N72-25688
NASA CR 126973

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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Prepared Under Contract No. NAS 7-100
National Aeronautics and Space Administration

PREFACE

The work described in this report was performed by the Engineering Mechanics Division of the Jet Propulsion Laboratory.

ACKNOWLEDGEMENT

The author would like to thank Marjorie Bickler for the preparation of all test specimens and her valuable assistance throughout the entire program.

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ABSTRACT

Ten different conductive inks used in the fabrication of thick-film circuits were evaluated for their physical and handling properties. Viscosity, solid contents and spectrographic analysis of the unfired inks were determined. Inks were screened on ceramic substrates and fired for varying times at specified temperatures. Selected substrates were given additional firings to simulate the heat exposure received if thick-film resistors were to be added to the same substrate.

Data is presented covering the (1) printing characteristics, (2) solderability using Sn 63 and also a 4% silver solder, (3) leach resistance, (4) solder adhesion, and (5) wire bonding properties. Results obtained using different firing schedules were compared.

A comparison was made between the various inks showing general results obtained for each ink. The changes in firing time or the application of a simulated resistor firing had little effect on the properties of most inks.

I. INTRODUCTION

A. Purpose

The purpose of this task was to determine the characteristics of different conductive inks intended for the making of thick-film circuits, and to investigate the effect of various firing schedules on the solderability, leach resistance, and bonding properties of conductors made from these inks.

B. Scope

The program involved the testing of ten different inks of four types from two different vendors. The four types of inks included: (1) gold, (2) platinum gold, (3) palladium gold, and (4) palladium silver. The actual identification of the inks, together with their manufacturers are available from the author upon request.

An average of 180 ceramic substrates were printed with each ink. The suitability of each ink for printing fine lines and spaces was evaluated. The printed substrates were fired for varying lengths of time at the vendor's recommended temperature. A portion of the fired substrates were also given additional firings to simulate the heat exposure received if thick-film resistors were to be printed and fired on the same substrate.

Testing consisted of solder coating the substrates from eight of the ten inks (gold inks were not solder-coated) using two different solders, and determining (1) the most suitable flux and soldering temperature, (2) leach resistance of the inks, and (3) adhesion of solder-coated pads when pull tested at both 45° and 90° angle. The wire bonding properties of the fired inks were also tested. Resistance measurements were made on fired conductors. Spectrographic analysis, viscosity, and solid contents of the inks were determined.

C. General Information

1. Procurement. A total of ten different conductive inks were procured from two separate vendors. One ounce of each ink was obtained. Materials were thoroughly stirred before use and maintained on a roller mill when not being used, unless specifically prohibited by manufacturer's directions.

2. Type of Inks. The inks were of four different types, as can be seen from the spectrographic analysis. The number of the ink, together with type, are as follows:

(1) Palladium-Gold: Ink Nos. 1 and 2.

(2) Platinum-Gold: Ink Nos. 3, 4, and 5.

(3) Palladium-Silver: Ink Nos. 6, 7, and 8.

(4) Gold: Ink Nos. 9 and 10.

II. TESTING OF BULK INKS

A. Viscosity of Inks

1. Test Procedure. Viscosity determination was run on the ink as received prior to any other tests. Viscosity tests were run on a Brookfield Model HBT Viscometer used with a No. SC4-1416 small sample adapter and spindle. The temperature of the material during test was maintained at 26.6 °C (80 °F), using a Haake Model NE circulating water bath. Tests were run at several different spindle speeds to determine thixotropy.

The ink container was opened, and the sample thoroughly mixed for 2 min. using a small spatula. Immediately after mixing, the chamber was loaded with approximately 2 cm^3 of the material. The spindle was inserted, and the level of material adjusted as necessary. The sample was allowed to stand for 15 ±1 min. before starting viscosity check in order to stabilize the sample temperature and viscosity. The speed control was set to 0.5 rpm and the viscometer motor started. At the end of 60 s the viscosity was read and recorded, and simultaneously the speed was increased to the next higher test speed. This higher speed was again maintained for 60 s before taking the viscosity reading. Again the speed was increased with the viscosity readings being taken at each increment in speed after a 60-s interval. When it was obvious that increasing the speed to a higher point would exceed the scale of the instrument, the speed was then decreased, going to the next lower speed and taking the readings at 60-s intervals, and repeating this procedure until the lowest speed was again reached. Viscosity readings obtained at increasing speeds, and decreasing speeds were compared.

2. Results. All of the viscosity readings obtained are shown in Table 1. Ink Nos. 7 and 9 were not tested, as Ink No. 7 was not available at the time the viscosity tests were run, and the bottle of ink was opened and used before it was realized that the viscosity tests had not been accomplished. Ink No. 9 was a very heavy, thick paste, and the viscosity test was not feasible. In all of the inks, the viscosity at the slow speed at the start of the test was different from the viscosity obtained at the slowest speed at the end of the test. The change was particularly great with Ink Nos. 3 and 4. Ink Nos. 3 and 4 increased greatly in viscosity as a result of stirring whereas

the viscosity on most of the remaining inks ended up close to the original starting point, or else decreased slightly in value. There appeared to be no definite correlation between the viscosity readings and the results obtained in actual printing of the inks.

B. Solids Content

1. Test Procedure. Porcelain crucibles and lids were cleaned, dried for 15 min., and then fired in a muffle furnace for 4 h at 1,000°C. Crucibles and covers were used as matched pairs and weighed together on the laboratory balance. The inks to be tested were thoroughly stirred prior to weighing 1 to 2 g in each crucible. The respective cover was placed over each crucible immediately after weighing of the ink. The uncovered crucibles were placed, together with their respective covers, in a cold muffle furnace, which was then heated to 125°C for 4 h. At the end of this time, the crucibles and covers were removed, allowed to cool, and the covers replaced on their respective crucibles, and again weighed. The percent loss in weight was arbitrarily termed, "volatile content". The crucibles and covers were again placed in the muffle furnace, and heated to 450°C for 4 h. The crucibles and covers were removed, and again weighed following the same procedure as before. The percent loss in weight after this firing was arbitrarily termed, "percent organic binder". Crucibles and covers were again weighed and the amount of loss determined. This percent loss was denoted as "percent weight loss on firing".

2. Test Results. The test results are shown in Table 2. Differences among the various inks appeared to follow no specific pattern. One of the greatest differences noticed was that the two gold inks had a much lower percentage of volatile material than the other inks tested. The solids content of all inks agreed quite closely with the results obtained when determining loss on ignition during the spectrographic analysis of the same inks.

C. Spectrographic Analysis of Conductive Inks

1. Procedure. The spectrographic analysis of all inks was conducted by the Pacific Spectro-Chemical Laboratory, Los Angeles, California.

2. Test Results. A complete analysis of all inks tested is shown in Table 3. The differences in the major constituents of the inks are relatively

minor in the palladium-gold inks. Constituents of the palladium-silver inks are also quite similar, except for Ink No. 8, where the amount of palladium is a great deal less than that contained in Ink Nos. 6 or 7. This is explained by the fact that Ink No. 8 is from a different vendor than Ink Nos. 6 or 7. Platinum-gold inks are all quite similar, and the biggest difference in the gold inks lies in the amount of gold contained in each ink.

III. PREPARATION OF TEST SPECIMENS

A. Artwork

1. Dimensions. The ink test pattern used in the testing of all conductive inks is shown in Fig. 1 and in the photos of the applied inks. Figure 1 is an enlarged print of the actual negative used in fabrication of the screens for printing. The size and location of the pads used for wire bonding, solder adhesion, and solder leaching tests are shown in Fig. 1. The lines and spaces running in both the horizontal and vertical directions are identical and consist of three groups of lines, with each group having a different spacing between the lines. The spacing is detailed in Fig. 1 and is 106 μ (0.0042 in.) in the first group of lines, 185 μ (0.0073 in.) in the center group, and 264 μ (0.0104 in.) in the third. Each group consists of six lines of varying width measuring 523 μ (0.0206 in.), 287 μ (0.0113 in.), 129 μ (0.0051 in.), 51 μ (0.002 in.) and 523 μ (0.0206 in.). The large spaces between the groups of lines were varied in size in an effort to make each group of lines correspond with the mesh openings in a 325-mesh screen.

2. Fabrication. The master artwork was fabricated using Stabilene "Cut N Strip" film at a 10:1 enlargement on an "Epod" coordinatograph. The master was photographically reduced to the desired 25.4 mm × 25.4 (1 in. × 1 in.) size by Century Graphics, Inc., Los Angeles, California.

B. Substrates: Type and Preparation

The ceramic substrates used were 25.4 mm square by 0.635 mm (0.025 in.) thick made of 96% Al_2O_3 obtained from the American Lava Co. All substrates were checked for warpage using a dial indicator accurate to 2.5 μ (0.0001 in.). Substrates were placed on a flat parallel surface and the distance measured from the top of the substrate to the parallel surface. The substrate was then turned over and the distance again measured. Substrates were not used if the difference in the measurements between the concave and convex side of the substrate was greater than 51 μ (0.002 in.). The concave side of the substrate was identified, so that all printing could be done on the convex side. Substrates were cleaned in 1, 1, 1 trichloroethane in an ultrasonic cleaner for 3 min. The substrates were then removed, allowed to drain for a few seconds, and rinsed in a clean container of 1, 1, 1 trichloroethane. After cleaning, the substrates were not handled with the bare hands.

C. Screen Preparation

1. Cleaning and Preparation. 325-mesh, stainless steel screens, having a wire diameter of 28 μ (0.0011 in.) and a mesh opening of 51 μ (0.002 in.) were used for all printing. These screens were obtained from Industrial Reproductions, Inc. (their No. 700), and were 127 mm × 127 mm (5 in. × 5 in.) in size.

Screens were cleaned by hand scrubbing with cleanser, given a thorough water-rinse and, finally, a rinse in an ultrasonic tank for three min. using hot, deionized water. Screens were allowed to dry, and then coated with "Azocol R".

The "Azocol R" was sensitized in accordance with the manufacturer's directions, and thoroughly stirred prior to use. A small amount of the mixed emulsion coating was applied along one edge of the screen and distributed evenly within the mesh of the screen by means of a plastic squeegee. The screen was then turned over and the emulsion distributed on the inside of the screen, using one or two strokes of the squeegee. The sensitized emulsion completely filled the screen mesh, and was equal in thickness to the thickness of the wire mesh. No attempt was made to apply emulsion above or below the plane of the wire mesh. The sensitized emulsion was allowed to dry at room temperature for a few hours prior to exposure and development.

2. Exposure and Development. The coated screen was placed over a red-light table, and the negative of the conductive pattern located in the center of the screen. A microscope was used to position the negative correctly, so that the edges of the lines and squares were lined up as accurately as possible with the edges of the wires comprising the screen mesh. The negative was then taped in place and exposed to a mercury vapor light source for a predetermined time period. The light source used was a nu Arc "Flip-Top" plate-maker. The actual exposure setting on this particular piece of equipment was 0.833. After exposure, the screen was soaked in warm water for 2 min., and then the unexposed emulsion was washed from the screen mesh, using a fine, high-pressure stream of water. The screen was examined under a microscope to make certain that the lines were sharp and clear, and that all of the undesired emulsion had been removed. The

completed screen was dried overnight at room temperature prior to any printing operations.

