

[54] **PHOTOSENSITIVE PROCESS FOR
PRODUCING PRINTED CIRCUITS
EMPLOYING ELECTROLESS DEPOSITION**

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[57]

ABSTRACT

An adhesive system which provides adhesive compositions to be applied to an insulating base and form printed images thereon. The adhesive compositions include agents which render them photosensitive, and receptive to electroless metals.

9 Claims, No Drawings

PHOTOSENSITIVE PROCESS FOR PRODUCING PRINTED CIRCUITS EMPLOYING ELECTROLESS DEPOSITION

This invention relates to an adhesive system whereby a film of adhesive composition is applied to an insulating substrate to form printed circuit images thereon. The adhesive composition comprises agents which render the adhesive layer sensitive to light and receptive to an electroless metal, e.g., copper.

It is an object of this invention to provide an adhesive system which is both light-sensitive and receptive to an electroless metal.

Another object of this invention is to form printed circuit images with an adhesive composition which is receptive to the deposition of an electroless metal.

A further object of this invention is to provide an adhesive composition capable of forming a good bond with an insulating base.

Heretofore, it has been customary to seed and sensitize insulating substrata on which it is desired to electrolessly deposit metal, e.g., copper, by contacting the substrata sequentially with aqueous acidic solutions of stannous tin ions and precious metal ions, e.g., palladium, or with a single acidic aqueous solution comprising a mixture of stannous tin ions and precious metal ions such as palladium. For example, one such treatment involves immersing the insulating base material first in an acidic aqueous solution of stannous chloride followed by washing after which the substratum is immersed in an acidic aqueous solution of palladium chloride. In an alternative system, the substratum is immersed in an acidic aqueous solution comprising a mixture of stannous chloride and palladium chloride.

Seeding systems are known wherein electroless metal deposition is initiated on solid particles catalytic to the deposition of electroless metal and dispersed throughout an organic adhesive base.

The adhesive system of the present invention represents an improvement over the seeding and sensitizing systems heretofore available in that it greatly simplifies the application of printed circuits on an insulated base board.

Among the advantages of the present system is that a printed circuit may be provided without altering or deteriorating the base or insulating base on which the image is printed. With the present adhesive system there is no need to etch the whole base. The image may be added to the base only where it is desired or needed.

Another advantage of the present system is that it provides a clean background for selection of the area where the image may be placed. Also, with the present system, the insulated base is not tampered with by mechanical abrasion, chemical etching or with an overcoating with the adhesive composition.

An important advantage of the present invention is that it provides a sharp, clear and definable photoprint image. With the present system, the images may be printed with the lines being as thin as 0.1 millimeters or less, and the spaces between the lines may also be 0.1 millimeters or less. This advantage of the present adhesive system is emphasized in comparison with the prior silk-screening process where the refinement of the lines could only be as low as 0.5 to 0.8 millimeters.

Moreover, the adhesives of the present system are curable by light and thus, the detrimental heat cure cycles may be eliminated which are necessary in producing images by other systems such as silk-screening.

The adhesive system of the present invention is also readily adaptable to a wide variety of substrata and economical. The present adhesive system in addition to providing printed circuits may be used to provide various designs including metallic designs, decorative designs, and the like on a variety of bases.

The present adhesive system includes an adhesive base composition having dispersed therein a catalyst, e.g. a palladium chloride catalyst and a sensitizing agent, e.g. a diazonium salt. The catalyst and sensitizing agent impart desired properties to the adhesive composition of receptiveness to electroless metals and light-sensitiveness, respectively.

The adhesive base composition of the present invention will ordinarily comprise an adhesive polymer or copolymer, an adhesive resin alone or in combination with a thermosetting resin of the type described hereinbelow, and solvent material. Typical of the adhesive polymers which may be used in the present system are polyvinyl acetal resins, polyvinyl alcohol, polyvinyl acetate, polyvinyl pyrrolidone, polyamides, polyvinyl butyral natural and synthetic rubbers, and the like. Preferred for use as the polymer are acrylonitrile butadiene copolymers, and other polymers containing ethylenic groups.

The resins which may be used according to this invention include thermosetting resins such as allyl phthalate; furane; melamine formaldehyde; phenol formaldehyde and phenol-furfural copolymer, alone or compounded with butadiene acrylonitrile copolymer or acrylonitrile-butadiene-styrene copolymers; polyacrylic esters; silicones; polyesters; and the like.

