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(71) Applicant (for all designated States except US): UV SPECIALTIES, INC. [US/US]; 48 N. Airport Drive, Kimball, MI 48074 (US).

(72) Inventor; and

(75) Inventor/Applicant (for US only): KROHN, Roy, C. [US/US]; 3540 Orvall Drive, Fort Gratiot, MI 48059 (US).

(74) Agents: PROSCIA, James, W. et al.; Brooks & Kushman, 1000 Town Center, Twenty-Second Floor, Southfield, MI 48075 (US).

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(54) Title: UV CURABLE TRANSPARENT CONDUCTIVE COMPOSITIONS

(57) Abstract: The present invention discloses an ultraviolet light curable transparent conductive composition and method for making such a composition that may be used to produce a transparent conductive coating on a suitable substrate. These coatings may be used in such applications as touch screens, membrane switches, TV screens, and VCRs. The disclosed composition does not contain any significant amount of volatile organic.

UV CURABLE TRANSPARENT CONDUCTIVE COMPOSITIONS

TECHNICAL FIELD

The present invention relates to ultraviolet light (UV) curable compositions capable of producing a transparent conductive coating.

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BACKGROUND OF THE INVENTION

UV radiation curable transparent conductive compositions are applied to a substrate through spraying, screen printing, dipping or brushing, thus forming a transparent conducting film. Transparent conducting films find application in automobiles, airplanes, etc. as electrodes for liquid crystal devices, exothermic resistors and photosemiconductors.

10

The UV curable conductive films offer advantages over typical heat curable films typically produced by chemical vapor deposition, sputtering and sol-gelling. Heat curable compositions require the use of organic solvents that contain a significant amount of volatile organic compounds (VOCs). These VOCs escape into the atmosphere while the heat curable composition dries. Such solvent based systems are undesirable because of the hazards and expenses associated with VOCs. The hazards include water and air pollution and the expenses include the cost of complying with strict government regulation on solvent emission levels. In contrast, UV curable compositions contain reactive monomers instead of solvents; thus eliminating the detrimental effects of the VOCs.

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The use of heat curable compositions not only raises environmental concerns but other disadvantages exist with their use as well. Heat curable compositions suffer from slow cure times which lead to decreased productivity. These compositions require high energy for curing due to energy loss as well as the energy required to heat the substrate. Additionally, many heat curable compositions yield poor film properties that result in decreased value of the end product.

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Although UV curable compositions exhibit superior properties and performance over their heat curable counterparts, UV curable compositions themselves suffer from certain disadvantages. Generally, UV compositions have high molecular weights and a substantial degree of cross linkage due to the highly reactive nature of the composition. As a result, many of these compositions suffer from low durability and resin shrinkage. With the use of many such compositions, an inordinately high amount of UV light is required to cure. With some compositions, suspended solids fall out of solution after a period of one to two days. This dispersion adversely affects the gloss and clarity of the finished product.

Accordingly, there exists a need to provide environmentally safe UV curable transparent conductive compositions which exhibit improved appearance and workability. Additionally, there is a need to provide a method of applying an improved composition which furthers the goal of improved performance.

SUMMARY OF INVENTION

It is an object of the present invention to provide an improved composition that upon curing by ultraviolet light produces a transparent conductive coating.

It is another object of the present invention to provide an improved composition suitable for use in touch screens, membrane switches, TV screens, and VCRs.

It is another object of the present invention to provide an improved composition suitable for coating a suitable substrate that can be applied by spraying, screen printing, dipping, and brushing.

It is still another object of the present invention to provide an improved composition that does not contain any significant amount of volatile organic solvents that do not become incorporated in the active layer after curing.

The present invention discloses an ultraviolet light curable transparent conductive composition and method for making such a composition that may be used to produce a coating on a suitable substrate. The disclosed composition does not contain any significant amount of volatile organic solvents that do not become
5 incorporated in the active layer after curing. Specifically, the transparent conductive composition contains 5% or less volatile organic solvents by weight.