D. Printing

1. Procedure. The substrates used for all tests were printed on a Presco Model 150 Printer using their standard squeegee setup. The process parameters were varied for each ink in order to obtain the most satisfactory printing results, but, in general, are listed below:

(1) Squeegee: polyurethane; Shore hardness-80A.

(2) Break-away distance: 762 to 1,016 μ (0.030 to 0.040 in.).

(3) Squeegee pressure: 1,359 to 2,718 g (3 to 6 lb).

(4) Squeegee speed: 76 mm (3 in.) per s.

All of the inks tested, except No. 6, were printed using the above process parameters. Ink No. 6 was extremely difficult to print, and it was found that the best results could be obtained using contact printing. Ink No. 6 was printed using a squeegee pressure of 1,472 to 1,925 g (3 1/4 to 4 1/4 lb), with a break-away distance of 51 μ (0.002 in.).

The actual squeegee pressure on the Presco 150 Printer is controlled by an air-pressure regulator. It was found that uniform pressures were very difficult to obtain, particularly the required low pressures of 1300 to 2700 g. As a result of this experience, the decision was made to obtain the Presco CS-1 squeegee head, in which the squeegee pressure is controlled by spring tension. The new squeegee head was obtained, installed, and all inks reprinted using the new squeegee head. The process parameters were approximately the same, except a squeegee pressure setting of 2 was used. This pressure setting was equivalent to approximately 3,100 g (7 lb) pressure. Comments concerning the printability of the various inks are based on the results obtained when using the new CS-1 squeegee head.

The printed substrates were allowed to set at room temperature for 5 to 10 min., and then dried in a small conveyorized infrared oven. The conveyor speed and temperature of the infrared oven was checked using a thermocouple. Each substrate was dried for approximately 15 min. at a temperature of 100°C.

2. Printing Characteristics and Results. The printing characteristics of the ten different inks are shown in Table 4. These results were obtained under one specific set of conditions using a certain type of equipment, and may not be representative of the results obtained when using different equipment, or other process parameters.

Evaluation of the printing results obtained, are also listed in Table 4. Printing of the 51 μ (0.002 in.) line was not satisfactory with any of the inks tested. With the possible exception of Ink Nos. 9 and 10, the lines printed using the 102 μ (0.004 in.) spacing between lines would not be considered satisfactory. While in most cases, the lines did not run together, the ink did spread enough that the space between the lines would be considered inadequate for normal usage. Photographs of substrates printed with the various inks are shown in Figs. 2, 3, and 4. The photos are quite typical of the results obtained.

The dry-film thickness was checked on two substrates of each ink using a light section microscope. Readings were taken at seven different points on the substrate and averaged. After firing, the thickness of the ink was again determined at the same relative locations on the substrate. No attempt was made to obtain the fired thickness at the precise point on the substrate at which the dry-film thickness was obtained. The average dry-film thickness and fired thickness for the two substrates tested are shown in Table 4.

E. Firing Schedule and Identification

All substrates were identified with a number/number/letter combination with the following code:

(1) First number: Identification number of the ink.

(2) Second number: Indicates length of time of the basic firing schedule. Substrates were fired for either 5, 10, or 20 min. at the temperature shown under A below.

(3) Last letter: Indicates whether the substrate received only the basic firing schedule or received a simulated resistor firing in addition to the basic schedule.

A = basic firing schedule only. Inks were fired at the following temperatures (temperatures shown are vendors recommendations):

Ink Nos. 1, 6, 7, 8	850°C
Ink Nos. 4, 5, 9, 10	900°C
Ink Nos. 2, 3	950°C

B = inks received the basic firing schedule as in A plus an additional firing of 10 min. at 775°C.

C = inks received the basic firing schedule as in A plus an additional firing of 10 min. at 900°C.

Example: Substrate marked:

fired for 20 min. at 850°C
↓
Ink No. 6 → 6-20-B
↑
received basic firing schedule of 850°C plus 10 min. at 775°C.

All substrates were identified using a ceramic pencil whose markings were resistant to the furnace temperature.

The actual number of substrates used for each test together with the various firing schedules and simulated resistor firings are shown in Tables 5, 6, and 7.

F. Firing of Printed Substrates

1. Equipment. All substrates were fired at the time and temperature indicated using a conveyorized four-zone furnace with quartz muffle and inclined 2 deg from the horizontal.

2. Firing Profile. Prior to firing any substrates, the temperature in the four-zones of the furnace were adjusted, as necessary, to obtain the desired temperature profile. The profile was determined by placing 30 dummy substrates on the belt, followed by a Chromel/Alumel "Type K" thermocouple. Temperature data was recorded on a chart recorder. Adjustment of both the temperature in the various zones, and the speed of the

conveyor, were made until the desired profile was obtained. Requirements for these profiles were as follows:

(1) Peak temperature to be within $^{+5}_{-0}$ °C of target temperature.

(2) The time at or between the target temperature (T) and the target temperature minus 15°C (T-15) shall be: (a) 5 ±1/2 min., (b) 10 ±1/2 min., or (c) 20 $^{+0}_{-1}$ min. as indicated by the various firing schedules that the substrates are subjected to. The simulated resistor firings profiles of 10 min. at 775°C or 10 min. at 900°C were obtained in the same manner, and within the same requirements regarding peak temperature and time as outlined above.

3. <u>Firing Procedure</u>. Immediately after a satisfactory firing profile had been obtained, the conveyor was loaded with 30 dummy substrates followed by the printed substrates being fired. Those inks requiring the same firing temperature were fired at the same time wherever possible.

IV. TEST PROCEDURES AND RESULTS

A. Electrical Resistance

1. Test Procedure. The resistance of the 508 μ (0.020 in.) line was determined for each ink on two separate substrates. The average width of the printed line was measured with a Wilde Microscope using a micrometer eyepiece attachment. Test probes were placed at the end of each line and the resistance determined with a digital voltmeter. Length of each line was 23.8 mm (0.940 in.).

2. Results. The average width of the lines together with the resistance is shown in Table 4. Resistance of the gold inks was especially low.

B. Solder Coating of Substrates (63% Tin, 37% Lead Solder)

1. General Procedure. Conductive inks on the substrates were solder coated with 63% tin/37% lead solder using a wave soldering machine from Hollis Engineering Corporation. The substrates were held in a horizontal holder and passed through the solder wave at varying speeds, as described in the detailed tests. The solder wave employed a thin film of oil on the surface to reduce dross and to smooth out the wave.

2. Preliminary Tests. Preliminary tests were run on substrates from each of the eight inks (gold inks were not solder-coated) being tested in order to determine the best possible combination of solder temperature, flux and conveyor speed for coating the various inks. A matrix of tests was set up, employing the following variables:

(1) Flux: A -- Lonco No. 5177
B -- Alpha No. 100
C -- Alpha No. 611
D -- Alpha No. 711

All fluxs were reduced 3 to 1 with Ethyl Alcohol.

(2) Solder temperatures used in the preliminary tests were: 205°C, 215°C, 225°C, and 240°C.

(3) Conveyor speeds used were: (a) speed setting of 5, which is equivalent to 25.4 mm (1 in.) of travel in 6.45 s, and (b) speed setting of 20, which is equivalent to 25.4 mm (1 in.) in 1.83 s.

Substrates soldered using combinations of the above process parameters were examined and evaluated. Based on these preliminary tests, the following process parameters were established for each ink:

Ink No. 1: Solder temperature of 215°C at a conveyor speed setting of 20 using Alpha No. 711 flux.

Ink No. 2: Solder temperature of 215°C at a conveyor speed setting of 20, using Lonco 5177 flux.

Ink No. 3: Solder temperature of 215°C at a conveyor speed setting of 5, using Lonco 5177 flux.

Ink No. 4: Same process parameters as Ink No. 3.

Ink No. 5: Solder temperature of 225°C at a conveyor speed setting of 5, using Alpha No. 711 flux.

Ink No. 6: Solder temperature of 205°C at a conveyor speed setting of 20 using Alpha No. 711 flux.

Ink No. 7: Solder temperature of 225°C at a conveyor speed setting of 5, using Alpha 711 flux.

Ink No. 8: Solder temperature of 215°C at a conveyor speed setting of 20 using Lonco 5177 flux.

3. Solder Coating and Leaching Procedure. Five substrates of each combination of ink and firing procedure (see Tables 5, 6, and 7) were solder-coated following the procedure and process parameters listed above. After the first solder coating, three soldered substrates of each combination were set aside for solder adhesion or other tests. The two remaining substrates of each combination were given additional passes through the solder wave in order to determine the extent of leaching that occurred. Substrates were evaluated after each pass through the solder wave. The test was generally discontinued when a minimum of 50% leaching of the lines or pads occurred.

4. Test Results.

a. Ink No. 1. The first solder coat was uniform and shiny with a few pinholes appearing. The 20B's and 20C's were not shiny, but uniformly coated. The second solder coating caused approximately 2% leaching of the fine lines on all the schedules. The solder-coated pads appeared smoother and flatter with no noticeable leaching. The third solder coating leached the large pads and lines above 15% to 20%. The small pads throughout all schedules remained completely intact. No difference was noted between the various schedules. The fourth solder coating caused 30 to 40% leaching throughout the entire schedules. The different firing times appeared to have no effect on the leach resistance of this ink. The 5's, 10's and 20's in systems A, B and C were equally leached.

b. Ink No. 2. The solder was shiny, although excessive solder was apparent where it had bridged across lines. The second solder coating caused approximately 10% leaching of the lines and the large pads. The small pads were still shiny and intact. The third solder coating caused about 15 to 40% leaching of the large pads, and about 90% leaching of the lines. Some of the small pads were still completely intact. The fourth solder coating caused leaching of more than 50% on the large pads, and 90 to 95% leaching of the lines. The different firing times appeared to have no effect on the leach resistance.

c. Ink No. 3. The first solder coating was shiny and uniform with no pinholes except for the 20A, 20B, and 20C schedules, where the solder had a dull appearance. The second solder coating caused 5% leaching of the pads and 10 to 15% leaching of the lines. The third solder coating caused 10% leaching of the pads and 20 to 25% leaching of the lines. After the fourth solder coating, the pads were still holding up remarkably well, with about 15% leaching throughout all the schedules. Holes had appeared in the solder coated lines, and approximately 50% leaching had occurred. The different firing times appeared to have no effect on the leach resistance.

d. Ink No. 4. The first solder coating was shiny and uniform on the small pads. The large pads were alternately bright and dull. Pinholes were scattered throughout the large pads on all schedules. The solder had flowed across the closely printed lines. The second solder coating caused leaching of 5 to 15%. General appearance of the solder changed very little from the first coating. The third solder coating caused 75 to 95% leaching of the lines with 10 to 30% leaching of the pads. Some of the smaller pads were still virtually untouched. No differences in leaching were noted between the various schedules.

e. Ink No. 5. The first solder coating was shiny and uniform, although there was a large accumulation of solder between the lines. The second solder coat did not cause any leaching, but appeared to smooth the pads and eliminated many of the large accumulations of solder on the run-together lines. The third solder coating caused 5 to 10% leaching of the lines. The pads were practically untouched, although a few did show about 10% leaching. The fourth solder coating caused 50% leaching of the lines, and up to 30% leaching of some of the pads. No differences in leaching were noted between the various schedules.

f. Ink No. 6. The first solder coating appeared uniform on the 5 and 10 schedules with some scattered leaching of the small pads occuring on the 20B and 20C schedules. The second solder coating caused 10 to 40% leaching of the 10 schedules, 25 to 60% leaching of the 5 schedules and 60 to 75% leaching of the 20 schedules. Due to the bad leaching, no further solder coating was done.

g. Ink No. 7. The first solder coating was uniform on the 5 and 10 schedules, but spotty with random large and small pads unsoldered on the 20 schedules. The second solder coating caused almost no leaching, even on the lines. The pads did not appear as shiny as after the first coating and had a mottled, irridescent appearance. The third solder coating caused 85 to 95% leaching of the lines with both large and small pads being leached from 50 to 100%. There were scattered pads, however, that were leached very little.

h. Ink No. 8. A shiny, uniform solder coating was obtained on all schedules. The second solder coating caused 75 to 90% leaching on the 10 schedules, 5 to 40% leaching on the 5 schedules and 5 to 50% leaching on the 20 schedules. The third solder coating caused 98% to 100% leaching on all schedules.