The adhesive base composition may also include phenolic resins such as bromo methyl alkylated phenol formaldehyde and thermosetting phenolics, to reinforce and impart adhesive properties to the adhesive base compositions. Certain epoxy resins, epoxy-urethane resins, and urethane resin also may be used to enhance the adhesive properties of the adhesive compositions.

The adhesive base compositions of the present adhesive system have dispersed therein an agent which is catalytic to the reception of an electroless metal dissolved in an organic system which may comprise one or more organic solvents, one or more organic polymers, or one or more organic solvents in combination with one or more organic resins.

The catalytic agent may be a salt or an oxide, or may be any of the metals of Groups 8 and 1B of the Periodic Table of Elements, compounds of any of these metals or mixtures of such metals. Salts or oxides of iron, cobalt, and iridium will ordinarily be used as the dissolved catalytic agent, with a palladium chloride catalyst being preferred, i.e. a catalyst identified as PEC-8 according to the present invention, the preparation of which is described hereinbelow.

Any organic solvent which is capable of dissolving the catalytic salts or oxides are used, providing the organic solvent does not precipitate the adhesive from its solvents.

Preferred catalytic compounds for dissolution in polymers and solvents of the type described are chlorides, bromides, fluorides, fluoroborates, iodides, nitrates, sulfates, acetates, and oxides of the metals and elements described hereinabove. Complexes of rhodium, iridium, platinum and palladium with stannous chloride are also suitable catalysts for dissolution in the polymers and solvents.

According to the present invention the adhesive compositions are rendered sensitive to light by adding to the adhesive compositions diazonium compounds, azides, ferrous compounds or bichromate compounds dissolved in organic solvents as sensitizers.

The diazonium compounds that may be suitably used according to the present invention include p-diethylamino-2-ethoxybenzenediazonium chlorozincate, 4 ethylamino-3-methyl benzenediazonium chlorozincate, p-morpholenobenzenediazonium fluoroborate, 4-Diethylamino-2-methylbenzene-diazonium chlorozincate, 2-butyl-3-diazo-3-pseudoindole, p-amino-N-benzyl-N-ethylbenzene diazonium chlorostanate, p-Amino-N-benzyl-N-ethylbenzene diazonium chlorozincate, 3-chlor-4-diethylamino benzene diazonium chlorozincate, p-diethylaminobenzene-diazonium fluoroborate, p-diethylaminobenzene-diazonium chlorozincate, 3-diazo-2-methyl-5-phenyl-3-pseudoindole, 3-diazo-2(2-naphthyl)-3-pseudoindole, 3-diazo-5-nitro-2-phenyl-pseudoindole, 3-diazo-2-phenyl-3-pseudoindole, 3-diazoindazole, 1-diazo-1-benz(e) indazole, 3-diazo-5,7-dibromindiazole, 3-diazo-5-phenylindiazole, 3-diazo-2,4-diphenyl-3H-pyrrolenine, 2-p-chlorophenyl-3-diazo-4,5-diphenyl-3H-pyrrolenine, 2,5-bis(p-chlorophenyl)-3-diazo-3H-pyrrolenine, 2-(4-biphenyl)-3-diazo-4,5-diphenyl-3H-pyrrolenine, N-methyl-4'-(3-diazo-4,5-diphenyl-3H-pyrrolen-2-yl)-benzenesulfonanilide, 3-diazo-2,4,5-triphenyl-3H-pyrrolenine, and 4-diazo-2,5-diphenyl-4H-isoimidazole.

The aromatic azide compounds that may be used as a sensitizer include 4,4'-diazidostibene, p-Phenylbis(azide), p-Azidabenzophene, 4,4'-diazidobenzophenone, and 4,4'-diazidodiphenylmethane.

The organic solvents in which the diazonium compounds, azides, and other sensitizing compounds are dissolved include diethyl formamide, dimethyl acetamide, methyl cellosolve, n-methyl-2-pyrrolidone, dioxane and the like. The sensitizer compounds are dissolved in organic solvent concentrates to provide a more uniform distribution of the sensitizer in the adhesive system. The compounds are added to the solvent and dissolved using mechanical agitation.