In accordance with one aspect of the invention, an ultraviolet light curable transparent conductive composition is provided. The transparent conductive composition comprises an aliphatic acrylated oligomers, wherein the aliphatic
10 acrylated oligomer is present in an amount of about 10% to 40% of the transparent conductive composition. All percentages of the transparent conductive composition as expressed in this document refer to the weight percentage of the stated component to the total weight of the transparent conductive composition in its fluid state at standard temperature and pressure.

The transparent conductive composition preferably comprises an
15 acrylated epoxy oligomer in an amount of about 3% to 11%, an isobornyl acrylate monomer in an amount of about 10% to 40% of the transparent conductive composition, a photoinitiator in an amount of about 2% to 10% of the transparent conductive composition, a flow promoting agent in an amount of about 0.1% to 8%
20 of the transparent conductive composition, and a conductive powder in an amount of about 20% to 50% of the transparent conductive composition.

In accordance with yet another aspect of the invention, a method is provided for depositing a transparent conductive coating on a substrate. The method comprises a first step of applying to the substrate a transparent conductive fluid-
25 phase composition ("transparent conductive composition"). The transparent conductive composition comprises a mixture of aliphatic acrylated oligomers, wherein the aliphatic acrylated oligomer is present in an amount of about 10% to 40% of the transparent conductive composition. The transparent conductive composition preferably comprises an acrylated epoxy oligomer in an amount of about
30 3% to 11%, an isobornyl acrylate monomer in an amount of about 10% to 40% of

the transparent conductive composition, a photoinitiator in an amount of about 2% to 10% of the transparent conductive composition, a flow promoting agent in an amount of about 0.1% to 8% of the transparent conductive composition, and a conductive powder in an amount of about 20% to 50% of the transparent conductive composition.

The method also includes a second step of illuminating the transparent conductive composition on the substrate with an ultraviolet light to cause the transparent conductive composition to cure into the transparent conductive coating.

In accordance with this method, the transparent conductive composition can be selectively deposited on the substrate at specific locations where transparent conductive plating is desired. It need not be applied to the entire substrate.

BEST MODE FOR CARRYING OUT THE INVENTION

Transparent conductive Compositions

Reference will now be made in detail to presently preferred compositions or embodiments and methods of the invention, which constitute the best modes of practicing the invention presently known to the inventor.

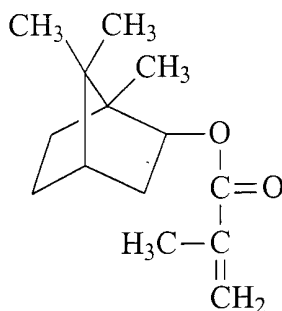
In accordance with one aspect of the invention, a presently preferred ultraviolet light curable transparent conductive composition ("transparent conductive composition") is provided. In this preferred embodiment, the transparent conductive composition includes a mixture of aliphatic acrylated oligomers. The aliphatic acrylated oligomer mixture is present in an amount of about 10% to 40% of the transparent conductive composition. The aliphatic acrylated oligomer mixture is more preferably present in an amount of about 20% to 30%, and most preferably about 27%. The aliphatic acrylated oligomer preferably comprises one or more urethane oligomers. Suitable aliphatic acrylated oligomers include Radcure Ebecryl 244 (aliphatic urethane diacrylate diluted 10% with 1,6-hexanediol diacrylate),

