Gold Plated Contacts: Effect Of Thermal Aging on Contact Resistance

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Abstract

A study was made of the effect of thermal aging on the contact resistances of pure and of cobalt- and nickel-hardened gold platings (Hard Golds) on copper. Samples were aged at several temperatures, and contact resistance was determined by probing according to ASTM B-667. It was found that, although nickel underplate retards the rate of increase of contact resistance of pure gold, it may accelerate the rate for Hard Golds. This effect becomes more pronounced with increasing hardener content and Hard Gold thickness. Nickel-golds are more stable than cobalt-golds.

Key Words: Contact resistance, thermal aging, gold plate, nickel-gold plate, cobalt-gold plate, nickel underplate, diffusion.

I. Introduction and Background

Cobalt- and nickel-containing electrodeposited golds from cyanide bath chemistries (Hard Golds) have been the preferred finish for the contacts of separable electronic connectors. This is due to their unique combination of properties: nobility, durability, resistance to fretting corrosion, and ease of plating.

Hard Golds are described in specifications issued by the U.S. Military [1a], ASTM [2], Bellcore [3] and by connector manufacturers and users. These documents usually include requirements for gold content, hardness, and thickness, and make recommendations for products in which they can be used. For example, regarding purity, the ASTM Specification recommends deposits having at least 99.7% gold for general purpose, high reliability electrical contacts, and 99% minimum gold for static separable connectors. The Military Specification lists three electrodeposits having at least 99.9, 99.7, and 99.0% gold (Types III, I, and II). An older version of this Specification describes the Type II deposit as having a minimum of 95.5% gold (1b). The Bellcore Specification describes Hard Golds as containing 0.06-0.5% cobalt or nickel. All percentages in this paper are weight percent.

There is considerable variation in users' specifications regarding gold electrodeposits for connector contacts. Finishes containing 99.9-96.0% gold have been found by the author in commercial products, although platings ranging from 99.85-99.6% gold appear to be the most common. It should be pointed out that deposit analysis is made usually for cobalt, nickel, iron, zinc, copper, cadmium, silver, and a few other heavy metals. Analysis routinely is not made for potassium, carbon, and other light elements in the "intrinsic polymers" [4], in the Hard Golds. The gold content is calculated by difference, subtracting the metals analyzed from 100.0%.

The requirements for hardener metal level of Hard Golds originate in sliding wear studies [5,6]. It has been found that deposit ductility and hardness are determining factors that control durability [7,8]. Yet, no simple relationship exists between performance and composition, since plating process variables (perhaps through their effect on deposit structure) also determine the suitability of the gold for any application [9]. Acceptable wear behaviors have been found for deposits containing as little as 0.06% [10] and as much as 4% cobalt and nickel [5]. However, the development of high temperature-stable contact lubricants has tended to make less important the question of hardener metal content, since the adhesive and fretting wear of any finish can be minimized by them [11,12].

The use of ductile nickel underplate for Hard Golds that are less than 5 µm thick is nearly universal. The nickel thickness usually is between 1.25-2.5 µm. Among the advantages of nickel underplate are its enhancement of: (a) the adhesive and abrasive sliding wear resistance of the gold [13], (b) gold durability during fretting [14], and (c) nobility of the contact if the gold is porous and sulfur, hydrogen sulfide, and some other pollutants are present (although nickel may be severely corroded by sulfur dioxide at pore sites [15]). In addition, thin gold deposits may be less porous when some nickel electroplating processes are used [16]. Another advantage of nickel is its effectiveness as a barrier to the thermal diffusion through the gold of copper, zinc, and other metals [17]. The nickel diffuses at a slower rate than most metals, although relative rates of diffusion are temperature dependent. Thus, nickel has been presumed able, in elevated temperature service, to stabilize the contact resistance of gold plated contacts which eventually would increase due to the appearance and subsequent oxidation at the surface of diffused metals to non-conductive compounds. This is a complex issue, since in clean air the nickel oxide thickness is self-limiting; but in polluted atmospheres, such as those containing traces of chlorine, diffusion coefficients are higher due to sinking effects [18].