There are certain types of dyes and pigments which have been found to increase the photosensitizing activity of the diazonium salt in the adhesive films to light.

The dyes are of a chemical class which include triarylmethane, anthraquinone, stilbene, xanthene, monazo compounds, e.g., Eriocyanine A, Alizarin, Alizarin Blue S, Erythrosin, Eriochrome Black T, Iosol Greene, and the like.

The pigments include, for example, ammonium ferro-ferri cyanide, phthalocyanine blue, zirconium silicate, cab-o-sil, aluminum stearate and the like. The pigments were also found to strengthen the adhesive film and provide a color contrast on certain types of base materials.

There are phenolics and epoxy resins which have been found to impart adhesive properties to the base polymers. The phenolics include bromo methyl alkylated phenol formaldehyde, oil soluble heat reactive phenolics, and thermosetting phenolics.

A chlorosulfonated polyethylene elastomer can also be used to reinforce and impart adhesive properties to the base polymers. The bromo methyl alkylated phe-

nol-formaldehyde resin may also be used to improve the photoprintable properties of the adhesive system.

Typical of the electroless copper solutions which may be used are those described in U.S. Pat. No. 3,095,309, the description of which is incorporated herein by reference. Conventionally, such solutions comprise a source of cupric ions, e.g., copper sulfate, a reducing agent for cupric ions, e.g., formaldehyde, a complexing agent for cupric ions, e.g., tetrasodium ethylenediaminetetraacetic acid, and a pH adjustor, e.g., sodium hydroxide.

Typical electroless nickel baths which may be used are described in Brenner, "Metal Finishing," November 1954, pages 68 to 76, incorporated herein by reference. They comprise aqueous solutions of a nickel salt, such as nickel chloride; an active chemical reducing agent for the nickel salt, such as the hypophosphite ion; and a complexing agent, such as carboxylic acids and salts thereof.

Electroless gold plating baths which may be used are disclosed in U. S. Pat. No. 2,976,181, hereby incorporated herein by reference. They contain a slightly water soluble gold salt, such as gold cyanide, a reducing agent for the gold salt, such as the hypophosphite ion, and a chelating or complexing agent, such as sodium or potassium cyanide. The hypophosphite ion may be introduced in the form of the acid or salts thereof, such as the sodium, calcium and the ammonium salts. The purpose of the complexing agent is to maintain a relatively small portion of the gold in solution as a water soluble gold complex, permitting a relatively large portion of the gold to remain out of solution as a gold reserve. The pH of the bath will be about 13.5, or between about 13 and 15, and the ion ratio of hypophosphite radical to insoluble gold salt may be between about 0.33 and 10.1.

In the following examples, Epon 1001 is a epoxy resin made by reacting bisphenol A with epichlorohydrin, having a melting point of 64°-76°C., and an epoxide equivalent of 450-525. SP-1055 resin is a heat reactive, bromo-methyl alkylated phenolformaldehyde resin having a melting point of 135°C. SP-126 resin is an oil soluble, heat reactive lump form, phenolic resin, having a melting point of 155° and a specific gravity of 1.10. SP-6600 resin is a powdered phenolic resin containing 6 to 8 percent hexa and has a melting point of 155°-170°F. and a specific gravity of 1.15-1.20.

The following example illustrates the method of producing the palladium chloride catalyst PEC-8.

EXAMPLE 1

642 grams of dimethyl formamide solvent was added to 3685 grams of an epoxy resin in a reaction vessel. The mixture was stirred until the epoxy resin was dissolved in the solvent. The mixture was continuously stirred and heated to a temperature of about 60°C. Then 73.3 grams of palladium chloride was slowly added to the mixture and stirred and heated to a temperature range of 110° to 120°C. The resulting mixture was stirred at this temperature for a period of 1 hour. The liquor was a deep black opaque color. The heat was then removed and the mixture was continued to be stirred and cooled to approximately 80°C. Then the material was stored in a non-metallic or a suitably lined metal drum.

The formulations of photosensitizers of this invention which are distributed in the present adhesive system

are illustrated in the following examples. The sensitizer salts, i.e., diazonium salts, are dissolved in organic solvent concentrates to promote a more uniform method of distribution of the sensitizer in the adhesive system. The salts are added to the solvent and dissolved by mechanical agitation.

