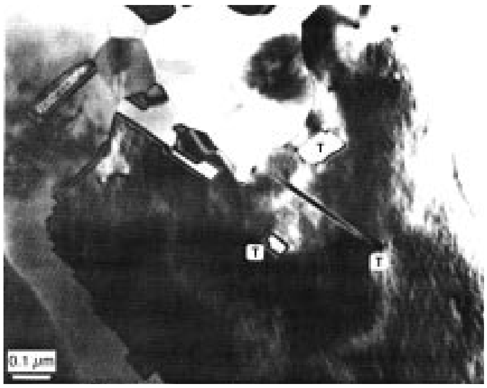

Fig. 7—Transmission Electron Micrograph: Heat-treated sample; T shows “twin” crystals.

Material Properties:

There is no question that the exothermic DSC peak observed at 379 °C is a recrystallization phenomenon related to electrodeposition of small-grain Pd-Ni in a metastable state. TEMs with *in-situ* heating show clearly a “ballistic” recrystallization process, whereupon hitting the “trigger” temperature, a wave front can be seen sweeping forward and eliminating the existing small-grain deposit. The question of interest relates to the effect of this process on the material properties of this deposit and whether or not advantage can be taken in a practical application.

Deposit Hardness and Ductility

As reported by Abys *et al.*,[4] the hardness of Pd-Ni alloys in the range of 16 to 31 wt percent nickel is from 450-550 KHN_{50}. Figure 8 shows the hardness for a high-speed (200 mA/cm^2) plated Pd-Ni in the composition range, 0-32 wt percent Ni at pH 7.2-7.8. Table 1 and Fig. 9 show the as-plated ductility of Pd-Ni, as obtained via ASTM-B-489-85. In this test, foils (of known thickness) are bent around a mandrel and examined for microscopic cracks. In Table 1, the letters N and Y stand for no (N) cracks observed after bending, or yes (Y), cracks were observed after bending. This ductility indicates approximately 8-12 percent elongation. Table 2 allows comparison of these properties to a sample that was heat-treated. As can be seen, the hardness seems to decrease by ~17 percent, while the ductility nearly doubles. It was concluded, therefore, that heat treating this particular proprietary Pd-Ni alloy would provide a deposit hard enough to allow excellent wear resistance, and ductile enough to stamp and form after plating.

The ability to utilize this “ballistic recrystallization” phenomenon was first demonstrated in our laboratories by annealing plated Pd-Ni (20 wt percent) over OFHC copper foils at 625 °C and 650 °C for 30 sec under a nitrogen atmosphere. The goal of this effort was to define an initial set of process annealing conditions for on-line stamping and forming of Pd-Ni connectors, in conjunction with other manufacturing steps. For example, the annealing step could be placed at the end of a reel-to-reel plating line or directly before stamping and forming equipment.

Table 1
Elongation Test Results for Selected Pure Metals & Alloys at Two Different Plate Thicknesses

0.1 mil foil

Bend Angle		**90°**			**120°**			**180°**	
Diam (mils)	**Pd-Ni**	**Au**	**Co-Au**	**Pd-Ni**	**Au**	**Co-Au**	**Pd-Ni**	**Au**	**Co-Au**
132	N	N	N	N	N	Y	N	N	Y
111	N	N	N	N	N	---	N	N	---
86	N	N	N	N	N	---	N	N	---
70	N	N	N	N	N	---	N	N	---
50	N	N	N	N	N	---	N	N	---
40	N	N	N	N	N	---	N	N	---
20	N	N	Y	N	N	---	Y	N	---

0.2 mil foil

Bend Angle		**90°**			**120°**			**180°**	
Diam (mils)	**PdNi**	**Au**	**CoAu**	**PdNi**	**Au**	**CoAu**	**PdNi**	**Au**	**CoAu**
132	N	N	Y	N	N	Y	N	N	Y
111	N	N	---	N	N	---	N	N	---
86	N	N	---	N	N	---	N	N	---
70	N	N	---	N	N	---	N	N	---
50	N	N	---	N	N	---	N	N	---
40	N	N	---	N	N	---	N	N	---
20	Y	N	---	Y	N	---	N	N	---

N = No cracks observed after bending Y = Cracks observed after bending

The results of the ductility tests performed on these samples are presented below:

Percent Elongation Imposed on PdNi Foil

		Annealed for 30 Seconds at	
	Not Annealed	**625 °C**	**650 °C**
3%	Not Cracked	Not Cracked	Not Cracked
5.1%	Not Cracked	Not Cracked	Not Cracked
6.9%	Not Cracked	Not Cracked	Not Cracked
8.4%	Not Cracked	Not Cracked	Not Cracked
9.3%	Cracked	Not Cracked	Not Cracked
10.7%	Cracked	Not Cracked	Not Cracked
11.4%	Cracked	Not Cracked	Not Cracked
12.7%	Cracked	Not Cracked	Not Cracked
16.9%	Cracked	Few Cracks	Not Cracked
20.2%	Cracked	Few Cracks	Not Cracked
20.5%	Cracked	Cracked	Few Cracks
33.2%	Cracked	Cracked	Cracked

These data clearly support the feasibility of this process for many pre-plated "stamp and form" connector applications.