An early study by the author [19] showed nickel underplate to be able to stabilize the contact resistance of thin pure gold deposits on copper. Samples were aged in clean laboratory air at various temperatures, and contact resistance was determined by probing with a gold rod by a procedure later specified in ASTM B667 [20]. Fig. 1 is representative data; for experimental convenience, the contact was considered to have failed when its contact resistance increased by a factor of approximately 2, to 1 milliohm from the initial value, since extended heating always resulted in a further elevation of contact resistance. That study also showed golds containing cobalt or other base metals to be less thermally stable than pure gold. A hard gold-palladium alloy from sulfite bath chemistry, 50 µm thick on copper, had nearly invariant contact resistance on aging at 200°C for 1000 hours [21]. Since this gold is polymer-free and contains no base metals, its stable contact resistance further supports the evidence that oxidation of non-noble diffused metals at Hard Gold surfaces can cause their contact resistance to rise.

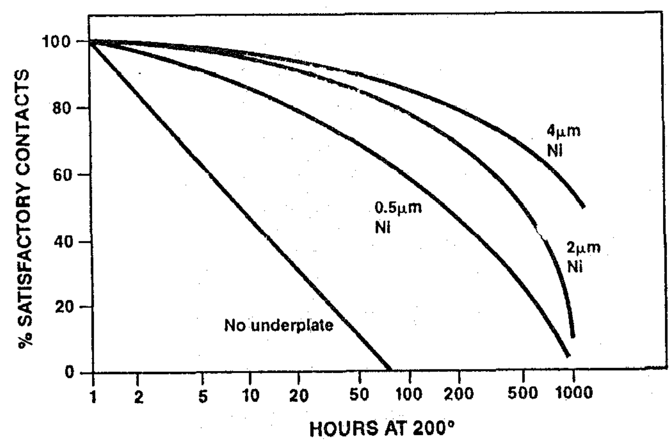

Fig. 1. Effect of heating at 200°C on the contact reliability of 0.5 µm thick pure gold plate on a polished copper substrate without and with various thicknesses (0.5, 2, 4 µm) of nickel underplate. Probed at 1 N. Criterion of satisfactory contact: contact resistance not in excess of 1 milliohm [19].

Recently, there has been increased attention to the need for materials systems having stable contact resistance for extended use at higher temperatures than were formerly required; the thermal limit for Hard Gold electrodeposits on nickel underplate on copper and copper alloy substrates is generally considered to be about 125°C. There is interest in the transportation sector for vehicle underhood service at 145°C and 175°C [22]. There may be still higher temperature requirements in the future. Aging at such temperatures can severely stress common spring metals, insulators, and supporting structures which ultimately may be the limiting materials for such use. Gold flashed palladium-based electrodeposits [23] and claddings [24], and vapor deposited pure gold on stainless steel and copper alloy substrates [25], with nickel [23-25] or Pd20Ni [25] underlayers, have been proposed for the contacts in this application. Heat treated thin pure gold electrodeposited on solid nickel or a nickel-based spring metal has also been developed for high temperature contacts [26]. However, when fretting occurs, palladium-containing coatings may not be suitable because of the possibility of frictional polymer formation with subsequent degradation of contact resistance [27,28].

In view of current interest in high temperature finishes, the varied specifications and users' practices for Hard Gold, and the sensitivity of contact behavior to the composition of the electrodeposit, it was decided to reexamine the thermal stability of contact resistance of some Hard Golds. In particular, the dependence of contact resistance on the hardener level, whether it is cobalt or nickel, the gold thickness, and the effect of nickel underplate were evaluated.

II. Experimental

Pure, cobalt- and nickel-golds, 0.5, 2.5, and 50 µm (±10%) thick from cyanide baths, were obtained from a commercial source. The 50 µm samples were of interest as controls since, because of their large thickness, any thermal effects would unlikely be due to substrate diffusion. The substrates were coupons of OFHC copper, 3.8 cm square by 0.16 cm thick, finished to a roughness of about 0.25 µm CLA by random abrasion on metallographic papers. The cobalt and nickel percentages were 0.07, 0.25, and 0.5 (±10%). Metallic impurities in these deposits did not exceed 0.1%. Nickel underplate, 2.5 µm (±10%) thick, was applied on one set of coupons. The golds were deposited directly on copper in a duplicate set.

Aging was conducted in ovens at 50, 100, 150, and 200°C in laboratory air in a room separated from the main work area. Oven temperatures were controlled within 2°C. There were no known localized sources of air pollutants. Periodically the samples were removed, allowed to cool in a desiccator, probed, and then thermal aging was resumed.