C. Solder Coating of Substrates (60% Tin, 36% Lead, 4% Silver)

1. Solder Coating and Leaching Procedure. The first solder coating of Ink Nos. 5, 7, and 8 was done by loading the substrates onto a conveyor and passing them through the solder wave. Solder temperature was 243 °C with a conveyor speed of 25.4 mm in 7-1/2 s. After substrates in the first three inks had been coated, difficulty was experienced with the solder wave

equipment, and it was found impossible to obtain a uniform solder wave. The remainder of the substrates were coated by holding the substrates individually with a pair of tongs, and immersing the substrate completely into the solder wave for 3 s. Leaching tests were done in the same manner, i.e., by immersing the substrate in the solder wave for varying periods of time. The immersion times used for the leaching tests were 3 s, unless otherwise indicated.

The flux used in all tests with the 4% silver solder was Lonco 5177 flux, thinned 3 to 1 with ethyl alcohol.

2. Test Results.

a. Ink No. 1. The solder was bright and shiny on the 10 and 20 schedules although it appeared slightly tarnished on the 5 schedules. Solder-coated lines were sharp and clean. The second solder coat dulled the shininess of the solder, but leaching did not appear to occur. The third solder coat again caused no leaching although the lines flattened out on all schedules. Solder appeared more grainy, but complete coverage was still apparent. No leaching occurred on the fourth, fifth or sixth immersion in the molten solder, even though the immersion times were increased to 10 to 15 s on each dip. The solder did lose its shiny appearance and seemed very dull looking. The seventh immersion in the molten solder was for 20 s and leaching did start on the lines during this immersion.

b. Ink No. 2. The first solder coat was fairly shiny on all schedules with no noticeable difference between them. In all cases, however, there was either insufficient coverage or leaching of a 25 to 50 μ border around every line and pad. The second coat solder appeared to fill in this 25 to 50 μ border around the lines and pads with no evidence of leaching. The third solder immersion caused very little change in appearance, although the lines did flatten out and the solder appeared more grainy on the 10's and 20's. Solder on the 5's was still shiny, although duller than on the second dip. The fourth, fifth, and sixth immersions in the molten solder caused no leaching, even though immersion times were increased to 10 to 15 s. Solder appearance was generally mottled and dull.

c. Ink No. 3. The first solder coating was bright and shiny on the 10's and 20's, but the 5's had a tarnished appearance. There was good solder coverage of the lines and pads. Substrates were given a total of six immersions in the molten solder, with the times being increased from 10 to 15 s per immersion. No leaching of the lines or pads occurred, although the solder did become grainy, bumpy and dull.

d. Ink No. 4. The first coat of solder had a dull, flat, tarnished, and grainy appearance on all schedules. The second coat of solder did not cause any change in appearance, although there was no leaching. The third coat of solder caused very slight leaching of the lines, and some pads in the C schedules. After the fourth coat of solder, the lines in all schedules were leached. Lines in the 5 schedules were 50% destroyed while lines in the 10's and 20's schedules were 10% destroyed. After the fifth coat of solder, the 5's were 70 to 90% leached, while the 10's and 20's were 25% leached.

e. Ink No. 5. The first coat of solder was very grainy although coverage was good. No difference was noted in the different schedules. The second coat of solder changed only the general appearance of the solder as it became mottled. The third coat of solder had the appearance of a "leaden" look. No leaching occurred. No leaching occurred on the fourth solder coating. Even portions of the 51 μ (0.002 in.) lines which had printed showed no leaching. The fifth and last solder dip was made extremely slow. No leaching occurred, and the ink appears impervious to leaching.

f. Ink No. 6. Appearance of the first solder coat was bright and shiny, but solderability was very random as some lines were coated, while others were not. Leaching occurred on the second coat of solder, with the 5 schedules leached 50%, the 10 schedules leached 20%, while the 20 schedules looked the best. The third coat of solder caused complete leaching of all 5's together with the C's in both the 10 and 20 schedules. Leaching of the other schedules was very erratic with no discernable pattern.

g. Ink No. 7. The first solder coating was brilliant and smooth with excellent coverage of lines and pads on all schedules. The second solder dip showed almost no change, except the solder began to look a little frosty in random spots. No change was noted after the third dip in solder. The 51 μ (0.002 in.) partially printed lines were still intact. The fourth and fifth dips in molten solder also produced no leaching.

For the sixth dip in molten solder, the time was lengthened to 15 s. The 5C's, 10C's and 20C's lost about 20% of the fine lines and small squares. The remainder of the schedules appeared unaffected.

h. Ink No. 8. The solder was bright, smooth and shiny with excellent coverage of lines and pads in the 5 and 10 schedules. These schedules appeared to have a sharper line coverage than the 20's. The "20" lines were not as well covered, and had a more grainy appearance although the pads showed no difference between the 5's, 10's, or 20's. The second solder dip showed almost no change from the first. A few spots in the solder coating began to look slightly frosty. No change was visible after the third or fourth solder dip in the molten solder. It appeared as if repeated dips in the molten solder improved the appearance of this system. The fifth and sixth dips in the molten solder were lengthened to approximately 15 s. Slight leaching - approximately 10%- now appeared throughout the entire schedule with the leaching primarily being evident in some of the fine lines and smaller squares. No difference was noted between the various schedules.

D. Adhesion of Solder Coated Inks

1. General Procedure Using 63% Tin, 37% Lead Solder. Two substrates from each firing schedule for Ink Nos. 1 thru 8 were given a single coat of Sn 63 solder using the process parameters outlined in Subsection B-2 of this section.

Strips of nickel ribbon 0.25 mm (0.010 in.) thick by 0.50 mm (0.020 in.) wide were tinned with Sn 63 solder. The tinned strips were reflow soldered in a horizontal position to the solder coated 1.27 mm (0.050 in.) by 1.27 mm (0.050 in.) pads on the substrates. A Unitek 1-137-01 reflow solder system was used with the following process parameters:

Power Setting - Low
Temperature - 315°C
Duration - 7
Pressure - 566 g (20 oz)
Amplitude - 900
Heat Control - Off
Electrodes - R.W.M.A. No.2, 0.61 × 0.61 mm (0.024 × 0.024 in.)
Gap - 0.38 mm (0.015 in.)

Five strips were soldered to each substrate. The strips on one substrate were carefully bent to a 90° angle and pulled at 90° to the plane of the substrate using a Hunter Pull Tester calibrated in ounces and run at a speed of 25.4 cm (10 in.) per min.

The five strips on the second substrate were carefully bent to a 45° angle and pulled at 45° to the plane of the substrate using the same equipment.

2. General Procedure Using 4% Silver, 60% Tin, 36% Lead Solder. The general procedure for determining the adhesion of inks coated with 4% silver solder was the same except for the changes noted below.

(1) Substrates were coated with a single coat of 4% silver solder using the process parameters listed in Subsection C-1 of this section.

(2) Nickel strips were dip tinned using the 4% silver solder at a temperature of 243°C.

(3) Reflow soldering of strips to substrate used the following process parameters:

Power - Low
Temperature - 343°C
Duration - 9
Pressure - 566 g (20 oz)
Amplitude - 900
Heat Control - Off
Electrodes - R. W. M. A. No. 2, 0.61 × 0.61 mm (0.024 × 0.024 in.)
Gap - 0.38 mm (0.015 in.)

3. Results. Detailed results of all adhesion tests are listed in Table 8. The results are shown in both newtons and ounces, and are the average of the 5 pull tests made on each substrate.

E. Gold Wire Bonding

1. General Procedure. Process parameters were developed for both ball and wedge bonding of a 25.4 μ (0.001 in.) gold wire to the No. 10 gold ink. Wire tensile strength was 8.3 g. Equipment used was a Kulichi and Sofa Wire Bonder Model 420 with the following process parameters:

Heated column temperature - 320°C
Bonding dwell time - ball = 2.5 s; wedge = 1.25 s
Capillary Tip Force - 45 g
Ball Size - ±5.1 μ(0.0002 in.)
Capillary temperature - 320°C

Ten ball bonds and 10 wedge bonds were made to the rectangular pads of one substrate from each of the schedules. The process parameters for all inks were identical and as listed above. The bond strength of all inks were comparable to the No. 10 gold ink and no attempt was made to develop the optimum process parameters for each ink.

The 10 ball bonds and 10 wedge bonds were pulled using a Hunter Pull Tester measuring in grams.

2. Results. The results of all pull tests are shown in Table 9 and are an average of the 10 bonds tested together with the maximum + or - deviation from the average. The type of wedge bond failure was also noted for each of the bonds tested.

V. DISCUSSION AND COMMENT

A. General

1. Viscosity and Solids Analysis. No correlation was apparent between the viscosity and the printing results obtained. The ink composition was quite similar within the same type even though the inks were from different vendors. The primary purpose of obtaining the viscosity and solids analysis was comparison with future lots of identical inks. This data would provide a possible basis to account for any differences in handling or physical properties that might occur when using different lots of the same ink.

2. Printability. All inks were satisfactory for printing of 254 μ (0.010 in.) lines and spaces. Printing of 102 μ (0.004 in.) or 51 μ (0.002 in.) lines and spaces is a difficult operation requiring great control over all the process details involved in silk screen printing. No effort was made to explore all possible combinations of process parameters for each ink. It is possible that under the right conditions any ink could print fine lines and spaces in a satisfactory manner.

Ink Nos. 7 and 8 appeared to handle the easiest and gave good results in printing of fine lines and spaces.

