Partnerships Limited, Inc.

MONTHLY STATUS REPORT

Contract No. N00014-94-C-0236

Office of Naval Research

Waste Minimization in Circuit Board Manufacturing

by

Powder Coating and Metallo-Organic Decomposition

CLIN 0001AE

28 February., 1995

Paul H. Kydd, Principal Investigator

Proprietary information of

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Executive Summary

Task 1. Fluidization Studies

Free-flowing powders containing MOD compound and finely divided metals have been successfully demonstrated. This is a major step toward our goal of circuit board production by computer-controlled ink jet or laser printing. An invention disclosure has been filed. The trade name PARMOD has been selected for the materials and process.

Task 2. Coating Studies

Additional tests were performed to determine the influence of various constituents of screen inks and toner powders. Neither the amount of MOD compound nor of silver flake has a dramatic effect on conductivity or adhesion. The MOD compound does have a dramatic effect on the quality of the screened image. Silver-coated nickel powder is inferior to silver flake in all respects, but should be acceptable in small amounts.

Task 3. Decomposition Studies

The toners mentioned in Task 1 were successfully powder coated on screened images simulating ink jet printing. Image quality is not yet equal to the best screened PARMOD test specimens, probably due to electrostatic effects. Conductivity is within a factor of two of the best achieved so far. Adhesion is equal to that of screened images. **These results demonstrate the feasibility of creating printed circuits by printing adhesive images and augmenting their conductivity by powder coating, which is the objective of this Phase I program.**

Task 4. Circuit Board Compatibility Studies

Quantitative measurements show that the peel strength of PARMOD conductors on FEP-coated Kapton substrates is 1/3 to 1/2 the target value of 1229 Newtons per meter. Adhesion to FR-4 epoxy-glass substrates is at the target or above. In some cases the samples failed in the FR-4 laminate itself. **Direct bonding** of simulated component leads by MOD has been demonstrated, which could eliminate the soldering required for final assembly. It would also eliminate the lead-containing solders, which are an environmental and workplace hazard. An Invention Report has been filed on this extension of the PARMOD process

Plans for Next Month

Supplementary data will be obtained primarily to support the commercialization activity, which has started in earnest. The Phase I final report will be prepared.

Experimental Details

This month the emphasis has been on powder coating screened patterns simulating ink jet printed images, which is the main subject of this Phase I Feasibility demonstration, and on quantitative measurement of the adhesion of the conductor traces produced both by screening and powder coating. Some preliminary experiments have ben done on direct bonding of simulated circuit component leads to unfired MOD circuit patterns.

Following last month's extensive work on conductivity, little further effort has been devoted to this aspect of the technology. The conductivity already achieved, while short of our (perhaps overoptimistic) goal of 80% of the conductivity of pure silver is comparable to the 1/2 to 1/4 of bulk conductivity achieved in conventional thick film technology (1). Since we have achieved half the bulk conductivity of pure silver on Kapton and one quarter of the bulk conductivity on FR-4 epoxy-glass boards, and since these conductivities are acceptable commercially, we have decided to devote the limited time remaining in Phase I to expanding the applicability of what we are now calling the PARMOD technology to computer controlled imaging, and to further optimizing and characterizing the materials and circuits produced.

Direct Bonding of Components

Using either ink jet or electrostatic imaging to create circuit patterns and developing adequate conductivity by powder coating and heat treatment can achieve major savings in cost and hazardous waste production in circuit board manufacture. The number of steps can be reduced from twenty or more to two, printing and firing. An additional degree of process integration can be achieved by using the uncured image to bond components to the circuit as well as making the circuit itself. This concept has been demonstrated in a cursory way by bonding 0.25 mm (10 mil) copper wires simulating component leads with dabs of B-30 ink to sample B-30-4, a screen printed MOD pattern on Kapton 300 FN-929, before oven treatment.