Probing was conducted according to ASTM B667-92 using a solid gold hemispherically-ended rod. The contact resistance measurements were randomly made on each sample. From 5-10 aging time intervals were chosen in developing the curves described below.

The relationship of contact resistance obtained by a standardized procedure, such as ASTM B667, to the contact resistance of contacts in components that are incorporated in a circuit has been the subject of many studies [29-34]. For example, for contacts that become coated with insulating films, both mechanical and electrical factors determine contact resistance and any changes in it which may occur, as in the present investigation of thermal aging. Thus, the hardness of mating surfaces and its difference if they are dissimilar, normal load, surface roughness, contact shape and size, the effect of wipe or vibration on making closure, circuit details like open circuit voltage, and many other factors influence contact resistance. In interpreting the results of this study, it is the direction of contact resistance change in response to a variable, and not its absolute value or magnitude, which should be considered.

The use of a relatively large (1.6 mm radius) hemispherically-ended solid gold probe in ASTM B667 originates in its sensitivity for detecting surface films [31]. In any study which extends over a long time interval, it is necessary to assure the repeatability of the equipment used to measure contact resistance. The procedure for checking the probe is described in the Appendix.

III. Results

Changes in contact resistance at 50 and 100°C were small. At 150 and 200°C, increases in contact resistance were significant, and data are shown for these two temperatures. For brevity, only contact resistances at 1 N are plotted; slightly larger changes were found at 0.25 N. The curves in Figs. 2-7 are smoothed lines from the median contact resistances after various durations of aging.

Fig. 2 is a plot of the contact resistance of 50 µm thick pure and 0.25% cobalt-golds on extended aging at 150°C. In addition to having a higher initial contact resistance due to an increased hardness and a larger bulk resistivity, the cobalt-gold is thermally unstable beyond a few hundred hours of aging. The contact resistance of the pure gold was stable in this test.

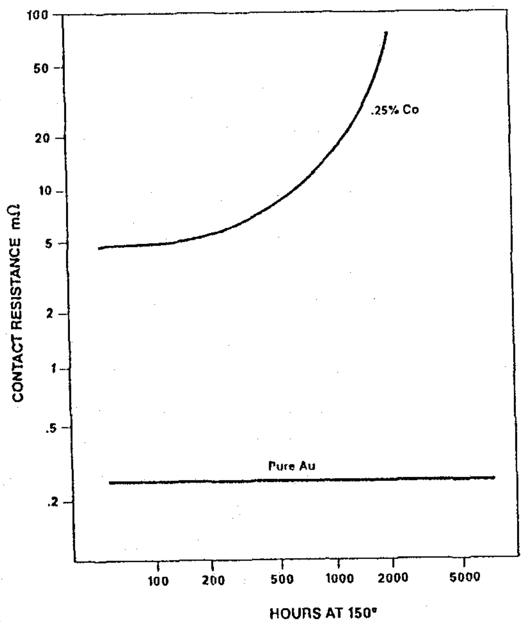

Fig. 2. Thermal stability of contact resistance at 1 N of 50 µm thick gold plate. Aged at 150°C. Effect of cobalt (0.25%) addition to gold.

Fig. 3 illustrates contact resistance changes on aging at 200°C of 50 µm thick Hard Golds having the same (0.25%) cobalt and nickel content. The nickel-gold is much more stable than the cobalt-gold deposit. Degradation at 200°C is more rapid than at 150°C. The contact resistance of pure gold falls slightly after brief aging, an effect sometimes observed at 150 and 200°C with other deposits.

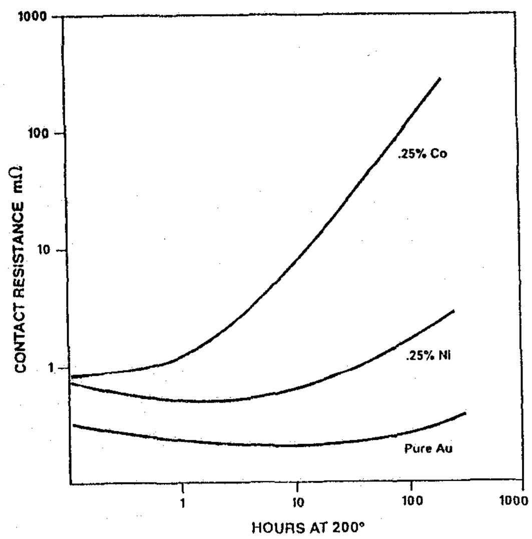

Fig. 3. Thermal stability of contact resistance at 1 N of 50 µm thick gold plate. Aged at 200°C. Effect of cobalt and nickel additions (0.25%).