EXAMPLE 2

Dimethyl Formamide	395 grams
p-diethylamino-2-ethoxy benzenediazonium chlorozincate	5 grams

EXAMPLE 3

Dimethyl Formamide	490 grams
4-Ethylamino-3-methyl-benzenediazonium chlorozincate	6-10 grams

EXAMPLE 4

Dimethylformamide	490 grams
p-Morpholinobenzenediazonium fluoroborate	10 grams

The sensitizers described in Examples 2 through 4 may be combined with a dye or pigment, and the PEC-8 catalyst. This combination is then distributed in the adhesive system.

Typical formulations of this combination of the catalyst, dye and sensitizers are illustrated in the following examples.

EXAMPLE 5

Eriocyanine A	0.1 grams
PEC-8 catalyst	2.0 grams
Sensitizer of example 4	50 milliliters

EXAMPLE 6

Iosol Green	0.4 grams
PEC-8 catalyst	2.0 grams
Sensitizer of example 4	50 milliliters

EXAMPLE 7

Erythrosin B cert	0.20 grams
PEC-8 catalyst	2.0 grams
Sensitizer of example 4	50.0 milliliters

The dyes and pigments together with the PEC-8 catalyst or some other suitable catalyst may be added to an adhesive base material prior to the addition of the sensitizer to form the present adhesive system.

The formulation of various adhesive bases are described in the following examples.

EXAMPLE 7A

Methyl Ethyl Ketone	grams
Acrylonitrile butadiene (Hycar 1431)	1600
Phenolic Resin (SP-1055)	380
	20

EXAMPLE 8

Ethylene glycol monoethyl ether acetate (Cellosolve acetate)	grams
Methyl Ethyl Ketone	175
Acrylonitrile-butadiene (Hycar 1431)	150
Pyrogenic fumed Silicon dioxide (Cab-o-sil)	grams
PEC-8 catalyst	36
Toluene	6
Oil Soluble Heat Reactive Phenolic resin (SP 126)	12
Thermosetting Phenolic resin (SP-6600)	50
Bisphenol A type epoxy resin (Epon 1001)	15
	5
	10

EXAMPLE 9

Ethylene glycol monoethyl ether acetate	grams
	1906.8

Silane Resin	11.8
PEC-8 catalyst	68.1
Pyrogenic fumed Silicon dioxide (Cab-o-sil)	59.0
Zirconium Silicate	118.0
Acrylonitrile-butadiene	376.0
Thermosetting Phenolic Resin	59.0
Oil Soluble Heat Reactive Phenolic Resin	177.0
Bisphenol A type epoxy resin	118.0
High Flash Naptha Solvent	1050.0
Chlorosulfonated Polyethylene	118.0

EXAMPLE 10

10 Ethylene glycol monoethyl ether acetate (Cellosolve acetate)	175
Methyl Ethyl Ketone	150
Acrylonitrile-butadiene (Hycar 1431)	36
Pyrogenic fumed Silicon dioxide (Cab-o-sil)	6
AuCl ₃ (in 50 grams of N-methyl-2-pyrrolidone)	2.0
Toluene	50
15 Oil Soluble Heat Reactive Phenolic resin (SP 126)	15

EXAMPLE 11

20 Ethylene glycol monoethyl ether acetate	Grams
Methyl Ethyl Ketone	50
Acrylonitrile-butadiene	200
Triethanolamine	30
Pyrogenic fumed silicon dioxide	0.3
PEC-8 catalyst	1.0
	3.0

EXAMPLE 12

25 Methyl Ethyl Ketone	grams
Acrylonitrile-butadiene	2000
Phenolic resin (SP-1055)	300
PEC-8 catalyst	40
Ethylene glycol monoethyl ether acetate	40
	620

EXAMPLE 13

35 Ethylene glycol monoethyl ether acetate (Cellosolve acetate)	175
Methyl Ethyl Ketone	150
Acrylonitrile-butadiene (Hycar 1431)	36
Pyrogenic fumed Silicon dioxide (Cab-o-sil)	6
AuCl ₃	
Stannous Chloride	(in 50 grams of N-methyl-2-pyrrolidone)
Toluene	2.00
Oil Soluble Heat Reactive Phenolic Resin (SP 126)	1.50
	50
40	15