Figure 1 shows cross sections of one of the bonded wires and of the trench left when another wire was pulled off the conductor after heat treatment. Despite the fact that no precautions were taken to clean or prepare the wire before bonding, and the oven was operated without any special atmosphere, the cross section shows a solid looking bond, and the trench left when the wire was pulled off shows that the wire removed some of the silver with it. This suggests that circuits could be assembled by screening or otherwise printing an image with PARMOD compositions, placing the components on the wet image and heat treating the assembly to produce the conductors and the connections to the components in one operation. In addition to eliminating an additional set of steps to screen solder paste and remelt it to bond the components to the circuit, the PARMOD

1) Larry, J.R., Rosenberg, R.M., Uhler, R.O., IEEE CHMT-3 (2) 217.

approach eliminates lead-containing solder and yet another environmental and worker safety concern. With silver plated leads, the bond may actually be better than solder. Of course this approach requires components which can take the heat treatment temperature of the PARMOD compound, currently 325 C. This should prove no inherent problem for resistors, ferroelectric capacitors and ceramic packaged IC's, though it will require a special line of components qualified for this application. Direct bonding of chips for tape automated bonding is a possibliity with higher resolution. Figure 2 shows a photo-micrograph of another of the copper wire bonds from above. An invention report has been filed on this concept.

Effect of Ink Composition

A limited set of tests was performed to explore the effect of metal content and MOD content on the performance of screen inks as a preliminary to developing powder toners and the adhesion tests. The results are shown in Tables I and II. Inks B-36 and -37 had less and more silver flake content than the mixture which had evolved as a standard in previous tests. They were evaluated by screening the short circuit and conductivity patterns with a new, finer screen designed to deposit a trace which would convert to the target 10 micron thickness mentioned in the Phase I proposal after oven treatment. We anticipated that the higher metal content ink would produce worse screen images and that ink B-36 would be preferable.

The finer screen did produce images of about the target thickness, 12 microns for B-36 and 14 microns for B-37. The metal traces were smoother and better looking than those from coarser screens, as expected. The conductivity values are essentially indistinguishable at 3.00 microohm cm (s=0.08) for B-36 and 3.08 microohm cm (s=0.13) for B-37. Both inks gave conductivity patterns with breaks in them, and short circuits in the short test pattern. I think this is primarily because the screen image lapped over the hold-down fixture with the new screen, allowing edges of the substrate to move under the screen. In sum the results show that the performance of the ink is very insensitive to the amount of metal.

Ink B-38 was a null mixture with no silver neodecanoate to test whether the MOD compound is critical to adhesion and conductivity. The screen images with this composition are truly terrible, and the conductivity results are not clear cut. They may be less good than those of inks with the MOD compound included. An earlier, cruder null test with a pen-ruled line of α-terpineol powdered with dry silver flake suggested a conductivity of 16 microohm cm, plus or minus a factor of two.

Table II shows a more systematic investigation of the effect of MOD content. Inks B-40 through B-42 have successively less MOD compound and serve as a baseline for subsequent adhesion tests to determine the effect of MOD on adhesion to FEP-coated Kapton. They were heat treated in a belt furnace with a six minute heat up and approximately one minute above 330 C, the kind of heat treatment which would be used in quantity production. The most dramatic effect of removing the MOD compound is that the screened image quality degrades dramatically. However, there may be other organic additives which could compensate for this. The real issue is whether the MOD compound improves conductivity due to bonding the silver flake and whether it improves adhesion. The conductivity results suggest that MOD improves bonding of the silver flake, but not dramatically.

Powder Coating Results

Table III shows the screen inks used to make images for powder coating experiments with various toner compositions. Ink B-34 had been used for the abortive fluid bed coating tests mentioned last month. We established then that it is not possible to powder coat and heat treat in one bed because the bed will defluidize. Additional tests were performed this month in which the screened MOD image was dusted with silver flake and heat treated to make a thicker deposit than the MOD compound could alone. This process did result in continuous traces rather than the string of dots to which the MOD compound contracts when heated on Kapton without metal powder added. The patterns were extensively shorted because the silver flake tends to coat everything. By scribing between the legs of the conductivity pattern it was possible to get a measure of the conductivity shown in tests F-3 to F-6. The trace thickness is about standard, and the conductivity is poor but not terrible. The patterns pass the Scotch Tape adhesion test.

Silver flake is not an acceptable free-flowing toner product because it is an electrical conductor. This rules out electrostatic imaging which is the ideal ultimate expression of the PARMOD technology. In thinking about how to coat and insulate the finely divided metal particles which we have shown to be an essential component in the toner, it occurred to me that this could be done by adding the metal during the synthesis of the MOD compound. This has proven to be a very successful idea which has provided us with our first samples of free-flowing, insulating toner with promise of achieving all of the goals of the Phase I program. An Invention Report has been submitted on this idea as well.