Fig. 4 shows changes in the contact resistance of three cobalt-golds on aging at 150°C. The golds were 0.5 µm thick, and data are given without and with a nickel underplate. The 0.5% cobalt-gold deposit on copper has a higher contact resistance than the 0.25% cobalt-gold. The contact resistance of the 0.07% cobalt-gold changes on aging at a different rate, having the lowest contact resistance on brief aging, then increasing beyond 1000 hours to the level of the 0.5% cobalt-gold. Nickel underplate appears to be advantageous for the 0.07% cobalt-gold past 1000 hours, but degrades the contact resistance of the other samples.

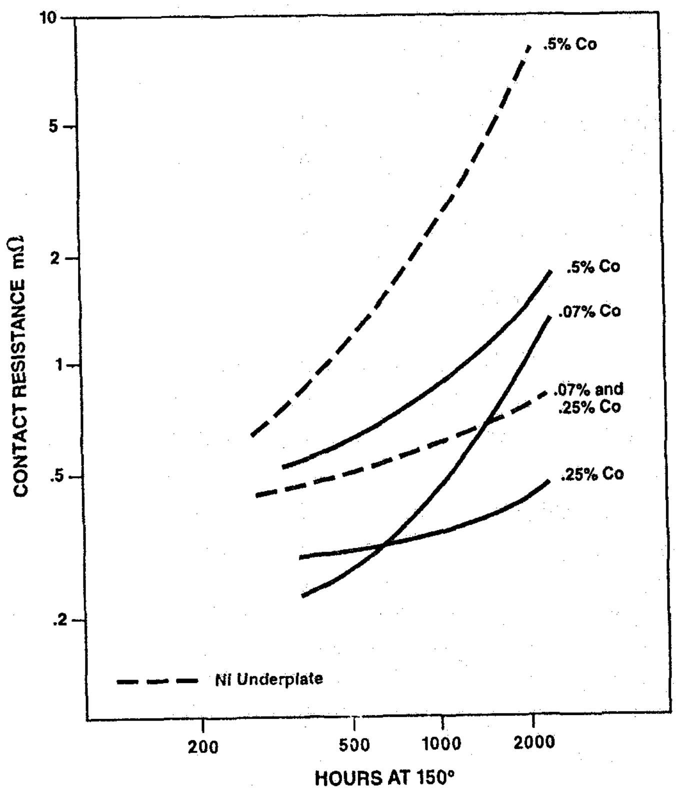

Fig. 4. Thermal stability of contact resistance at 1 N of thin (0.5 µm) Hard Gold plates on copper containing 0.07, 0.25, or 0.5% cobalt without (solid lines) and with (dashed lines) 2.5 µm nickel underplate. Aged at 150°C.

Fig. 5 compares the same golds of Fig. 4, but at a greater thickness, 2.5 µm. A significant effect is the degradation of contact resistance stability of the 0.25 and 0.5% cobalt-golds with nickel underplate, compared to the samples without the underplate. Also, the increases of contact resistance on aging are greater for these deposits than for the thinner platings of Fig. 4. However, the 0.07% cobalt-gold in Fig. 5 is benefited by the nickel underplate.