EXAMPLE 14

45 Ethylene glycol monoethyl ether acetate (Cellosolve acetate)	grams
Methyl Ethyl Ketone	175
Acrylonitrile-butadiene (Hycar 1431)	150
Pyrogenic fumed Silicon dioxide (Cab-o-sil)	36
AgNO ₃	6
Toluene	2.0
Oil Soluble Heat Reactive Phenolic resin	50
	15

The adhesive bases of the type illustrated above and described in the various examples are then mixed with photosensitizers and solvents to produce a photosensitive adhesive system according to this invention whereby a film of adhesive material is applied to various insulated bases. The formulations of the adhesive systems are illustrated in the following examples.

EXAMPLE 15

To 100 grams of the adhesive base material of example 9, there was slowly added 60 milliliters of the photosensitizer of example 2, i.e. p-diethylamino-2-ethoxy benzenediazonium chlorozincate and 40 grams of a solvent, dimethyl formamide. The sensitizer was added during mechanical agitation of the adhesive base material and sufficient time was allowed to assure complete distribution of the sensitizer. The solid content of the adhesive base was adjusted by the addition of the sol-

vent to produce the desired film thickness of the photosensitive adhesive material to be applied to a base.

A film of the photosensitive material was applied to a base and allowed to dry and was exposed to ultra violet light. After being exposed, the undeveloped portion of the film was removed from the base and a printed circuit image was formed thereon. Then the printed circuit image was electrolessly plated in a copper solution. A good bond was obtained between the copper plating and the adhesive printed circuit image.

The bond was determined by the force required to peel the electroless copper at a 90° angle from the adhesive image. The force is measured as the "peel strength" in pounds per inch width of plated metal removed.

The peel strength of the bond was about 6.0 lbs. per inch width of the copper removed from the printed adhesive image.

EXAMPLE 16

Another adhesive composition was prepared as described in example 15 which consisted of the following:

Adhesive base of example 11	100 grams
Photosensitizer of example 3	35 milliliters
Methyl Ethyl Ketone	40 grams

A film of the composition was applied to a base. The film was exposed to ultra-violet light, developed and electrolessly plated with a copper solution.

The peel strength of the bond of the plated copper to the adhesive image was about 7.0 lbs. per inch width.

EXAMPLE 17

A third adhesive composition was prepared as described in Example 15, and consisted of the following:

Adhesive base of example 12	100 grams
Ethylene glycol monoethyl ether acetate	30 grams
Zirconium Silicate	1.0 grams
Phthalocyanine Blue	0.3 grams
Photosensitizer of example 4	50 milliliters

The peel strength of the bond of adhesive image to the plated copper was about 6 lbs. per inch width.

EXAMPLE 18

A fourth adhesive composition was prepared, and consisted of the following:

N-methyl-2-pyrrolidone	25.0 grams
Zinc chloride	1.0 grams
1, 3, 6 Napthalene tri sulfonic Acid tri sodium salt	0.3 grams
Fluoboric acid	1.0 grams
4-Diethylamino-2-methylbenzene-diazonium chlorozincate	2.0 grams
Anionic Wetting Agent	0.3 grams
PEC - 8	3.3 grams
Aniline Blue (spirit soluble)	0.1 grams
Adhesive base of example 7A	100.0 grams
High Flash Naphtha	100.0 grams
Methyl Ethyl Ketone	200.0 grams
Pyrogenic fumed Silicon dioxide (cab-o-sil)	3.0 grams

The peel strength of the bond of adhesive image to the plated copper was about 6 lbs. per inch width.

The photosensitive adhesive material may be applied to a base or substrate by any conventional means suitable for applying a uniform coat. The preferred thickness is 0.001 inch. The coating is dried, such as in an oven set at 45° to 70°C for a minimum of 10 minutes or until the film is dry to the touch. Films of more than 0.001 inch may require a longer time in the oven.

After the adhesive film was dried, it is exposed through a negative or positive circuit pattern using an ultra-violet light source, e.g., carbon arc, pulsed xenon, mercury lamp. The time for the exposure depends on the thickness of the adhesive film, the type of base material and the light source.