Two samples of PARMOD toner powders have been tested to date. L-4 is based on silver-coated nickel powder from Novamet, Inc. The toner contains approximately twice as much metal as MOD by weight giving a calculated MOD layer 56% as thick as the radius of the silver-coated nickel sphere. Actually, as shown in Figure 3, the toner is made up of fairly large aggregates of silver-nickel spheres bonded together with MOD compound. An interesting aspect of the SEM work was that the toner was found to be so good an insulator under electron beam bombardment that it was impossible to image. The pictures shown in Figure 3 had to be taken with the Environment SEM with a water vapor atmosphere of 10 Torr to neutralize the charge. The powder was sieved to prepare minus 60 mesh material for coating studies.

Toner L-5 is made with silver flake. The objective was to add four times as much metal as MOD, similar to our standard screen ink, but due to the 75% yield of the MOD synthesis, the metal/MOD ratio came out closer to 5:1. The toner is an insulator to the ohmmeter, but it is conducting in the SEM which produced the pictures in Figure 4. The toner was screened and gave a 57% yield of minus 60 mesh product.

Crude tests of both these toners were conducted by ruling lines on Kapton and epoxy substrates and coating them with powder. The results were free of shorts which was an improvement over silver flake, and they were well bonded and conductive. The problem was that the lines were so thick that it was hard to measure conductivity. Tests with mixtures of polyisobutyl methacrylate-toluene solutions showed that the polymer did not decompose cleanly enough and quickly enough to produce good conductor traces. α-terpineol was satisfactory, and ultimately it was possible to produce thin lines that could be connected with soldered bridge wires to provide a conductivity measurement of 27.5 microohm cm with toner L-5. Further tests were done with screened images of ink B-34.

As shown in Table III, these tests, marked B-34-F5-to-9-L5, gave results similar to those with screen inks. The images are not shorted randomly, and the thickness of the trace is similar to a screened image. The conductivity is half to a quarter of that of bulk silver with toner L-5. Toner L-4 gives a poorer image and unreliable conductivity measurements. Tests -F 11 to 14 were done with the toner in contact with the screened image overnight. This improved the quality of images powdered with silver flake but not these. The conductivity of the L-5 traces was somewhat worse than before, and that of the L-4 was of the order of 15 microohm cm after scoring to remove randomly distributed shorts.

Ink B-39 in Table III was made by dissolving 9 g of L-4 toner in 1.8 g of α-terpineol. It gave a conductivity of nearly 30 microohm cm. These results serve as a baseline for the powder coating tests with toner L-4. The conductivity with the silver-coated nickel powder, some of which will be required for any toner applied by a magnetic brush, is substantially worse than with silver, but some L-4 can probably be blended into L-5 without degrading it unacceptably.

Inks B-43 and B-44 shown in Table III were formulated to provide more tests of toner L-5, particularly for peel strength. Ink B-43 is exactly the same as B-34. Ink B-44 is the same as B34 and B43 except that the α-terpineol vehicle is replaced with α-terpineol containing approximately 8% of 20 nm colloidal silver from Nanophase Technologies.

A comparison of patterns of the two inks without toner addition shows that the colloidal silver markedly improves the image after heat treatment. The pure MOD pattern pulled into discrete dots of silver while the colloidal silver performed the usual function of added metal and preserved at least a semblance of the original image. When powdered with toner L-5, which had been further sieved to minus 200 plus 325 mesh, the conductivity approached that obtained with screen inks, and the images were free of random shorts. They are still rather messy looking with a haze of toner in discrete particles coating the entire substrate. I believe this is due to electrostatic charging of the Kapton substrate, which is powerful. The toner particles are attracted to the substrate and while much of the excess was be removed by tapping the back of the image, a uniformly distributed haze could not be removed, even after double screening to remove the very fine toner dust. We appear to be simulating ink jet printing and electrostatic laser printing in the same tests. Next month we will try to eliminate the electrostatic element to make higher quality images.

Peel Strength Measurements.

Adhesion is a critical parameter in evaluation of circuit board technology. Potential users will want assurance that the traces will stay on the substrate, come what may. Our test pattern is derived from MIL-P-13929 which specifies that the 1/32 inch wide traces be peeled from the pattern while the force is measured. This is not possible with the MOD traces because they are bonded to the substrate so well that they tend to break. This was easily remedied for Kapton samples by cementing the sample face down on an aluminum plate with epoxy resin and peeling the substrate off the metal trace after scribing. To get the most accurate width and a large enough force to measure accurately we used the 1/8 inch traces in the adhesion test pattern. The results were then converted to Newtons per meter of trace width to compare with the target peel strength of 1229 N/m.