Ebecryl 264 (aliphatic urethane triacrylate diluted 15% with 1,6-hexanediol diacrylate), Ebecryl 284 (aliphatic urethane diacrylate diluted 12 % by weight with 1,6-hexanediol diacrylate) urethanes, commercially available from Radcure UCB Corp. of Smyrna, Georgia; Sartomer CN-961E75 (aliphatic urethane diacrylate blended with 25% ethoxylated trimethylol propane triacrylate), CN-961H81 (aliphatic urethane diacrylate blended with 19% 2(2-ethoxyethoxy)ethyl acrylate), CN-963A80 (aliphatic urethane diacrylate blended with 20% tripropylene glycol diacrylate), CN-964 (aliphatic urethane diacrylate), CN-966A80 (aliphatic urethane diacrylate blended with 20% tripropylene glycol diacrylate), CN-982A75 (aliphatic urethane diacrylate blended with 25% tripropylene glycol diacrylate) and CN-983 (aliphatic urethane diacrylate), commercially available from Sartomer Corp. of Exton, Pennsylvania; TAB FAIRAD 8010, 8179, 8205, 8210, 8216, 8264, M-E-15, UVU-316, commercially available from TAB Chemicals of Chicago, Illinois; and Echo Resin ALU-303, commercially available from Echo Resins of Versaille, Missouri; and Genomer 4652, commercially available from Rahn Radiation Curing of Aurora, IL. The preferred aliphatic acrylated oligomers include Ebecryl 264 and Ebecryl 284. Ebecryl 264 is an aliphatic urethane triacrylate of 1200 molecular weight supplied as an 85% solution in hexanediol diacrylate. Ebecryl 284 is aliphatic urethane diacrylate of 1200 molecular weight diluted 10% with 1,6-hexanediol diacrylate. Combinations of these materials may also be employed herein.

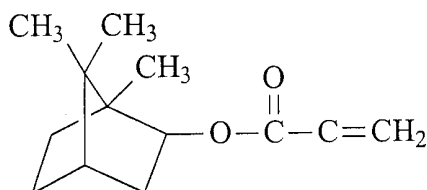
This preferred transparent conductive composition further includes an acrylated epoxy oligomer. The acrylated epoxy oligomer is present in an amount of about 3% to 11%. The acrylated epoxy oligomer is more preferably present in an amount of about 5% to 9%, and most preferably about 7%. Suitable acrylated epoxy oligomers include Radcure Ebecryl 3603 (novolac epoxy acrylate diluted 20% by weight with tripropylene glycol diacrylate), commercially available from Radcure UCB Corp.; Sartomer CN-120 (difunctional bisphenol based epoxy acrylate) and CN-124 (difunctional bisphenol based epoxy acrylate), commercially available from Sartomer Corp.; and Echo Resin TME 9310 and 9345, commercially available from Echo Resins. The preferred acrylated epoxy oligomer is Ebecryl 3603, which is a tri-functional acrylated epoxy novolac. Combinations of these materials may also be employed herein.

The preferred transparent conductive composition also includes an isobornyl acrylate monomer in an amount of about 10% to 40%. The isobornyl acrylate monomer is more preferably present in an amount of about 20% to 35%, and most preferably about 28%. Suitable isobornyl acrylate monomers include

5 Sartomer SR-423 (isobornyl methacrylate):



and SR-506 (isobornyl acrylate):



available from Sartomer Corp.; Radcure IBOA (isobornyl acrylate), commercially available from Radcure Corp.; IBOA and IBOMA, commercially available from CPS Chemical of Bradford, England; and Genomer 1121, commercially available from

10 Rahn Radiation Curing. The preferred isobornyl acrylate monomer is Radcure IBOA, commercially available from Radcure Corp. Radcure IBOA is a high purity, low color monomer. Combinations of these materials may also be employed herein.

This preferred transparent conductive composition also includes a photoinitiator in an amount of about 2% to 10% of the transparent conductive composition. The photoinitiator is more preferably present in an amount of about

15 4% to 6%, and most preferably about 5%. Suitable photoinitiators include Irgacure 184 (1-hydroxycyclohexyl phenyl ketone), Irgacure 907 (2-methyl-1-[4-(methylthio)phenyl]-2-morpholino propan-1-one), Irgacure 369 (2-benzyl-2-N,N-