Following the exposure cycle of the adhesive film, the printed circuit image is developed by removing the unexposed portions of the adhesive film by using a suitable solvent. Such solvents may be methyl ethyl ketone; 1, 1, 1-trichloroethane; trichlorethylene; cyclohexanone; or any combination of these solvents.

One method of developing the printed circuit image is the pressure spray technique. It is particularly effective in removing tenacious residues caused by the pressure of inert fillers and pigments. Generally, multiple spray rinses are required to assure a residual free background for the plating of the electroless metal. For example, 1, 1, 1-trichloroethane may be used in the first spray rinse, and isopropyl alcohol or water may be used in a second and/or third spray rinse.

The developed printed circuit image is then oven dried for about 10 to 20 minutes from 60° to 70°C or until the solvents are evaporated. Then the developed image is precured for about 1 hour at 160°C before it is ready for electroless metal deposition.

Before the printed circuit image is electrolessly plated, it may be activated to remove surface contamination and to open the surface of the adhesive material to expose the catalytic sites which are receptive to electroless metal deposition.

The adhesive material, for example, may be activated by being successively (a) immersed in a chromic-fluoboric acid etchant for about 5 to 10 minutes, (b) water rinsed for about 5 minutes, (c) immersed in a sodium bisulfite neutralizer for about 1 minute and (d) finally rinsed with water for about 5 minutes. The time in the chromic-fluoboric etchant is dependent on the film thickness of the photoprinted adhesive material during the immersion period.

The image may be plated with an electroless metal by any conventional method. For example, the printed circuit image may be immersed in an electroless copper bath for a period of 1/2 to 1 hour, for complete coverage of the adhesive image with copper. The length of time in the electroless copper bath is dependent on the density of the printed circuit pattern and the desired copper thickness.

The invention in its broader aspects is not limited to the specific methods, compositions and improvement described herein, but departures may be made within the scope of the appended claims without departing from the principles of the invention and without sacrificing its chief advantages.

What is claimed is:

1. A process for producing printed circuits on an insulated base which comprises applying to said base a film of a copolymer of acrylonitrile-butadiene having dispersed therein a catalyst selected from the group consisting of the salts, oxides or metals of Groups 1B and 8 of the Periodic Table of Elements including mixtures thereof and a light sensitive diazonium compound for enhancing the light sensitiveness of said composition, exposing said film imagewise to an actinic light source, removing the unexposed portion of the film with an organic solvent for said film whereby printed circuit images are formed on the surface of the base in

exposed areas, and immersing the printed circuit images in an electroless metal plating solution containing the metallic salt of the metal to be plated and a reducing agent.

2. A process according to claim 1 wherein said diazonium compounds are selected from the group consisting of p-diethyl-amino-2-ethoxy benzenediazonium chlorozincate, 4-ethylamino-3-methyl-benzenediazonium chlorozincate, p-diethyl-amino-2-ethoxybenzene-diazonium chlorozincate, 4-diethylamino-2-Methyl-benzenediazonium chlorozincate and p-morpholinobenzene-diazonium fluoroborate.

3. A process according to claim 1 wherein the catalyst is palladium chloride.

4. A process according to claim 1 wherein the catalyst comprises palladium chloride in admixture with an epoxy resin and an organic solvent.

5. A process according to claim 1 wherein the electroless metal plating solution contains a source of cupric ions.

6. A process for producing printed circuits on an insulated base which comprises applying to said base a film of a composition which comprises acrylonitrile-

butadiene and a thermosetting phenolic or epoxy resin having dispersed therein a catalyst selected from the group consisting of the salts, oxides or metals of Groups 1B and 8 of the Periodic Table of Elements including mixtures thereof and a light sensitive diazonium compound for enhancing the light sensitiveness of said composition, exposing said film imagewise to an actinic light source, removing the unexposed portion of the film with an organic solvent for said film whereby printed circuit images are formed on the surface of the base in the exposed areas, and immersing the printed circuit images in an electroless metal plating solution containing the metallic salt of the metal to be plated and a reducing agent.

7. A process according to claim 6 wherein the catalyst is palladium chloride.

8. A process according to claim 6 wherein the catalyst comprises palladium chloride in admixture with an epoxy resin and an organic solvent.

9. A process according to claim 6 wherein the electroless metal plating solution contains a source of cupric ions.

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