The results in all cases cover a range because the adhesion is variable. All the data is presented to give a feeling for this range and because in most cases an average is not very meaningful. The minimum values may be due to many causes including poor adhesion of the epoxy. The maximum values are characteristics of the limits of what is achievable with this substrate system. In all cases the coated Kapton samples had peel strengths less than our target value, often one third to one half of the desired 1229 Newtons per meter (7 lbs per inch). We will try to establish whether this is a failure of the FEP-metal or FEP-Kapton bond next month. In no case did the metal trace itself separate. It either adhered to the aluminum backing plate or to the coated Kapton substrate. There is no obvious correlation of adhesion with the MOD content of the screen ink. This is masked to some degree by the fact that the toner has MOD compound in it.

Table V shows the results of adhesion measurements with inks B-43 and -44 and Plus 325- minus 220 mesh toner L-5. There is no clear effect of the colloidal silver in ink B-44 on the adhesion.

Adhesion of the MOD compound to epoxy-glass substrates is much better than it is to FEP-coated Kapton. Table V shows results on two samples of ink B-9 which was a 1:1.1 mixture of silver neodecanoate and silver flake on FR-4 substrate heated to 200-210 C. A great deal of trouble was encountered in gripping the traces tightly enough to remove them. This was finally overcome by using high strength epoxy and glass reinforcing tape to adhere to the trace. The adhesion data range from half the target value to twice the target value, with the average comfortable above target in most cases. several of the samples delaminated when the trace was pulled off.

Commercialization Activities

Several meetings have been held with Technology Management and Funding to appraise them of the latest results and enlist their efforts in securing Phase III commitments. The most important such meeting was with Bill Marder, TMF's electronic industries expert. Bill was very much taken with the ink jet/laser printing aspect of our technology as applied to Tape Automated Bonding technology. A meeting is scheduled with a printing expert and an ink jet expert to get additional insight, and incidentally build the team for Phase II. A rough outline of the Phase II program has been put together. Major activities are an extension of PARMOD to copper conductors (joint with BOC because of the heavy atmosphere involvement), Extension of PARMOD to resistors, capacitors and the like, and extension of the technology to nonelectronic applications such as optical and decorative coatings on glass metal and plastic. The same arguments about hazardous waste-free coating apply to these applications as to the electronic field, which motivated this program in the first instance.

A near term program to commercialize what we already have will be the subject of discussion with potential industrial partners, some of whom have already been contacted by Alison Sahoo (BS, Physics), our project manager at TMF. Their input to the Phase II program in technical and commercial expertise, as well as in kind participation will be sought to strengthen the Phase II proposal.

Table I

Screen Printing Inks with Variable Metal and MOD Content

Number		Composition-----grams			Results/remarks		
		AgNd	AgF	Vehicle	Resistivity Microohm cm	Silver mass Milligrams (Microns)	Temperature C
B-36		3.0	9	1.5	Less metal than standard mix		
	-1, 2	New screen for 10 μ coating			3.06, 2.99	65.3, 61.6	328
	-3, 4	All but one fail short test			2.87, 3.05	60.2, 64.6	336
	-5, 6	All have opens			3.11, 3.02	63,1, 63.2	347
	-7, 8	Very good appearance			2.98, 2.88	60. 5, 60.2	334
B-37		3.0	15	2.0	More metal than standard		
	-1, 2	New ten micron screen			--, 2.89	--, 76.1	336
	-3, 4	Both pass short			3.10, 3.18	74.0, 77.4	337
	-5,6	Both pass short			3.18, --	74.5, 75.3	335
B-38			9	1.5	Null mixture with no MOD compound		
	-1,2	Very poor screening			~3 (one loop)		343
	-3,4	"			~5, 5.5	--, 102	338
	-5,6	"			8.2, 7.8	108, 106	322

Notes:
AgNd-silver neodecanoate   AgF-silver flake   Vehicle-α-terpineol
Resistance test pattern-3200 squares, 0.396 mm line width
Target 2.1 micro ohm-cm