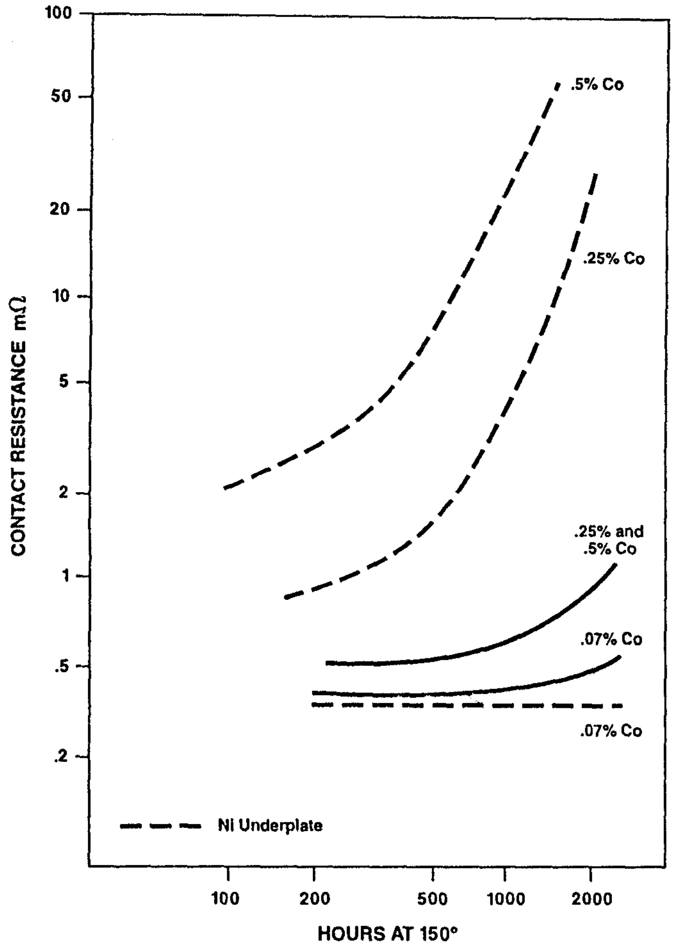

Fig. 5. Same as Fig. 4, except with thick (2.5 μm) cobalt-Hard Gold plates.

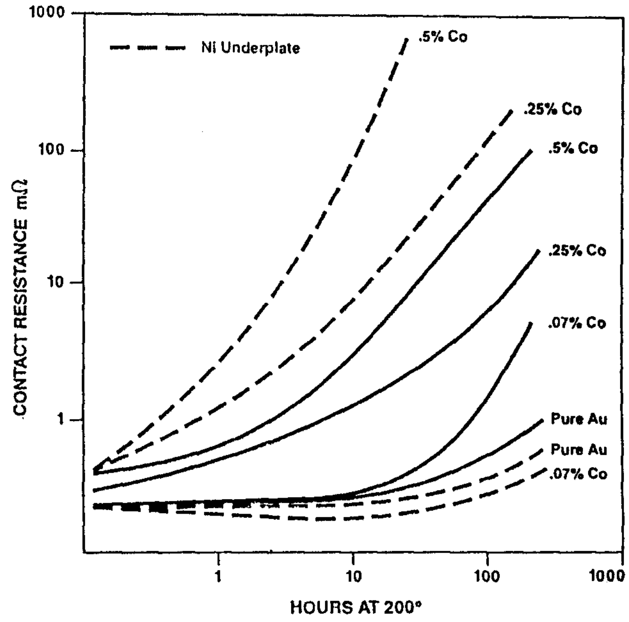

Fig. 6. Same as Fig. 4, except aged at 200° with thick (2.5μm) pure and cobalt-Hard Gold plates.

Fig. 6 is for aging at 200°C. Thick (2.5 μm) pure gold and Hard Golds with 0.07, 0.25, and 0.5% cobalt are compared, both without and with the nickel underplate. Cobalt additions to the gold degrade contact resistance, in agreement with results at lower temperatures, and to an increasing degree as the cobalt level is raised. Nickel underplate accentuates these contact resistance changes, except for the pure and the 0.07% cobalt-gold where it is of some value.