3. Solderability. All inks tested could be solder coated in a satisfactory manner although gobs of solder occurred where lines had run together during the printing operation. Solder coated substrates from Ink Nos. 7 and 8 had the best appearance with the solder coated lines separated from one another while Ink Nos. 1 and 2 were the poorest.

Ink Nos. 7 and 8 also had the best solderability when using the 4% silver solder while Ink Nos. 2, 4, and 6 were the least satisfactory. No. 1 ink was borderline, especially in the 5 schedules.

4. Leaching. Noticeable leaching occurred with all inks particularly when using the Sn 63 solder. Ink Nos. 1, 5, and 7 had the best leach resistance with No. 6 being the poorest.

Leach resistance was remarkably improved when using the 4% silver solder. Leach resistance of all inks was excellent except Nos. 4 and 6 which were rated poor to fair.

5. Adhesion of Solder Coated Inks. Correlation of the data between the 45° and the 90° pull was very poor. In general, it appeared that greater force was required when pulling at a 45° angle.

Use of the 4% silver solder improved adhesion by varying amounts on all inks except Nos. 2 and 3 where results were quite similar, regardless of which solder was used.

Pull test data was erratic among the various schedules but Ink Nos. 4, 5, 7, and 8 had the best overall adhesion especially when using the 4% silver solder.

6. Gold Wire Bonding. Gold wire bonding to all inks appeared satisfactory with very little differences noted between inks. Minor differences in bond strength and type of bond failures were noted between the various schedules but no definite pattern could be noted.

B. Effect of Different Firing Schedules on Test Results

1. Solderability (Sn 63 Solder). The solderability of Ink Nos. 1, 3, 6, and 7 was slightly affected in those schedules fired for 20 min. at the recommended temperatures. Results were satisfactory on the 5 and 10 min. schedules. No differences were noted in the schedules of the other inks.

2. Solderability (4% Silver Solder). A tarnished appearance of the solder occurred on the 5 schedules of Ink Nos. 1 and 3. No differences were noted in the other inks.

3. Leach Resistance (Sn 63 Solder). Noticeable leaching occurred on all schedules, particularly when using the Sn 63 solder. Except for Ink No. 6, however, the leach resistance of the inks was not affected by the firing time or temperature they had been subjected to. The 20 schedules of Ink No. 6 leached the worst followed by the 5 schedules. The 10 schedules were the least affected.

4. Leach Resistance (4% Silver Solder). Leach resistance of Ink Nos. 1, 2, 3, 5, 7, and 8 were unaffected by the firing time or temperatures. The 5 schedules of the Ink Nos. 4 and 6 together with all the C schedules of Ink No. 6 appeared to leach more than the other schedules of the same inks.

5. Effect of Firing Schedules on Adhesion. Results of the 45° and 90° pull tests were erratic within the various inks fired at different times and temperatures. There appeared to be no discernable trend in the results that would indicate that adhesion was affected by firing time or temperature.

6. Effect of Firing Schedules on Gold Wire Bonds. The length of firing schedule or the simulated resistor firings appeared to have no effect on the strength of the gold wire bonds made to the different inks.

VI. CONCLUSION

A. Ink Evaluation and Comparison

No single ink gave the optimum results in all of the tests conducted. Selection of an ink for a particular job should be based on the relative importance of the different properties of each ink as shown in Table 10.

Ink Nos. 5, 7, and 8 were very satisfactory for use where solder coating is required while either of the gold ink (Nos. 9 or No. 10) would be acceptable without solder coating.

B. Effect of Firing Times

Changes in firing time or the application of a simulated resistor firing had very little effect on the properties of most inks. Any effects noted were primarily in the solderability and leach resistance of certain inks. The best firing schedule for all inks was 10 min. at the manufacturer's recommended temperature.

VII. MATERIALS AND EQUIPMENT

A. Materials

Ceramic substrates No. 614: 96% Al_2O_3, 25.4 × 25.4 × 0.63 mm (1 × 1 × 0.025 in.); American Lava Corporation, Chattanooga, Tennessee.

Conductive inks: identification of the inks is available from the author upon request.

Azocol "R" Photosensitive Emulsion: Colonial Process Supply Corporation, East Rutherford, New Jersey.

1,1,1 Trichloroethane per Federal Spec. 0-T-620.

Film, "Cut N Strip", Stabilene No. 445547: Keuffel & Esser Co., Morristown, New Jersey.

Flux, Lonco No. 5177: Distributed by Claude Michael Corporation, Glendale, California.

Flux, Alpha No. 100, No. 611 and No. 711: Alpha Metals Corporation, Jersey City, New Jersey.

Solder: Tin, 63%; lead, 37%.

Solder: Tin, 60%; silver, 4%; balance lead.

Nickel ribbon: 0.25 × 0.50 mm (0.010 × 0.020 in.), tinned as required.

B. Equipment

Printer: Presco Model 150 Printer with CS-1 Squeegee Head; Precision Systems Co., Somerville, New Jersey.

Furnace: Transheat Model 10-42-412 F; BTU Engineering Corporation, Waltham, Massachusetts.

Light Section Microscope: Model 604994; Zeiss Corporation.

Exposure Light: nu Arc "Flip-Top" Plate Maker, Model F. T. 26AM; nu Arc Co., Chicago, Illinois.

Printing Screens: No. 700, 12.7 × 12.7 cm (5 × 5 in.); Industrial Reproductions, Inc., Nashua, New Hampshire.

Wave Solder Equipment: Model 45000; Hollis Engineering Corporation, Nashua, New Hampshire.

Wire Bonder: Nailhead Wire Bonder Model 420; Kuliche & Sofa Corporation, Fort Washington, Pennsylvania.

Muffle Furnace: Lindberg Furnace, Type 59344; Sola Basic Industries, Watertown, Wisconsin.

Pull Tester: Hunter Spring Co., Lansdale, Pennsylvania.

Coordinatograph: Epod Model G-4848; Development Associate Controls Co., Santa Barbara, California.

Digital Voltmeter: Model NLS Series X-2; Non-Linear Systems, Inc., Del Mar, California.

Reflow Solder Equipment: Unitek 1-137-01; Unitek Corporation, Monrovia, California.

Viscometer: Brookfield Viscometer, Model HBT with small sample container and spindle SC4--14/6; Brookfield Laboratories, Stoughton, Massachusetts.

Ultrasonic Cleaner: Model LP-1HD, Electromation Components Corporation, Farmingdale, New York.

Table 1. Ink viscosity

Spindle Speed, rpm	Viscosity, N · s/m² (× 10³ cP)[a]									
	Ink No. 1	Ink No. 2	Ink No. 3	Ink No. 4	Ink No. 5	Ink No. 6	Ink No. 7	Ink No. 8	Ink No. 9	Ink No. 10
0.5	560	1,110	240	780	560	560	Not tested	500	Not tested (ink was very thick)	860
1.0	480	850	220	700	450	450		380		630
2.5	376	612	180	580	371	356		272		428
5	316	484	154	490	322	306		220		304
10	260	380	125	402	272	255		180		217
20	208	297	98.2	317.5	226	217		140		150
50	139	Off scale	69.2	Off scale	163.8	148.4		96		86
100	Off scale	Off scale	52.4	Off scale	—	—	Not tested	69	Not tested (ink was very thick)	—
50	134	Off scale	67.8	Off scale	163.8	148.4	Not tested	91.8	Not tested (ink was very thick)	86
20	199.5	288	96.9	132.5	218	216		126.5		141
10	250	355	135	405	263	249		158		202
5	300	430	194	520	310	294		200		280
2.5	364	518	296	660	363	348		244		379
1.0	450	680	540	880	450	440		360		560
0.5	540	960	900	1,080	540	520	Not tested	520	Not tested (ink was very thick)	780

[a]1000 Centipoises = 1N · s/m²

Table 2. Weight loss of conductive inks

Ink No.	Volatile content, % (4 h at 150°C)	Organic binder, % (10 h at 450°C)	Weight loss on firing, % (4 h at 900°C)	Total loss, %
1	18.34	0.5	0.502	19.42
2	13.89	3.43	2.72	20.05
3	18.88	0.22	0.380	19.46
4	17.41	1.32	1.71	20.45
5	12.87	2.95	0.563	16.40
6	22.08	2.88	3.05	28.02
7	20.15	5.53	1.97	27.66
8	19.78	8.03	1.18	29.00
9	5.66	1.86	0.124	7.64
10	9.90	5.44	0.274	15.75

Table 3. Spectrographic analysis of conductive inks

Element	Ink No. 1 Palladium-Gold, %	Ink No. 2 Palladium-Gold, %	Ink No. 6 Palladium-Silver, %	Ink No. 7 Palladium-Silver, %	Ink No. 8 Palladium-Silver, %	Ink No. 3 Platinum-Gold, %	Ink No. 4 Platinum-Gold, %	Ink No. 5 Platinum-Gold, %	Ink No. 9 Gold, %	Ink No. 10 Gold, %
Gold	42.0	37.0	0.0015	0.0087	0.011	43.0	41.0	38.0	77.0	66.0
Silver	0.0058	0.0065	34.0	40.0	38.0	0.013	0.0074	0.010	0.0051	0.013
Palladium	25.0	24.0	14.0	15.0	6.4	0.081	0.032	6.2	0.0096	0.011
Platinum	–	0.028	–	–	–	26.0	24.0	19.0	–	–
Silicon	0.13	3.5	2.4	0.22	2.7	0.20	3.6	4.0	3.7	2.8
Bismuth	6.1	6.4	13.0	12.0	12.0	6.5	0.018	4.7	3.8	5.0
Lead	0.059	2.7	0.011	0.14	4.4	Trace	0.075	3.3	1.7	2.4
Cadmium	3.3	–	2.5	0.0093	0.0059	1.2	0.015	0.0089	–	0.93
Zinc	–	–	Nil	1.0	–	Nil	2.4		–	–
Aluminum	0.058	0.36	0.80	0.39	1.0	0.081	0.39	0.49	0.083	0.43
Titanium	0.0046	0.031	0.011	Trace	0.045	Trace	0.011	0.046	0.0035	0.020
Ruthenium	0.018	–	0.013	Nil	–	–	–		–	–
Calcium	0.0067	0.50	–	0.12	0.27	0.041	0.24	0.74	0.0055	0.52
Copper	0.00083	0.0012	0.0085	0.0076	0.0046	0.0019	0.0015	0.00089	0.0024	0.0070
Iron	0.017	0.029	0.018	0.014	0.012	0.39	0.40	0.041	0.0087	0.0058
Magnesium	0.0010	0.010	0.0064	0.0043	0.0055	0.0048	0.0064	0.0089	0.0019	0.0093
Boron	0.36	0.13	0.16	0.46	0.21	0.20	0.57	0.13	Trace	0.13
Manganese	–	Trace	–	–	0.023	–	–	0.0096	–	Trace
Nickel	–	Trace	0.00048	0.0012	0.026	–	–	Trace	–	–
Chromium	–	0.0038	0.0019	0.0020	0.0014	Trace	Trace	Trace	–	–
Zirconium	–	–	Trace	0.071	–	Nil	0.27	–	–	–
Yttrium	–	–	0.030	Nil	–	–	–	–	–	–
Strontium	–	–	0.0035	0.0027	–	Nil	Trace	–	–	–
Barium	–	–	–	–	–	–	Nil	0.27	–	–
Loss on Ignition	20.2	19.3	28.0	27.4	29.4	20.6	19.5	16.5	7.80	16.8