Deposit Porosity

The heat-treated and bent samples described above were subjected to the sulfurous acid vapor test described elsewhere.[4] The results closely paralleled the elongation test results. Samples that were not cracked developed no pore decorations, while unannealed samples bent more than nine percent and annealed samples bent more than 20 percent showed pore decorations along cracks. The results are summarized in the table below:

	Cracked at 20% Elongation	Passed Porosity Test
Unannealed	Yes	No
Annealed at 625 °C	Yes	No
Annealed at 650 °C	No	Yes

Table 2
Selected Properties of Pd-Ni 80-20 wt% Alloy

Property	As-Plated	Heat-Treated
Hardness	582 KHN_{50}	462 KHN_{50}
Ductility	8.5-9.5%	16-20%
Grain Size	50-100 Å	>1 µm

A Practical Application of the Ballistic Recrystallization Phenomenon

The annealing behavior of Pd-Ni (20 wt percent) to promote the ductility of the electrodeposit was applied in the stripe-on-strip plating and stamping and forming of a bifurcated female connector.[22,38] Two base metals were chosen for the study: Alloy 725 and phosphor-bronze 910P. Both were overplated with 150 to 200 µin. of ductile nickel, 30 to 50 µin. of Pd-Ni (80/20 wt percent) and 3 to 5 µin. of cobalt-hardened gold. The plated strips were annealed in a tube furnace such that the strip attained a surface temperature of 650 °C for 30 sec.

More than 10,000 contacts were stamped and formed from the annealed strips. Scanning electron micrographs of the annealed contacts are shown in Fig. 10. No microcracking was observed at 1000X. Moreover, when these contacts were exposed to the sulfurous acid vapor tests, no pore decorations developed in the contact area. SEMs of unannealed contacts are included for comparison in Fig. 11. These pictures show severe cracking, clearly visible at 100X magnification. Figure 12 contains SEMs of contacts made from the DRG-156 inlay finish often used for this application. Connectors fabricated from the heat-treated GFPdNi contacts were wear-tested, along with gold and diffused AgPd (DGR-156) inlay stamped and formed connectors. Both contact finishes were worn against a gold-flashed palladium (GFPd) male pin[22,38] for 200 insertion/withdrawal cycles. The contact load on this connector is ~150 g and relaxes to ~120 g after the wear test. Figure 13 is a plot of the change in contact resistance from cycle 1 to cycle 200 vs. normal probability (percent). As can be seen, both the GFPdNi and DGR 156 inlay show an insignificant change in the contact resistance. Under these conditions, the 0.75-µm-GFPdNi behaved similarly to the 2.5-µm DGR-156 inlay material.

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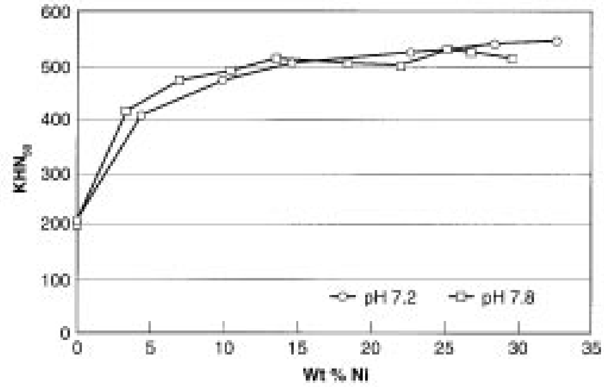

Fig. 8—Hardness vs. Pd-Ni alloy composition.

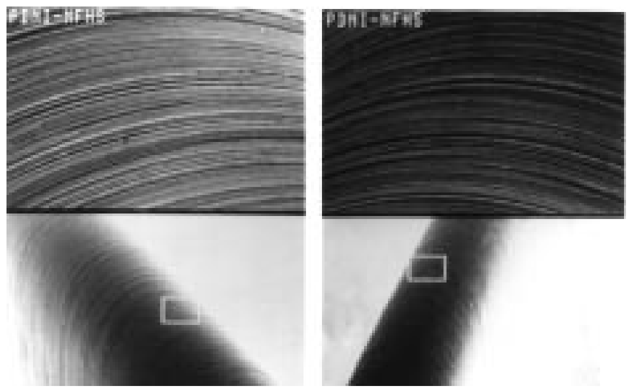

Fig. 9—Scanning Electron Micrograph - 35X, 350X; 25X, 250X; 0.2-mil sample bent 180° over 20-mil-diameter mandrel.