dimethylamino-1-(4-morpholinophenyl)-1-butanone), Irgacure 500 (the combination of 50% 1-hydroxy cyclohexyl phenyl ketone and 50% benzophenone), Irgacure 651 (2,2-dimethoxy-1,2-diphenylethan-1-one), Irgacure 1700 (the combination of 25% bis(2,6-dimethoxybenzoyl-2,4,4-trimethyl pentyl) phosphine oxide, and 75% 2-hydroxy-2-methyl-1-phenyl-propan-1-one) DAROCUR 1173 (2-hydroxy-2-methyl-1-phenyl-1-propane) and DAROCUR 4265 (the combination of 50% 2,4,6-trimethylbenzoyldiphenyl-phosphine oxide and 50% 2-hydroxy 2-methyl-1-phenyl-propan-1-one), available commercially from Ciba-Geigy Corp., Tarrytown, N.Y.; CYRACURE UVI-6974 (mixed triaryl sulfonium hexafluoroantimonate salts) and CYRACURE UVI-6990 (mixed triaryl sulfonium hexafluorophosphate salts) available commercially from Union Carbide Chemicals and Plastics Co. Inc., Danbury, Conn.; and Genocure CQ, Genocure BOK, and Genocure M.F., commercially available from Rahn Radiation Curing. The preferred photoinitiator is Irgacure 1700 commercially available from Ciba-Geigy of Tarrytown, New York. Combinations of these materials may also be employed herein.

The preferred transparent conductive composition still further includes a flow promoting agent in an amount of about 0.1% to 8%. The flow promoting agent is more preferably present in an amount of about 3% to 5%, and most preferably about 3.5%. Suitable flow promoting agents include Genorad 17, commercially available from Rahn Radiation Curing; and Modaflow, commercially available from Monsanto Chemical Co., St. Louis, Missouri. The preferred flow promoting agent is Modaflow which is an ethyl acrylate and 2-ethylhexyl acrylate copolymer that improves the flow of the composition. Combinations of these materials may also be employed herein.

The preferred transparent conductive composition still further includes a conductive powder in an amount of about 20% to 50%. The conductive powder is more preferably present in an amount of about 30% to 40%, and most preferably about 33%. Suitable conductive powders include silver powder, Minatec 30, and Minatec 40. The preferred conductive powders are Minatec 30 and Minatec 40 commercially available from EM Industries located in Hawthorne, New York.

To illustrate, the following example sets forth a presently preferred transparent conductive composition according to this aspect of the invention.

Example 1

This example provides a preferred transparent conductive composition according to the invention. The transparent conductive composition was made from the following components:

Component	Approximate Mass %
Ebecryl 264	26.7
IBOA	28.3
Irgacure 1700	5.0
Ebecryl 3603	6.6
Modaflow	3.5
Minatec 30	33.4
Total	100.00

In this example the IBOA and Irgacure 1700 are mixed in a pan with a propeller blade mixer for 30 seconds at a speed of 500 to 1000 rpm. In the next step, the Ebecryl 264, the Ebecryl 3603, and Modaflow are introduced into the pan and mixed for 1 to 2 minutes at a speed of 2000 rpm. In the final step, the Minatec 30 is added and mixed at 2000 rpm for 1 to 2 minutes. The mixing is temporarily suspended if the temperature exceed 100°F.

Example 2

This example provides a preferred transparent conductive composition according to the invention. The transparent conductive composition was made from the following components:

Component	Approximate Mass %
Ebecryl 264	26.7
IBOA	28.3

Component	Approximate Mass %
Irgacure 1700	5.0
Ebecryl 3603	6.6
Modaflow	3.5
Minatec 40	33.4
Total	100.00

5

In this example the IBOA and Irgacure 1700 are mixed in a pan with a propeller blade mixer for 30 seconds at a speed of 500 to 1000 rpm. In the next step, the Ebecryl 264, the Ebecryl 3603, and Modaflow are introduced into the pan and mixed for 1 to 2 minutes at a speed of 2000 rpm. In the final step, the Minatec 40 is added and mixed at 2000 rpm for 1 to 2 minutes. The mixing is temporarily suspended if the temperature exceed 100°F.

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Method for Depositing a Transparent conductive Coating

In accordance with still another aspect of the invention, a method is provided for depositing an transparent conductive coating on a suitable substrate. The method comprises a first step of applying a transparent conductive fluid-phase composition ("transparent conductive composition") to the substrate.