Table 4. Printing of inks

Ink No.	Printing characteristics	Printing results	Dry-film thickness, μ	Fired thickness, μ	Conductivity of a 508 μ (0.0200 in.) line, Ω
1	The ink printed wet and heavy but was uniform from start to finish. The viscosity of the ink held up well, and screen cleaning and ink distribution were less than normal.	Printing results were good. Lines did not flow together, even with the 102 μ (0.004 in.) spacing. The 51 μ (0.002 in.) lines printed irregularly and would not be satisfactory.	18.5 and 21.3	14.3 and 15.3	7.7 [546 μ (0.0215 in.)][a]
2	The ink was a thin, free-flowing material requiring less screen cleaning or redistribution than any of the other inks tested.	Very little difference was noted between this ink and the No. 1 ink. The same comments would apply.	18.5 and 19.9	14.0 and 14.8	4.9 [591 μ (0.0233 in.)][a]
3	The ink worked very well and printed smoothly with very little screen cleaning or ink redistribution.	Printing results were fair. Moderate spreading occurred and the lines ran together with the 102 μ (0.004 in.) spacing. Printing of the 51 μ (0.002 in.) line was very irregular.	18.1 and 18.2	12.3 and 12.5	6.8 [534 μ (0.0210 in.)][a]
4	The ink was free-flowing and an excellent material with which to work. Very little screen cleaning or redistribution was necessary.	The printing results were very similar to Ink No. 3, although the material flowed slightly less. Printing of the 51 μ (0.002 in.) line was very irregular.	20.9 and 21.8	13.3 and 13.5	6.1 [515 μ (0.203 in.)][a]
5	The ink was a smooth flowing, excellent working material. A minimum of redistribution and screen cleaning was necessary.	The ink printed well and spread very little. Results were very similar to Ink Nos. 1 and 2.	20.2 and 21.5	12.3 and 15.4	5.6 [551 μ (0.0217 in.)][a]
6	Printing was very difficult with this material. Frequent screen cleaning and redistribution of ink were necessary. Silver metal appeared to float on top of the ink.	The ink flowed causing the lines to run together with the 102 μ (0.004 in.) spacing. Very little printing of the 51 μ (0.002 in.) line occurred.	15.5 and 15.7	7.7 and 11.1	8.3 [503 μ (0.0198 in.)][a]
7	The material printed satisfactorily with clean, uniform markings being obtained. Moderate cleaning and redistribution were necessary.	Printing results were sharp and clear with very little spreading of the ink. Printing of the 51 μ (0.002 in.) line was still irregular, although better than most inks tested.	19.2 and 19.7	9.1 and 10.1	2.0 [508 μ (0.0200 in.)][a]
8	The material was a fairly thin, free-flowing ink that was easy to work with. Moderate redistribution and screen cleaning were required.	Printing was sharp and clear and quite similar to Ink No. 7. Printing of the 51 μ (0.002 in.) line was the best of all the inks tested.	17.6 and 17.7	7.9 and 8.6	2.1 [465 μ (0.0183 in.)][a]
9	The ink appeared to be a rather dry paste. Screen cleaning and redistribution of ink were required after almost every substrate.	Printing was sharp and clear with very little spreading. Printing of the 51 μ (0.002 in.) line was irregular.	23.3 and 33.2	14.7 and 15.0	0.2 [534 μ (0.0210 in.)][a]
10	The ink handled well, but was of an extremely thin consistency. Redistribution of ink and screen cleaning were necessary only after printing a great many substrates.	Results were similar to Ink No. 9, and very little difference between the two inks could be detected.	15.8 and 16.3	10.1 and 10.7	0.3 [515 μ (0.0203 in.)][a]

[a]Indicates actual measured width (average) of four lines.

Table 5. Substrate requirements, no simulated resistor firing

Ink No.	Firing schedule, min. at °C	Simulated resistor firing, min. at °C	Solder test, preliminary[a]	Solder test, Sn 63[a,b]	Solder test, 4% silver[a,b]	Wire bonding
1	5 at 850	None	0	5	5	2
	10 at 850		18	5	5	2
	20 at 850		0	5	5	2
2	5 at 950		0	5	5	2
	10 at 950		18	5	5	2
	20 at 950		0	5	5	2
3	5 at 950		0	5	5	2
	10 at 950		18	5	5	2
	20 at 950		0	5	5	2
4	5 at 900		0	5	5	2
	10 at 900		18	5	5	2
	20 at 900		0	5	5	2
5	5 at 900		0	5	5	2
	10 at 900		18	5	5	2
	20 at 900		0	5	5	2
6	5 at 850		0	5	5	2
	10 at 850		18	5	5	2
	20 at 850		0	5	5	2
7	5 at 850		0	5	5	2
	10 at 850		18	5	5	2
	20 at 850		0	5	5	2
8	5 at 850		0	5	5	2
	10 at 850		18	5	5	2
	20 at 850		0	5	5	2
9	5 at 900		0	0	0	2
	10 at 850		0	0	0	2
	20 at 850		0	0	0	2
10	5 at 900		0	0	0	2
	10 at 900		0	0	0	2
	20 at 900	None	0	0	0	2

[a]Numbers indicate substrates used for each test.

[b]Three substrates were used for solderability and leaching tests and two for solder adhesion tests.

Table 6. Substrate requirements, simulated resistor firing 10 min. at 775 °C

Ink No.	Firing schedule, min. at °C	Simulated resistor firing, min. at °C	Solder test, preliminary[a]	Solder test, Sn 63[a,b]	Solder test, 4% silver[a,b]	Wire bonding
1	5 at 850	10 at 775	0	5	5	2
	10 at 850	(Code B)	18	5	5	2
	20 at 850		0	5	5	2
2	5 at 950		0	5	5	2
	10 at 950		18	5	5	2
	20 at 950		0	5	5	2
3	5 at 950		0	5	5	2
	10 at 950		18	5	5	2
	20 at 950		0	5	5	2
4	5 at 900		0	5	5	2
	10 at 900		18	5	5	2
	20 at 900		0	5	5	2
5	5 at 900		0	5	5	2
	10 at 900		18	5	5	2
	20 at 900		0	5	5	2
6	5 at 850		0	5	5	2
	10 at 850		18	5	5	2
	20 at 850		0	5	5	2
7	5 at 850		0	5	5	2
	10 at 850		18	5	5	2
	20 at 850		0	5	5	2
8	5 at 850		0	5	5	2
	10 at 850		18	5	5	2
	20 at 850		0	5	5	2
9	5 at 900		0	0	0	2
	10 at 900		0	0	0	2
	20 at 900		0	0	0	2
10	5 at 900		0	0	0	2
	10 at 900	10 at 775	0	0	0	2
	20 at 900	(Code B)	0	0	0	2

[a]Numbers indicate substrates used for each test.

[b]Three substrates were used for solderability and leaching tests and two for solder adhesion tests.

Table 7. Substrate requirements, simulated resistor firing 10 min. at 900°C

Ink No.	Firing schedule, min. at °C	Simulated resistor firing, min. at °C	Solder test, preliminary[a]	Solder test, Sn 63[a,b]	Solder test, 4% silver[a,b]	Wire bonding
1	5 at 850	10 at 900	0	5	5	2
	10 at 850		18	5	5	2
	20 at 850		0	5	5	2
2	5 at 950		0	5	5	2
	10 at 950		18	5	5	2
	20 at 950		0	5	5	2
3	5 at 950		0	5	5	2
	10 at 950		18	5	5	2
	20 at 950		0	5	5	2
4	5 at 900		0	5	5	2
	10 at 900		18	5	5	2
	20 at 900		0	5	5	2
5	5 at 900		0	5	5	2
	10 at 900		18	5	5	2
	20 at 900		0	5	5	2
6	5 at 850		0	5	5	2
	10 at 850		18	5	5	2
	20 at 850		0	5	5	2
7	5 at 850		0	5	5	2
	10 at 850		18	5	5	2
	20 at 850		0	5	5	2
8	5 at 850		0	5	5	2
	10 at 850		18	5	5	2
	20 at 850		0	5	5	2
9	5 at 900		0	0	0	2
	10 at 900		0	0	0	2
	20 at 900		0	0	0	2
10	5 at 900		0	0	0	2
	10 at 900		0	0	0	2
	20 at 900	10 at 900	0	0	0	2

[a]Numbers indicate substrates used for each test.

[b]Three substrates were used for solderability and leaching tests and two for solder adhesion tests.