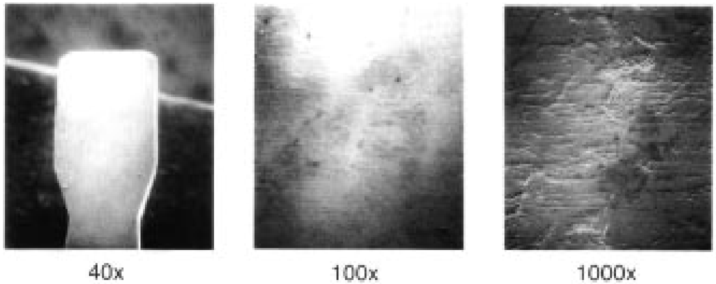

Fig. 10—SEMs of annealed GFPdNi contacts.

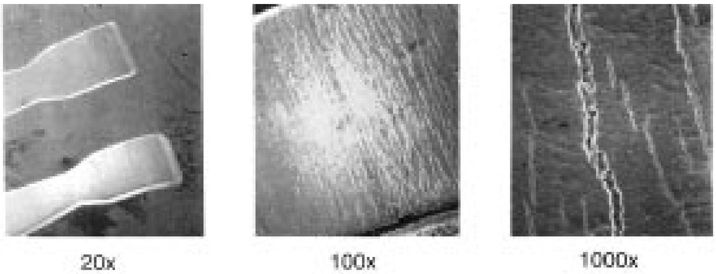

Fig. 11—SEMs of unannealed GFPdNi contacts.

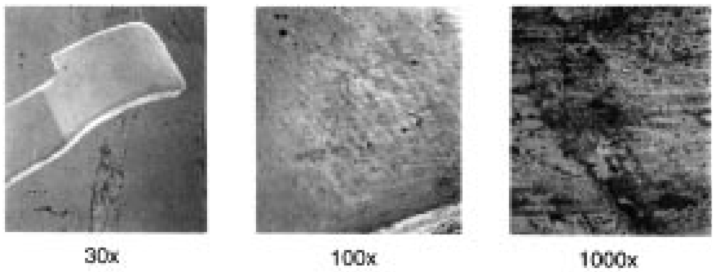

Fig. 12—SEMs of DGR-156 inlay finish contact.

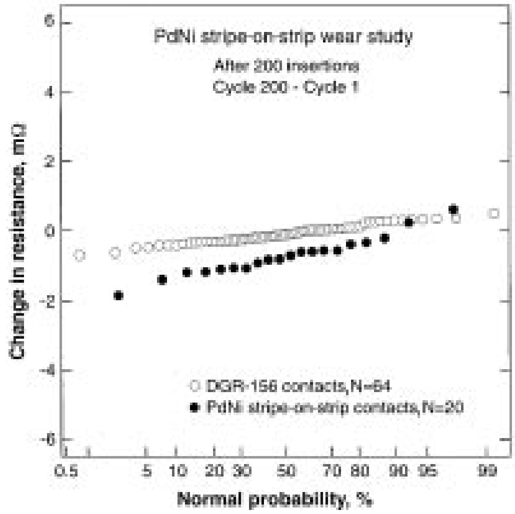

Fig. 13—Pd-Ni stripe-on-strip wear study: Resistance as a function of wear.

Summary

It has been shown that the annealing behavior of palladium-nickel alloy deposits in the range of 15-30 percent Ni can be utilized to produce a ductile electrodeposit amenable to stamping and forming of electronic contacts.[46] Differential scanning calorimetry, scanning electron microscopy and transmission electron microscopy have been utilized to explain the annealing behavior and reconcile the anomalous observations with the classical binary phase diagram information. The annealed deposit has been shown to retain many of the desired physical properties characteristic of the palladium-nickel alloy, especially hardness, ductility and corrosion resistance. Practical application of the annealing process has been demonstrated in the electroplating, annealing and stamping and forming of stripe-on-strip 725 alloy and 910P materials to make electronic contacts and connectors.

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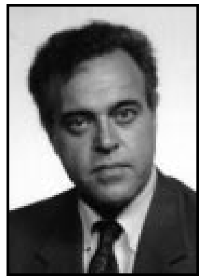

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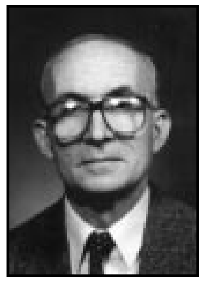

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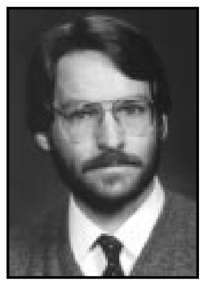

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