Table II

Screen Printing Inks

Baseline Data for Powder Coating

	Composition-----grams			Results/remarks		
Number	AgNd	AgF	Vehicle	Resistivity Microohm cm		Temperature C
B-40	2.0	8	1.2	Standard mix		B-40-43
						heated in
-1				3.04	94.4	belt furnace
-2				2.90	95.8	@ 330 for
-3	Half the pattern missing					1 minute
-4	Best pattern			3.03	104.4	nominal
B-41	1.0	8	1.1	Less AgNd		
-1				3.65	115	
-2	poor pattern			3.57	98	
-4	" "			3.25	104	
B-42		8	1.0	No AgNd		
-1	Stuck to ceramic support					
-2				3.54	204	
-3	More than half missing					

Notes:

AgNd-silver neodecanoate   AgF-silver flake   Vehicle-α-terpineol

Resistance test pattern-3200 squares, 0.396 mm line width

Target 2.1 micro ohm-cm

Table III

Screen Printing Inks for Powder Coating

	Composition-----grams			Results/remarks		
Number	AgNd	AgF	Vehicle	Resistivity Microohm cm		Temperature C
B-34	3.0		1.8	Adhesive for fluid bed coating		
-F-3	Coated with silver flake			shorted, not well bonded		317
-F-4	Same aged overnight			11.2	17 microns	322
-F-5,6	"			13.3, 11.2	12, 15 μ	323
-F7-L-5		No shorts		7.6	105 mg	324
-F-8,9-L5		"		7.6, 4.7	122, 123	334
-F10-L-4		poor screening				333
-F11, 12-L-5		Overnight		Not as good as F-8,9		339
-F 13,14- L-4		"		Not as good as 10		335
B-39	3.1	5.8	1.8	Base point for toner L-4 with silver/nickel		
-1,2	Screens very well			28.2, 28.2	60, 64	328
-3,4	Not so good			26.2, ---	54	335
-5,6	Better			27.5, 28.0	54, 53	330
B-43	3.0		1.8	Repeat of B-34		
-1				Dots		
-2-L-5				7.9	83	321
-3-L-5				9.2	79	320
B-44	3.0		1.8	Colloidal silver suspension in vehicle.		
-1				Poor but recognizable		
-2-L-5		Heavily powdered		7.95	103	308
-3-L-5		lightly powdered		8.4	99	333
-4-L-5		Lightly powdered		Poor pattern		

Notes:

AgNd-silver neodecanoate AgF-silver flake Vehicle-α-terpineol
Resistance test pattern-3200 squares, 0.396 mm line width
Target 2.1 micro ohm-cm

Table IV

Peel Strength Tests--Screen Printed Kapton Samples

Sample no.	Newtons/meter (Target 1229 N/m)	Remarks
B-40-1	250-400	Standard screen ink composition.
	250-310	
	250-525	
B-40-2	310-525	
	400-620	
	250-620	
B-40-3	220-460	
	400-620	
	400-620	
B-40-4	460-560	
	460-680	
	250-525	
B-41-1	560-680	Half the standard MOD content.
	400-560	
	370-460	
B-41-2	370-865	Mostly 370
	460-525	
	400-680	Mostly 500
B-41-3	400-710	Mostly 560-620
	525-680	
	400-680	
B-41-4	371-865	Mostly 620
	460-525	
	460-710	
B-42-2	370-620	No MOD content.
	400-620	
	400-775	
B-42-3	310-525	
	250-460	

Table V

Peel Strength Tests--Powder Coated Kapton Samples

Sample no.	Newtons/meter	(Target 1229 N/m)	Remarks
B-43-2	525-620		
	525-620		
B-43-3	370-525		
	310-525		
	310-400		
	250-370		
B-44-2	310-370		
	370-400		
	310-460	Avg. 400	
	400-525	Avg. 460	

Epoxy-Glass Samples

B-9-1	1390-2320	
	775-1700	Pulled the board apart
	1080-2630	Mostly 1500-1600, removed metal
	620-2630	Mostly 1500-1600, pulled board apart
B-9-2	620-2160	Mostly 1550-1850
	930-2160	Mostly 1235-1550
	930-2475	Mostly 1235-1550
	930-1850	Mostly 930-1235

Figure 1.

Sample B30-4 cross sections of direct MOD wire bond showing reasonably solid metal and silver removal after wire removal.