Table 8. Solder/ink adhesion

Substrate designation	Sn 63 solder		4% silver solder	
	90° pull,[a] N(oz)	45° pull,[a] N(oz)	90° pull,[a] N(oz)	45° pull,[a] N(oz)
1-5-A	2.91 (10.5)	9.14 (32.9)	3.47 (12.5)	3.50 (12.6)
1-5-B	2.30 (8.3)	4.11 (14.8)	4.00 (14.4)	4.08 (14.7)
1-5-C	1.91 (6.9)	7.11 (25.6)	3.22 (11.6)	3.36 (12.1)
1-10-A	1.86 (6.7)	8.00 (28.8)	5.14 (18.5)	5.83 (21.0)
1-10-B	2.14 (7.7)	7.14 (25.7)	3.91 (14.1)	4.55 (16.4)
1-10-C	1.69 (6.1)	6.69 (24.1)	4.55 (16.4)	4.69 (16.9)
1-20-A	2.05 (7.4)	9.64 (34.7)	4.28 (15.4)	7.22 (26.0)
1-20-B	2.41 (8.7)	9.47 (34.1)	3.89 (14.0)	7.00 (25.2)
1-20-C	2.41 (8.7)	6.72 (24.2)	2.89 (10.4)	5.42 (19.5)
2-5-A	6.17 (22.2)	9.89 (35.6)	5.69 (20.5)	8.00 (28.8)
2-5-B	3.83 (13.8)	15.56 (56.0)	4.42 (15.9)	10.17 (36.6)
2-5-C	4.17 (15.0)	10.09 (36.3)	5.94 (21.4)	8.56 (30.8)
2-10-A	5.03 (18.1)	10.92 (39.3)	5.03 (18.1)	7.83 (28.2)
2-10-B	2.39 (8.6)	9.53 (34.3)	3.78 (13.6)	6.67 (24.0)
2-10-C	2.22 (8.0)	10.00 (36.0)	4.50 (16.2)	7.17 (25.8)
2-20-A	4.75 (17.1)	11.37 (40.9)	5.05 (18.2)	7.17 (25.8)
2-20-B	2.91 (10.5)	13.09 (47.1)	4.00 (14.4)	7.67 (27.6)
2-20-C	2.78 (10.0)	9.11 (32.8)	4.42 (15.9)	7.75 (27.9)
3-5-A	2.16 (7.8)	1.55 (5.6)	2.22 (8.0)	3.05 (11.0)
3-5-B	2.05 (7.4)	2.39 (8.6)	2.02 (7.3)	2.22 (8.0)
3-5-C	1.97 (7.1)	1.69 (6.1)	2.66 (9.6)	2.55 (9.2)
3-10-A	2.08 (7.5)	1.91 (6.9)	1.75 (6.3)	2.30 (8.3)
3-10-B	1.58 (5.7)	2.25 (8.1)	1.55 (5.6)	1.77 (6.4)
3-10-C	1.50 (5.4)	1.47 (5.3)	1.61 (5.8)	1.77 (6.4)
3-20-A	1.55 (5.6)	1.33 (4.8)	2.41 (8.7)	2.39 (8.6)
3-20-B	1.44 (5.2)	1.36 (4.9)	2.65 (9.5)	2.39 (8.6)
3-20-C	1.39 (5.0)	1.33 (4.8)	2.11 (7.6)	2.08 (7.5)
4-5-A	7.61 (27.4)	13.01 (46.8)	8.20 (29.5)	15.34 (55.2)
4-5-B	6.33 (22.8)	12.17 (43.8)	8.47 (30.5)	17.23 (62.0)
4-5-C	4.64 (16.7)	13.28 (47.8)	6.33 (22.8)	14.53 (52.3)
4-10-A	8.89 (32.0)	12.95 (46.6)	7.22 (26.0)	12.20 (43.9)
4-10-B	6.06 (21.8)	11.53 (41.5)	6.47 (23.3)	20.85 (75.0)
4-10-C	4.08 (14.7)	11.20 (40.3)	7.58 (27.3)	16.17 (58.2)
4-20-A	8.22 (29.6)	10.89 (39.2)	8.22 (29.6)	18.95 (68.2)
4-20-B	5.36 (19.3)	14.45 (52.0)	7.70 (27.7)	16.23 (58.4)
4-20-C	7.22 (26.0)	10.34 (37.2)	8.67 (31.2)	15.84 (57.0)
5-5-A	6.67 (24.0)	11.50 (41.4)	6.78 (24.4)	15.29 (55.0)
5-5-B	7.14 (25.7)	10.73 (38.6)	5.47 (19.7)	15.90 (57.2)
5-5-C	4.14 (14.9)	8.11 (29.2)	5.56 (20.0)	15.17 (54.6)
5-10-A	4.86 (17.5)	8.22 (29.6)	5.08 (18.3)	13.14 (47.3)
5-10-B	5.05 (18.2)	6.67 (24.0)	4.19 (15.1)	12.45 (44.8)
5-10-C	5.78 (20.8)	5.86 (21.1)	5.25 (18.9)	13.48 (48.5)
5-20-A	2.91 (10.5)	5.47 (19.7)	4.61 (16.6)	7.20 (25.9)
5-20-B	4.28 (15.4)	5.03 (18.1)	4.33 (15.6)	3.00 (18.0)
5-20-C	3.72 (13.4)	4.89 (17.6)	5.42 (19.5)	4.72 (17.0)
6-5-A	4.42 (15.9)	2.83 (10.2)	3.53 (12.7)	8.70 (31.3)
6-5-B	3.08 (11.1)	2.41 (8.7)	3.00 (10.8)	12.56 (45.2)
6-5-C	5.03 (18.1)	2.16 (7.8)	1.44 (5.2)	3.83 (13.8)
6-10-A	2.33 (8.4)	2.14 (7.7)	1.91 (6.9)	2.41 (8.7)
6-10-B	1.86 (6.7)	1.58 (5.7)	3.28 (11.8)	4.36 (15.7)
6-10-C	3.36 (12.1)	1.61 (5.8)	3.55 (12.8)	3.75 (13.5)
6-20-A	5.05 (18.2)	6.75 (24.3)	4.50 (16.2)	6.50 (23.4)
6-20-B	4.00 (14.4)	3.08 (11.1)	4.44 (16.0)	11.62 (41.8)
6-20-C	2.97 (10.7)	4.28 (15.4)	4.44 (16.0)	11.14 (40.1)
7-5-A	5.33 (19.2)	5.30 (19.1)	4.67 (16.8)	11.28 (40.6)
7-5-B	3.64 (13.1)	7.58 (27.3)	6.25 (22.5)	10.00 (36.0)
7-5-C	1.86 (6.7)	2.33 (8.4)	4.17 (15.0)	10.00 (36.0)
7-10-A	3.55 (12.8)	4.58 (16.5)	7.70 (27.7)	12.87 (46.3)
7-10-B	3.08 (11.1)	4.61 (16.6)	7.06 (25.4)	15.70 (56.5)
7-10-C	1.86 (6.7)	3.50 (12.6)	8.25 (29.7)	9.67 (34.8)
7-20-A	2.47 (8.9)	3.33 (12.0)	3.97 (14.3)	10.28 (37.0)
7-20-B	2.86 (10.3)	4.28 (15.4)	5.53 (19.9)	8.89 (32.0)
7-20-C	2.08 (7.5)	3.16 (11.4)	2.44 (8.8)	5.36 (19.3)
8-5-A	5.39 (19.4)	13.03 (46.9)	3.72 (13.4)	14.26 (51.3)
8-5-B	7.17 (25.8)	9.42 (33.9)	4.86 (17.5)	7.70 (27.7)
8-5-C	13.50 (12.6)	8.00 (28.8)	4.83 (17.4)	5.50 (19.8)
8-10-A	4.50 (16.2)	7.42 (26.7)	6.69 (24.1)	16.29 (58.6)
8-10-B	3.86 (13.9)	6.89 (24.8)	6.31 (22.7)	17.09 (61.5)
8-10-C	3.69 (13.3)	6.67 (24.0)	6.33 (22.8)	15.51 (55.8)
8-20-A	5.44 (19.6)	12.03 (43.3)	5.72 (20.6)	12.78 (46.0)
8-20-B	4.83 (17.4)	10.53 (37.9)	7.25 (26.1)	12.67 (45.6)
8-20-C	4.72 (17.0)	9.84 (35.4)	6.33 (22.8)	11.28 (40.6)

[a]Pull strength is the average of 5 tests made on one substrate.

Table 9. Bonding comparisons of conductor inks

Ink No.	Substrate designation	Firing schedule, min. at °C	Ball bond strength, g		Wedge bond strength, g		Wedge failure[a]
			Average	± Deviation	Average	± Deviation	
1	1-5-A	5 at 850	7.1	+0.6 -0.7	6.2	+0.8 -0.8	A=7, C=3
	1-10-A	10 at 850	7.2	+0.6 1.0	4.8	+0.9 -0.5	A=3, C=7
	1-20-A	20 at 850	7.3	+0.5 -0.5	5.8	+0.9 -1.4	A=5, C=5
2	2-5-A	5 at 950	7.3	+0.7 -0.7	6.3	+1.0 -0.7	A=9, C=1
	2-10-A	10 at 950	7.0	+0.7 -0.5	6.3	+0.9 -2.3	A=2, C=8
	2-20-A	20 at 950	7.1	+0.6 -0.7	6.2	+0.9 -1.4	A=5, C=5
3	3-5-A	5 at 950	7.3	+0.7 -1.0	5.6	+0.9 -1.1	A=6, C=4
	3-10-A	10 at 950	7.2	+0.4 -0.8	4.8	+1.2 -1.1	A=4, C=6
	3-20-A	20 at 950	7.5	+0.4 -0.5	4.8	+0.4 -0.5	A=3, C=7
4	4-5-A	5 at 900	6.9	+0.7 -0.5	5.7	+1.0 -3.0	A=7, C=3
	4-10-A	10 at 900	7.5	+0.3 -0.3	6.1	+1.4 -0.8	A=10
	4-20-A	20 at 900	7.1	+0.4 -0.7	6.7	+0.5 -1.1	A=8, B=2
5	5-5-A	5 at 900	7.4	+0.5 -0.6	6.2	+0.9 -0.8	A=6, C=4
	5-10-A	10 at 900	7.3	+0.4 -0.5	5.0	+1.1 -1.6	A=0, C=10
	5-20-A	20 at 900	7.1	+0.4 -0.5	5.7	+1.5 -1.1	A=10
6	6-5-A	5 at 850	7.3	+0.3 -0.8	7.5	+0.4 -1.2	A=2, B=5, C=3
	6-10-A	10 at 850	7.2	+0.5 -0.6	6.6	+1.2 -5.1	A=4, B=4, C=2
	6-20-A	20 at 850	7.0	+0.6 -1.1	5.4	+2.4 -4.4	A=1, B=1, C=8
7	7-5-A	5 at 850	7.3	+0.3 -0.2	7.2	+0.7 -0.5	A=2, B=8
	7-10-A	10 at 850	7.6	+0.3 -0.3	6.1	+2.0 -0.9	A=3, B=1, C=6
	7-20-A	20 at 850	7.2	+0.4 -0.3	6.8	+0.8 -1.4	A=7, B=1, C=2
8	8-5-A	5 at 850	6.7	+0.9 -1.1	6.8	+0.8 -0.7	A=6, B=2, C=2
	8-10-A	10 at 850	7.6	+0.5 -0.3	4.9	+1.7 -2.9	A=4, C=6
	8-20-A	20 at 850	6.9	+0.6 -0.4	6.3	+0.9 -2.1	A=6, C=4
9	9-5-A	5 at 900	7.2	+0.4 -0.9	7.3	+0.7 -1.0	A=7, B=3
	9-10-A	10 at 900	7.5	+0.2 -0.7	6.7	+0.5 -0.7	A=10
	9-20-A	20 at 900	7.1	+0.7 -0.8	7.1	+0.8 -1.0	A=3, B=7
10	10-5-A	5 at 900	6.9	+0.6 -0.8	7.1	+0.8 -0.8	A=5, B=3, C=2
	10-10-A	10 at 900	7.4	+0.5 -0.6	6.5	+0.9 -2.3	A=4, B=1, C=5
	10-20-A	20 at 900	7.1	+0.5 -0.5	6.1	+1.4 -2.7	A=5, B=2, C=3

[a]Wedge failure data based on 10 samples: A = broke at bond
B = broke at other point
C = pulled off

Table 9 (contd)