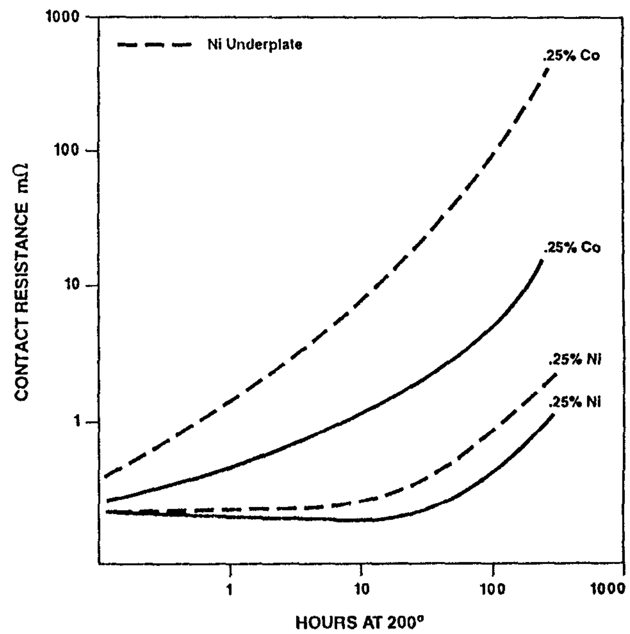

Fig. 7. Thermal stability of contact resistance at 1 N of 2.5 μm thick gold with additions of 0.25% cobalt or nickel, without (solid lines) and with (dashed lines) 2.5 μm nickel underplate. Aged at 200°C.

Fig. 7 compares 2.5 μm thick cobalt- and nickel-golds at the same percentage (0.25%) on aging at 200°C, without and with nickel underplate. The cobalt-gold is less stable than the nickel-gold. The underplate degrades behavior, especially that of the cobalt-gold.

IV. Discussion

A. Summary of Contact Resistance Results.

Empirical observations from this and the earlier study [19] by the author can be summarized:

1) Thermal aging in air of electrodeposited gold on copper can cause its contact resistance to increase. The longer the aging time and higher the temperature, the greater is the contact resistance rise.

2) However, contact resistance changes are small (at the conditions of probing) below about 125°C for 0.5 and 2.5 µm thick Hard Golds on aging for 1000 hours provided that the cobalt or nickel hardener content does not exceed a few tenths of a percent.

3) Contact resistance degradation may be significant after aging at 150°C and higher temperatures, especially for thick (2.5 µm) golds containing higher levels of cobalt.

4) Gold hardened with a small (0.07%) amount of cobalt behaves more like a pure gold than one containing much hardener.

5) Nickel-golds are more thermally stable than cobalt-golds having the same hardener content.

6) Nickel underplate is deleterious with golds containing higher hardener levels, especially cobalt, but can stabilize the contact resistance of pure gold.

7) The detrimental effect of nickel underplate increases with gold thickness at higher (0.25% and greater) hardener levels, particularly with cobalt-golds.

B. Mechanisms

Mechanistic studies to explain the empirical observations above were not made. Our current understanding of the structure of gold electrodeposits, of diffusion phenomena, the chemistry of film formations, and the mechanics of film displacement under contact load is incomplete. Nevertheless, some prior physical studies [4,5,17,18,35-44] can be related to this work.

1) Oxidation of base metals that have diffused to the gold surface results in the growth of insulating films which cause contact resistance, determined by probing, to increase. The diffused base metal originates in the substrate, underplate, or metals codeposited in the gold. Codeposited metals increase in amount with increase in their concentration in the plating solution.

2) Diffusion of base metals occurs by movement through the lattice or via defects in the gold such as dislocations, grain boundaries, and perhaps intrinsic pores. Diffusion through defects is much more rapid than lattice diffusion below about 300°C. The grain size of Hard Golds is less than that of pure gold, and the density of grain boundary diffusion paths is therefore greater.

3) The cobalt in gold is partly in solid solution and partly in a finely divided metal-organic complex containing potassium, carbon, and nitrogen which originates in $KAu(CN)_2$ in the plating solution. Nickel in gold from nickel doped bath chemistry has been presumed to be present in similar forms as those from cobalt. At sufficiently high temperature, the metal-organic complex in the gold decomposes. This is the origin of potassium compounds at the surface, found in some studies above 175°C. Carbon compounds may also contaminate the surface on heating, especially at very high temperature.

4) Insertion of a nickel underplate between gold and the underlying metal increases the diffusion path distance of substrate elements. Nickel from the underplate can diffuse, but it tends to form a self-limiting oxide at the gold surface and, therefore, is less deleterious to contact resistance than films from substrate metals such as copper. The net effect is that nickel underplate appears to be a "barrier" to the diffusion of substrate elements.

5) Codeposited cobalt and nickel, mainly as the intrinsic polymers, significantly increase the hardness and lower the ductility of the gold. This occurs by grain size reduction and by reduction of dislocation mobility.

6) The nickel underplate increases the composite hardness of the finish. This reduces its deformability when contact is made, and any insulating film on the surface is therefore less likely to fracture or be displaced to give a smaller contact resistance.