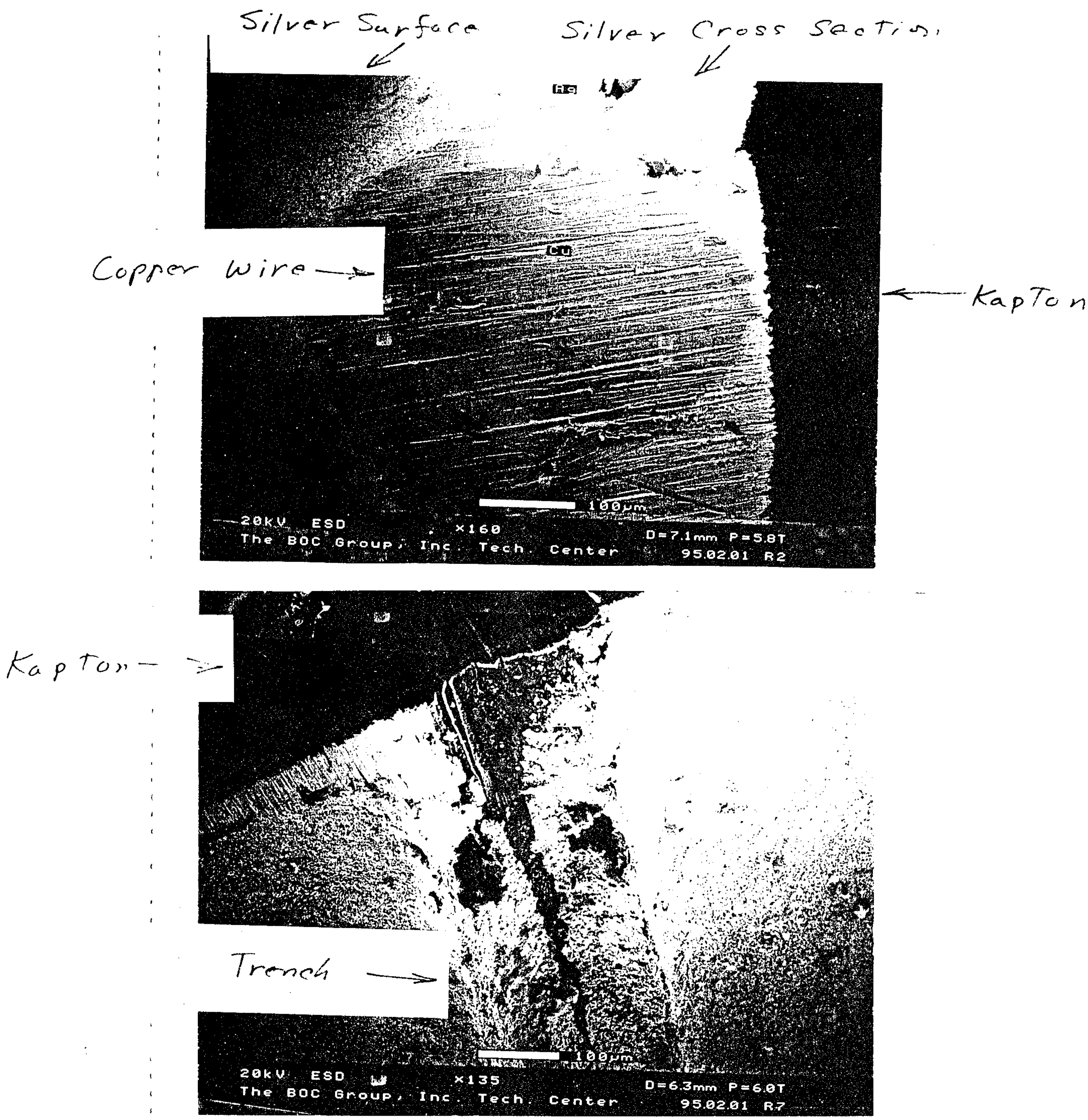


Figure 2

Photomicrograph of bonded copper wire.