Ink No.	Substrate designation	Firing schedule, min. at °C	Ball bond strength, g		Wedge bond strength, g		Wedge failure[a]
			Average	± Deviation	Average	± Deviation	
Refired 10 min. at 775°C							
1	1-5-B	5 at 850	7.1	+0.6 -0.8	6.1	+0.6 -0.8	A=5, C=5
	1-10-B	10 at 850	7.5	+0.4 -0.8	6.0	+0.7 -0.7	A=6, C=4
	1-20-B	20 at 850	7.2	+0.6 -0.5	5.8	+1.6 -1.0	A=7, B=1, C=2
2	2-5-B	5 at 950	7.1	+0.7 -0.5	6.4	+1.0 -1.1	A=8, B=1, C=1
	2-10-B	10 at 950	7.0	+0.7 -0.4	5.6	+1.2 -0.9	A=4, C=6
	2-20-B	20 at 950	7.2	+0.6 -1.3	6.3	+0.9 -1.0	A=10
3	3-5-B	5 at 950	7.0	+0.6 -0.8	5.7	+1.6 -1.5	A=8, C=2
	3-10-B	10 at 950	7.4	+0.6 -0.6	4.5	+2.2 -2.5	A=6, C=4
	3-20-B	20 at 950	7.3	+0.5 -0.8	5.5	+1.5 -0.7	A=7, C=3
4	4-5-B	5 at 900	7.1	+0.7 -1.0	6.3	+0.7 -1.1	A=10
	4-10-B	10 at 980	7.0	+0.7 -0.9	6.3	+0.8 -1.1	A=9, C=1
	4-20-B	20 at 900	7.1	+0.6 -1.3	6.2	+1.3 -1.4	A=10
5	5-5-B	5 at 900	7.2	+0.7 -0.6	5.6	+1.4 -1.5	A=10
	5-10-B	10 at 900	7.3	+0.4 -0.7	6.9	+1.2 -1.3	A=9, B=1
	5-20-B	20 at 900	7.3	+0.5 -1.0	6.2	+1.0 -1.6	A=9, C=1
6	6-5-B	5 at 850	7.0	+0.8 -0.6	4.4	+3.2 -3.4	A=3, C=7
	6-10-B	10 at 850	7.4	+0.8 -0.8	7.5	+0.7 -1.4	A=1, B=9
	6-20-B	20 at 850	7.0	+0.6 -0.8	6.9	+1.0 -0.6	A=3, B=6, C=1
7	7-5-B	5 at 850	7.2	+0.7 -0.6	6.3	+1.3 -2.1	A=4, B=2, C=4
	7-10-B	10 at 850	7.4	+0.7 -0.6	7.5	+0.5 -1.0	A=6, B=4
	7-20-B	20 at 850	7.1	+0.5 -0.4	6.1	+0.7 -1.1	A=4, B=1, C=5
8	8-5-B	5 at 850	7.0	+0.6 -0.5	6.8	+0.3 -1.0	A=6, B=3, C=1
	8-10-B	10 at 850	7.5	+0.2 -1.0	7.2	+0.6 -0.5	A=7, B=3
	8-20-B	20 at 850	6.6	+0.6 -0.3	6.9	+0.5 -0.6	A=1, B=9
9	9-5-B	5 at 900	7.0	+0.3 -0.3	6.8	+0.7 -0.6	A=10
	9-10-B	10 at 900	7.1	+0.6 -0.6	7.3	+0.7 -1.5	A=5, B=4, C=1
	9-20-B	20 at 900	6.7	+0.8 -0.3	6.9	+0.5 -0.5	A=3, B=7
10	10-5-B	5 at 900	7.1	+0.5 -0.8	6.5	+1.3 -5.5	A=7, B=1, C=2
	10-10-B	10 at 900	7.0	+0.7 -0.8	6.5	+1.1 -2.8	A=3, B=4, C=3
	10-20-B	20 at 900	6.7	+0.5 -0.6	6.7	+0.7 -1.5	A=3, B=5, C=2

Table 9 (contd)

Ink No.	Substrate designation	Firing schedule, min. at °C	Ball bond strength, g			Wedge bond strength, g			Wedge failure[a]
			Average	± Deviation		Average	± Deviation		
Refired 10 min. at 900°C									
1	1-5-C	5 at 850	7.2	+0.5	-0.5	6.0	+1.2	-1.3	A=8, C=2
	1-10-C	10 at 850	7.0	+0.5	-0.6	6.1	+1.1	-0.9	A=6, B=1, C=3
	1-20-C	20 at 850	5.7	+1.0	-0.7	6.0	+0.6	-0.7	A=7, B=3
2	2-5-C	5 at 950	6.7	+0.6	-0.9	6.9	+0.9	-1.6	A=9, C=1
	2-10-C	10 at 950	7.5	+0.8	-0.6	6.7	+0.8	-1.1	A=5, B=2, C=3
	2-20-C	20 at 950	5.8	+1.2	-0.6	6.0	+0.7	-0.4	A=4, B=6
3	3-5-C	5 at 950	7.2	+0.4	-0.8	5.3	+1.0	-0.7	A=7, C=3
	3-10-C	10 at 950	7.3	+0.9	-0.8	5.2	+0.6	-2.0	A=7, C=3
	3-20-C	20 at 950	5.4	+0.9	-0.5	5.1	+0.8	-0.7	A=4, C=6
4	4-5-C	5 at 900	7.1	+0.5	-0.5	6.7	+0.7	-0.7	A=9, C=1
	4-10-C	10 at 900	6.9	+0.7	-0.8	6.7	+0.6	-0.7	A=8, B=2
	4-20-C	20 at 900	5.5	+0.7	-0.4	5.3	+0.6	-0.5	A=7, B=2, C=1
5	5-5-C	5 at 900	7.1	+0.6	-0.7	6.1	+1.6	-1.3	A=9, B=1
	5-10-C	10 at 900	7.0	+0.6	-0.6	6.6	+0.6	-0.9	A=9, C=1
	5-20-C	20 at 900	5.4	+0.5	-0.5	5.0	+0.6	-1.0	A=7, B=3
6	6-5-C	5 at 850	7.2	+0.6	-0.8	5.8	+2.3	-4.3	A=2, B=3, C=5
	6-10-C	10 at 850	6.9	+0.8	-0.6	6.1	+1.6	-4.6	A=3, B=3, C=3
	6-20-C	20 at 850	5.4	+0.6	-0.5	5.8	+0.4	-1.4	A=1, B=6, C=3
7	7-5-C	5 at 850	7.4	+0.4	-0.6	6.8	+1.2	-2.2	A=2, B=4, C=4
	7-10-C	10 at 850	7.2	+0.5	-1.3	7.3	+0.5	-0.7	A=7, B=3
	7-20-C	20 at 850	5.3	+1.2	-0.5	5.5	+0.5	-1.0	A=3, B=6, C=1
8	8-5-C	5 at 850	7.2	+0.3	-0.7	7.0	+0.7	-1.4	A=6, B=3, C=1
	8-10-C	10 at 850	7.2	+0.6	-1.4	7.4	+0.5	-0.4	A=6, B=4
	8-20-C	20 at 850	5.8	+0.4	-0.8	5.1	+1.3	-1.5	B=2, C=8
9	9-5-C	5 at 900	7.4	+0.4	-0.9	7.1	+0.6	-0.6	A=9, B=1
	9-10-C	10 at 900	7.3	+0.4	-0.7	7.0	+1.1	-1.2	A=9, B=1
	9-20-C	20 at 900	5.5	+0.6	-0.5	5.5	+0.7	-1.0	A=5, B=5
10	10-5-C	5 at 900	7.0	+0.5	-0.9	7.1	+0.6	-0.7	A=7, B=3
	10-10-C	10 at 900	7.1	+0.6	-0.8	6.9	+0.4	-0.3	A=10
	10-20-C	20 at 900	5.8	+0.8	-0.8	5.8	+1.1	-0.5	A=6, B=4

Table 10. Ink comparison chart[a]

Ink No.	Ink type	Printing characteristics	Solderability		Leach resistance		Solder adhesion		Wire bonding	Effect of firing schedule
			Sn 63	4% Silver	Sn 63	4% Silver	Sn 63	4% Silver		
1	Palladium-Gold	3	2	3	2	2	4	3	3	2
2	Palladium-Gold	3	3	4	3	1	3	3	3	1
3	Platinum-Gold	4	3	3	3	1	4	5	3	2
4	Platinum-Gold	3	3	4	3	4	2	1	1	1
5	Platinum-Gold	3	2	3	2	1	2	3	3	1
6	Palladium-Silver	5	4	4	5	4	3	4	2	2
7	Palladium-Silver	2	2	2	2	1	3	2	2	1
8	Palladium-Silver	2	2	2	3	1	2	2	3	1
9	Gold	4	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested	1	1
10	Gold	3	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested	2	1

[a]Inks were rated from 1 to 5 with 1 being considered excellent and 5 being poor.