It is clear that there are interactive effects, some of the above factors serving to degrade contact resistance, while others are stabilizing. Thus, a rise in the hardener content of the gold increases the extent of film formation, especially with cobalt. An increase in the Hard Gold thickness also means that the source of hardener metal which can diffuse to the surface is greater.

The surprising deleterious effect of nickel underplate on the contact resistance of Hard Golds at the higher (0.25 and 0.5%) hardener metal levels can be explained by either of the following postulated mechanisms, one based on hardness (deformability) considerations, and the second based on relative diffusion rates:

1) *Hardness.* Nickel is a diffusion barrier for substrate copper, and with the tendency of any diffused nickel from the underplate to form self-limiting films, the net effect of aging is reduction of insulating film thickness -- and lower contact resistance -- compared to contacts without nickel underplate. This may be the reason why the nickel underplate is desirable for pure golds and those containing a small amount of hardener. But diffused species originating in gold deposit constituents instead of the substrate can be the prime source of surface films with high hardener levels. In this case, the hard nickel underplate is deleterious because of the "anvil" effect by making it more difficult to fracture the films when connection is made.

To clarify "anvil" effect, the hardness of cobalt-gold electrodeposits increases with increasing cobalt. It has been reported [45] to attain a maximum of about 215 kg/mm^2 at 25 g load (Knoop) with 0.3-0.4% cobalt. Nickel-golds are similar in this respect. The Knoop hardness of the pure, ductile nickel underplatings used on connector contacts is about 300-550 kg/mm^2 [13]. The hardness normal to the surface of multilayered finishes depends on the individual hardnesses and thicknesses, as well as on indentor load, of each layer [46]. Thus, the effect of inserting nickel underplate between gold plate and copper or other common substrate metals--nearly all of which are softer than the gold and nickel platings--is to reduce the deformability (the anvil effect) of the system.

2. *Diffusion.* Without the barrier layer the hardener species can diffuse both ways, to the external surface and into the substrate. The latter would reduce the concentration of the oxidizable species on the external surface and thus retard the growth kinetics of the resistive film. In the presence of the nickel barrier, the diffusion is only towards the external surface. Therefore, the amount of diffused metal at the gold surface (in the form of an insulating film) would be greater with the nickel underplate consequently with a more pronounced degrading effect on contact resistance.

V. Conclusions

The general conclusion is that at the conditions of this study, the most thermally stable Hard Gold electrodeposits on copper contain nickel (not cobalt) as the hardener. Such finishes with relatively thin (0.5 µm) golds and a nickel underplate have relatively stable contact resistance on thermal aging. Thin nickel-gold finishes may be acceptable for contacts in connectors at 150°C, and even to 200°C, depending on the intended duration of service. Connector design features, such as contact load, geometry, and wipe, and stresses during service like fretting, also play a major role in determining the suitability of contact finishes.

VI. Appendix

Assessment of Reproducibility of Contact Resistance Measurement.

Periodic measurements of the contact resistance of a control sample were made during the course of the study to check the reproducibility of probing technique. This takes into account both drift in the electrical and mechanical operations of the instrument and possible changes of the probe tip during use.

Two things which can occur to a probe that may drastically affect contact resistance are contamination by insulating material (oxides, dust particles, etc.), and roughening from use. Checks of a probe using a noble metal standard, such as a solid gold flat, while necessary, are less indicative of changes to the tip than checks with standards which consist of metal that is uniformly covered with a thin insulating film. The ability to penetrate the film is strongly dependent on the condition of the probe tip.

A satisfactory control sample is an oxide-covered metal that is both hard and environmentally stable. This requirement was met by a long-aged brass coupon coated with 15 µm of 65Sn35Ni alloy electrodeposit. The contact resistance, aging characteristics, and chemical stability of 65Sn35Ni have been reported earlier [47,48].

The control samples were stored in a covered container when not in use, and cleaned weekly in warm reagent grade methanol. The room in which the probe was located was maintained at 20-40% relative humidity and 23°C. Frequent checks of the milliohmeter and the loading mechanism of the instrument were also made.

Fig. 8 is a plot of the contact resistance values at 100 g of the 65Sn35Ni standard obtained over one year from 9 determinations at each interval. Control limits [49] for contact resistance were chosen within which it was assumed that the probe was operating satisfactorily. A log probability distribution plot of the log of the mean contact resistances is a straight line between 0.35 and 0.9 ohm. If the probe failed the check, contact resistance data obtained on the test samples between that determination and the previous control check were questioned and were generally discarded. It was necessary occasionally to replace a probe, and the same control limits were found to be applicable to all probes of the same type.