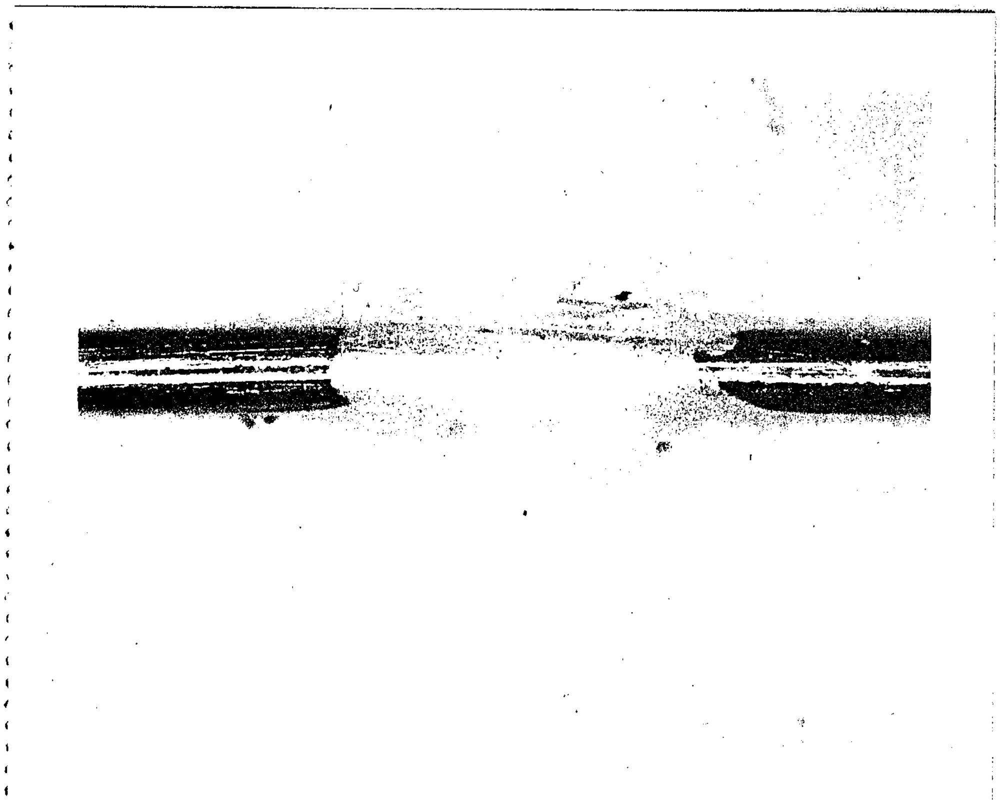
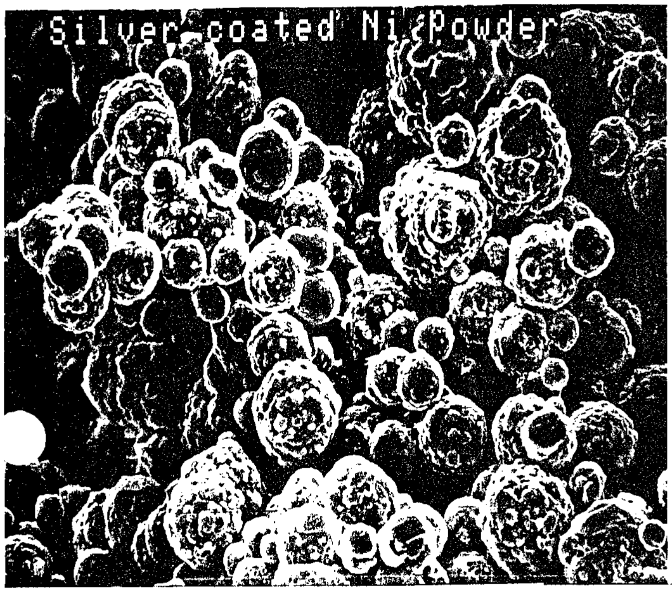