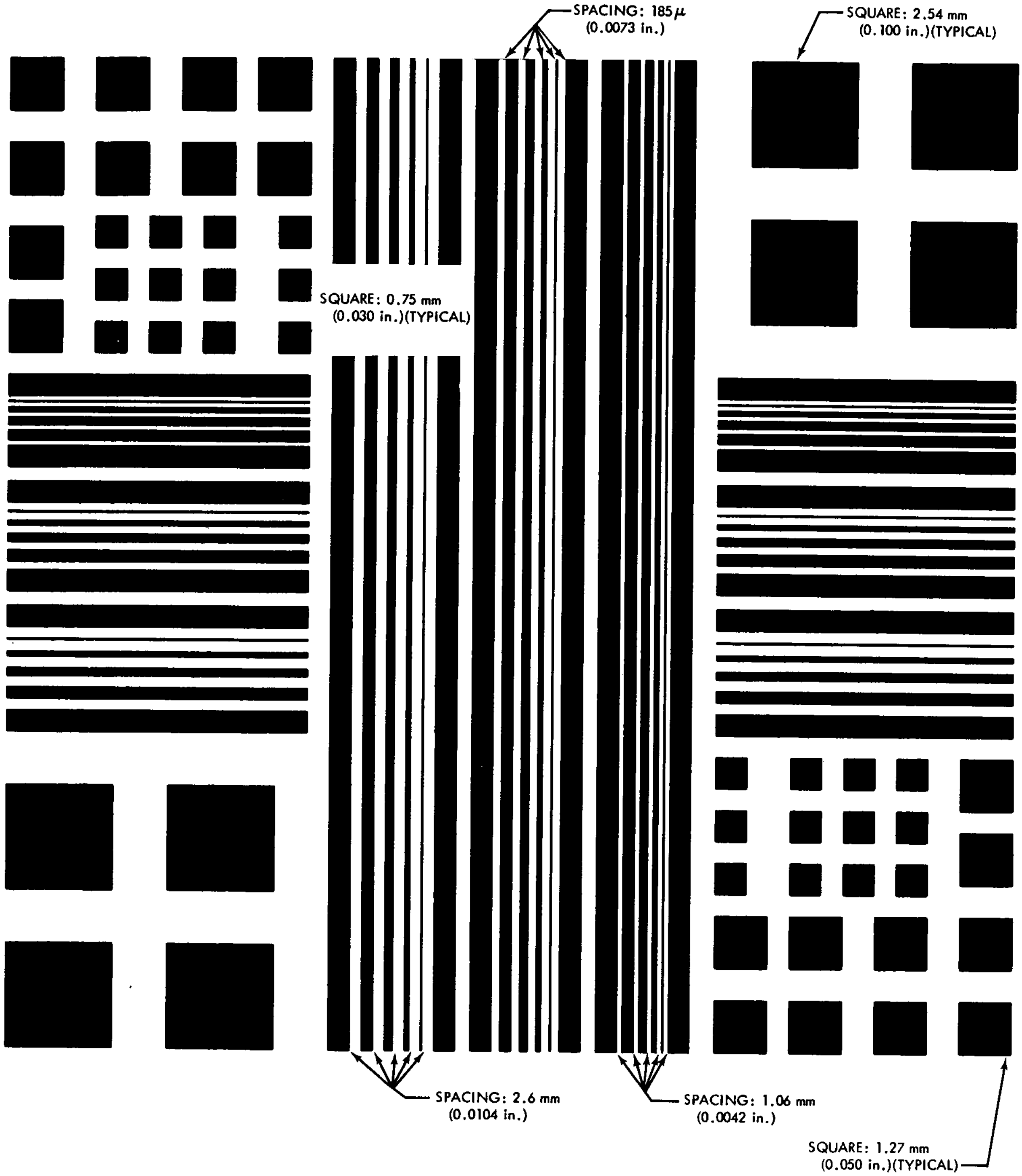

Fig. 1. Conductive ink pattern

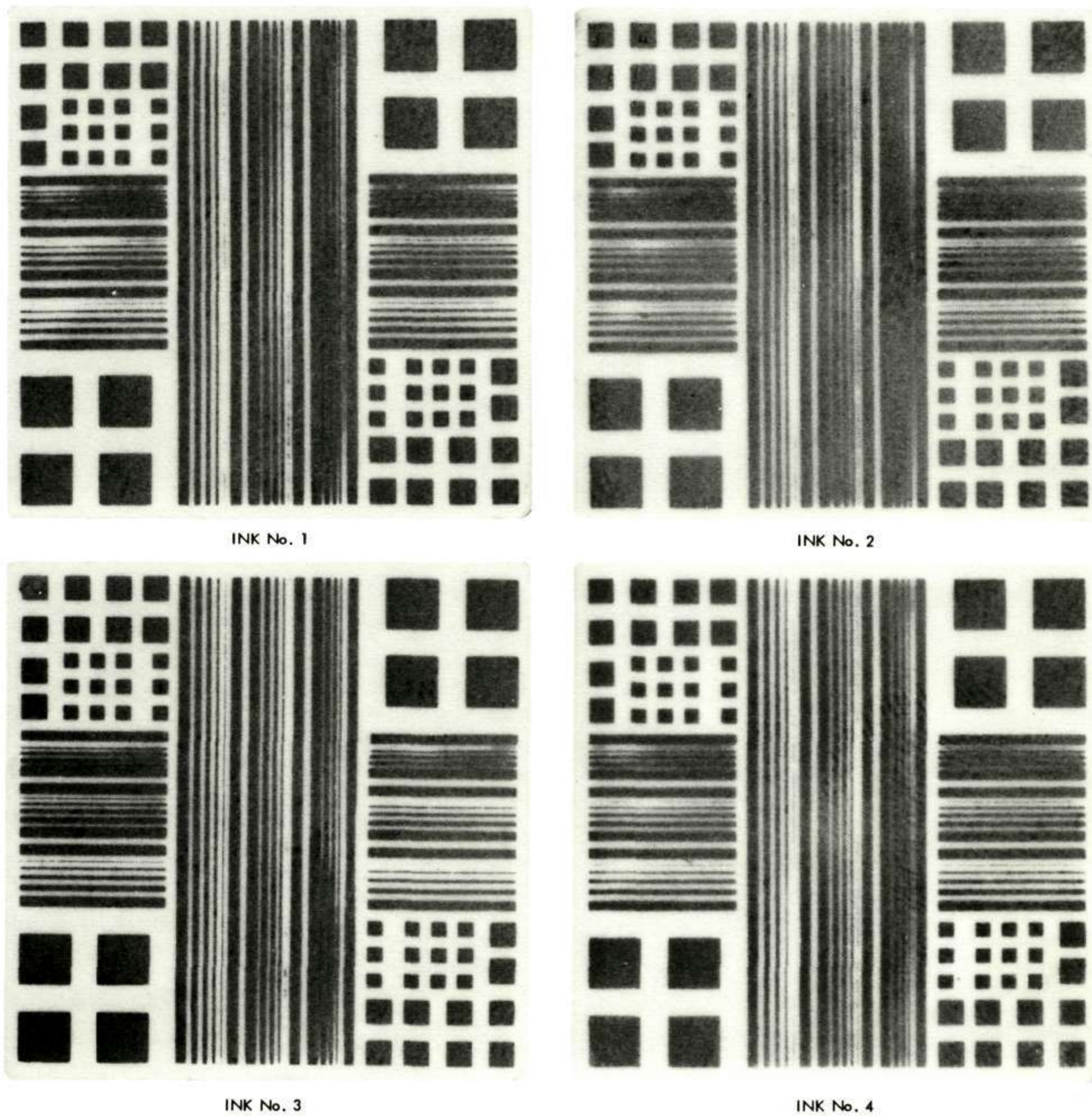

Fig. 2. Printed substrates: Ink Nos. 1, 2, 3 and 4

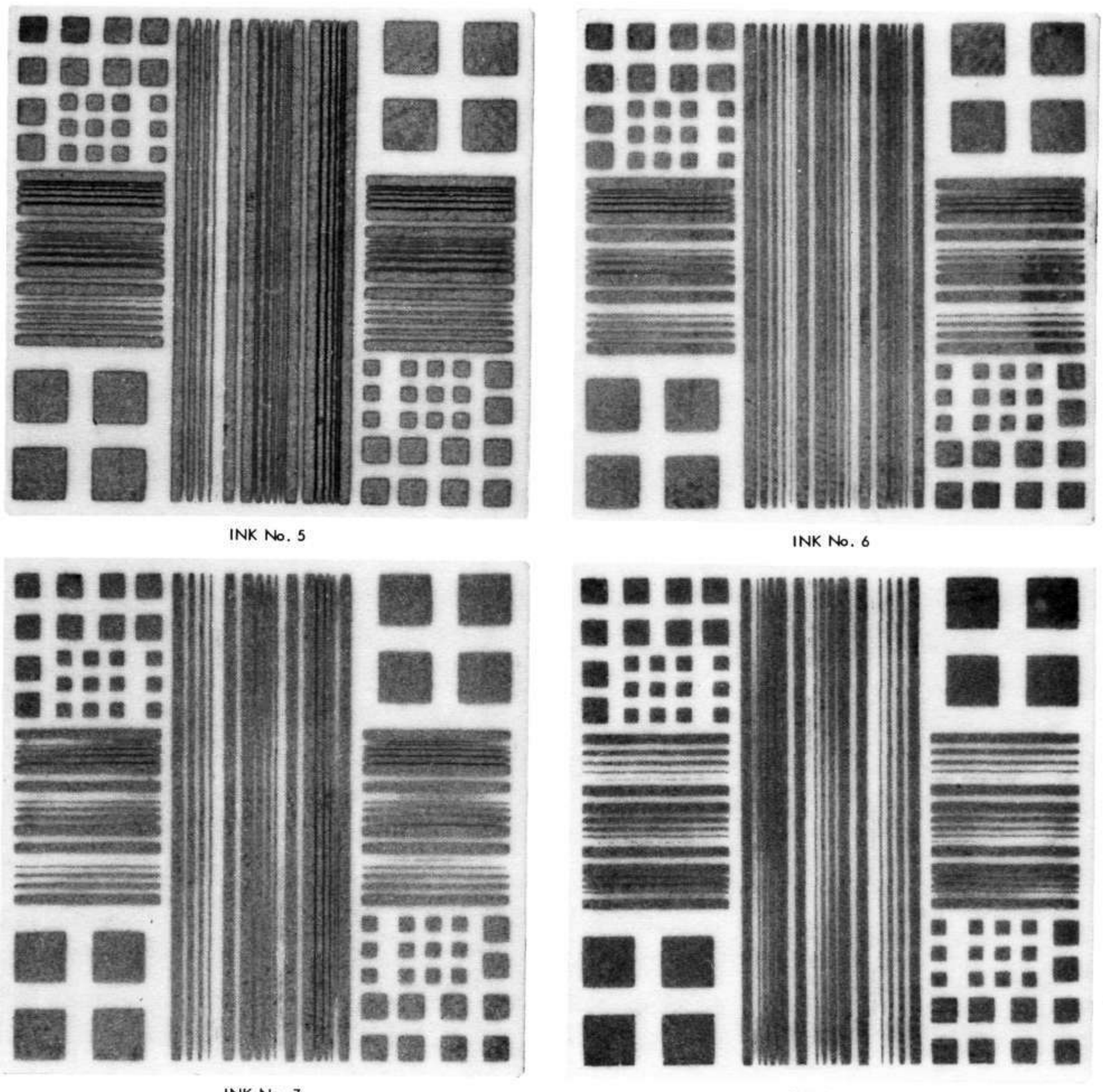

Fig. 3. Printed substrates: Ink Nos. 5, 6, 7 and 8

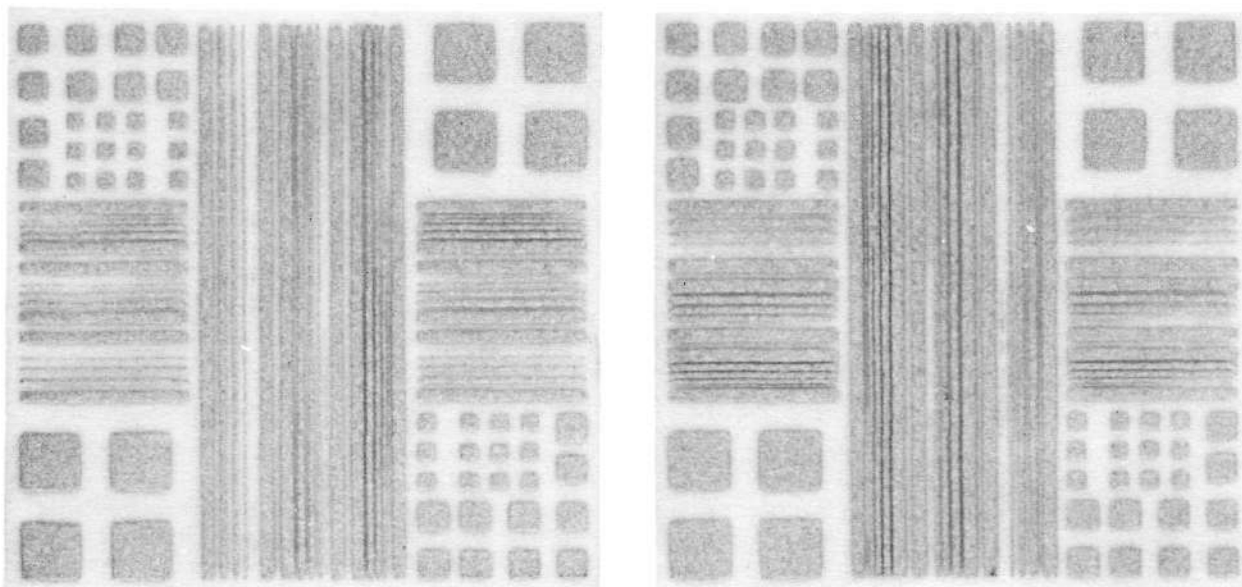

Fig. 4. Printed substrates: Ink Nos. 9 and 10

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