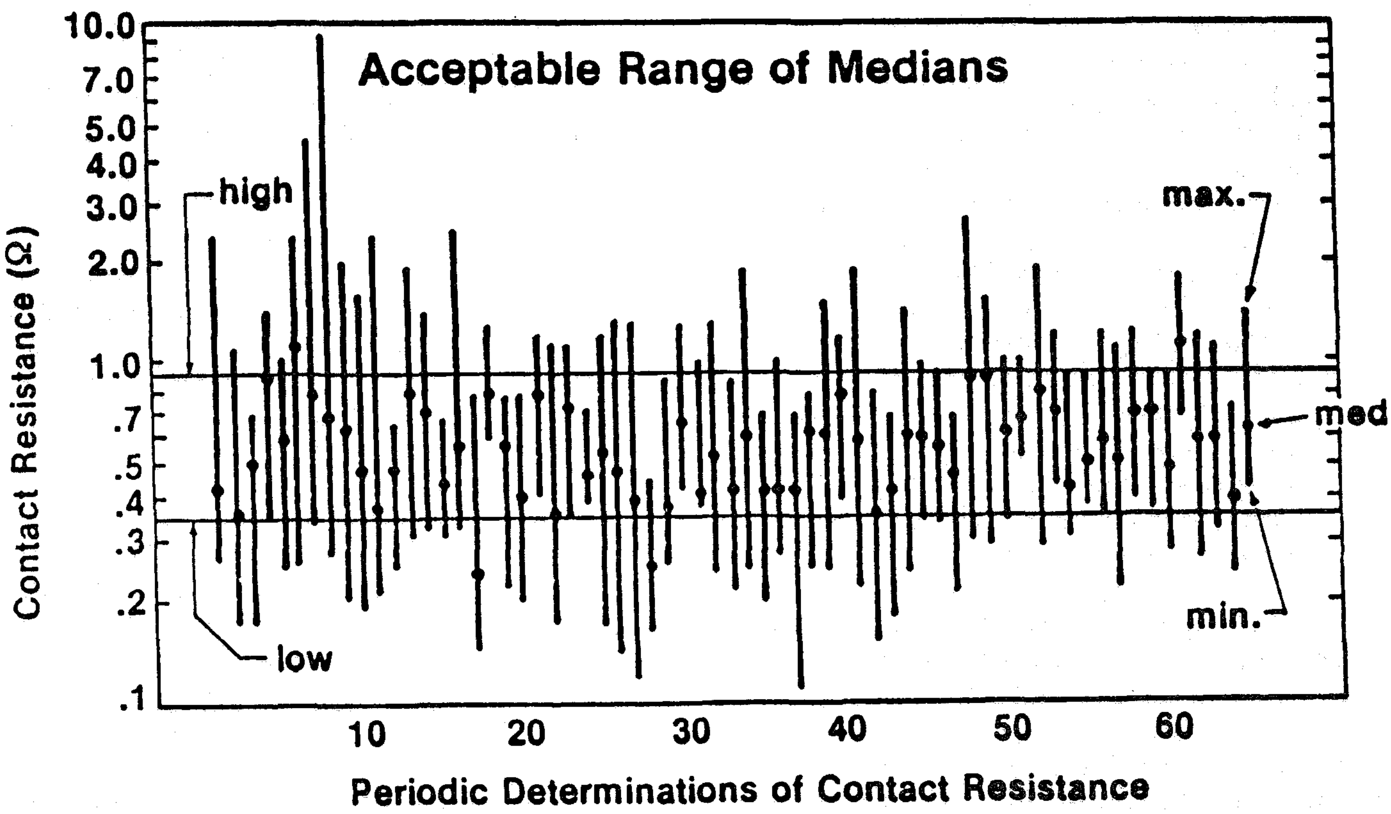

Fig. 8. Contact resistance at 1 N of a plated 65Sn35Ni standard, determined over one year. Nine determinations at each time interval. The control limits are 0.35 and 0.9 ohm. Any mean of contact resistances that lie outside of this range is cause for rejection of the probe.

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