Figure 3

Silver-coated nickel powder and toner L-4



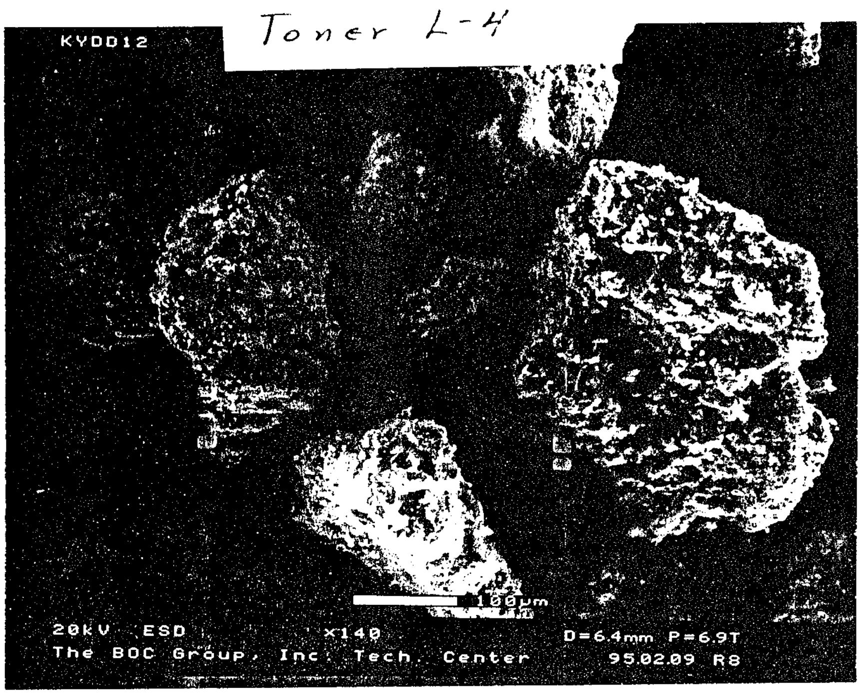
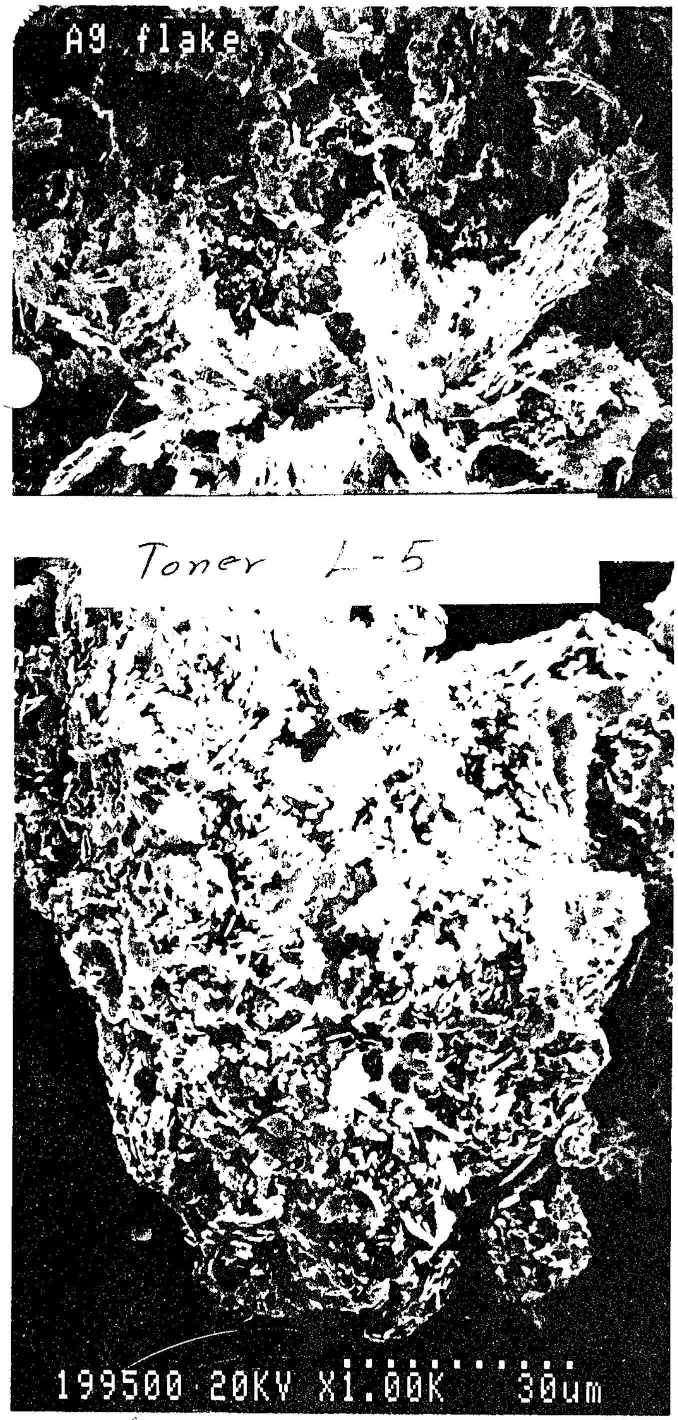


Figure 4.

Aldrich silver flake and toner L-5.



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N00014-94-C-0236
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