15

The transparent conductive composition comprises a mixture of aliphatic acrylated oligomers, wherein the aliphatic acrylated oligomer is present in an amount of about 10% to 40% of the transparent conductive composition. The transparent conductive composition preferably comprises an acrylated epoxy oligomer in an amount of about 3% to 11%, an isobornyl acrylate monomer in an amount of about 10% to 40% of the transparent conductive composition, a photoinitiator in an amount of about 2% to 10% of the transparent conductive composition, a flow promoting agent in an amount of about 0.1% to 8% of the transparent conductive composition, and a conductive powder in an amount of about 20% to 50% of the transparent conductive composition. The preferred transparent conductive compositions according to this method are those described herein, for example, including the compositions described in examples 1 and 2.

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The transparent conductive composition may be applied to the substrate using a number of different techniques. The transparent conductive composition may be applied, for example, by direct brush application, or it may be sprayed onto the substrate surface. It also may be applied using a screen printing technique. In such screen printing technique, a "screen" as the term is used in the screen printing industry is used to regulate the flow of liquid composition onto the substrate surface. The transparent conductive composition typically would be applied to the screen as the latter contacts the substrate. The transparent conductive composition flows through the silk screen to the substrate, whereupon it adheres to the substrate at the desired film thickness. Screen printing techniques suitable for this purpose include known techniques, but wherein the process is adjusted in ways known to persons of ordinary skill in the art to accommodate the viscosity, flowability, and other properties of the liquid-phase composition, the substrate and its surface properties, etc. Flexographic techniques, for example, using pinch rollers to contact the transparent conductive composition with a rolling substrate, also may be used.

The method includes a second step of illuminating the transparent conductive fluid-phase composition on the substrate with an ultraviolet light to cause the transparent conductive fluid-phase composition to cure into the transparent conductive coating. This illumination may be carried out in any number of ways, provided the ultraviolet light or radiation impinges upon the transparent conductive composition so that the transparent conductive composition is caused to polymerize to form the coating, layer, film, etc., and thereby cures.

Curing preferably takes place by free radical polymerization, which is initiated by an ultraviolet radiation source. The photoinitiator preferably comprises a photoinitiator, as described above.

Various ultraviolet light sources may be used, depending on the application. Preferred ultraviolet radiation sources for a number of applications include known ultraviolet lighting equipment with energy intensity settings of, for example, 125 watts, 200 watts, and 300 watts per square inch.

While embodiments of the invention have been illustrated and described, it is not intended that these embodiments illustrate and describe all possible forms of the invention. Rather, the words used in the specification are words of description rather than limitation, and it is understood that various changes
5 may be made without departing from the spirit and scope of the invention.

WHAT IS CLAIMED IS:

1. A photocurable transparent conductive composition comprising:
- 5 a photocurable organic mixture;
a conductive powder; and
a photoinitiator.
2. The photocurable transparent conductive composition of claim 1 wherein the photocurable organic mixture comprises:
- 10 at least one aliphatic acrylated oligomer;
an acrylated epoxy oligomer; and
an isobornyl acrylate monomer.
3. The photocurable transparent conductive composition of claim 2 further comprising a flow promoting agent.
4. The photocurable transparent conductive composition of claim 15 3 wherein:
- the acrylated aliphatic oligomer mixture is about 10% to 40% of the weight of the transparent conductive composition;
- the acrylated epoxy oligomer is about 3% to 11% of the weight of the transparent conductive composition;
- 20 the isobornyl acrylate monomer is about 10% to 40% of the weight of the transparent conductive composition;
- the photoinitiator is about 2% to 10% of the weight of the metallic composition;
- the flow promoting agent is present in an amount of about 0.1% to 25 8% of the weight of the transparent conductive composition; and
- the conductive powder is present in an amount of about 20% to 50% of the weight of the transparent conductive composition.

5. The photocurable transparent conductive composition of claim 3 wherein:

the acrylated aliphatic oligomer mixture is about 20% to 30% of the weight of the transparent conductive composition;

5 the acrylated epoxy oligomer is about 5% to 9% of the weight of the transparent conductive composition;

the isobornyl acrylate monomer is about 20% to 35% of the weight of the transparent conductive composition;

10 the photoinitiator is about 4% to 6% of the weight of the metallic composition;

the flow promoting agent is present in an amount of about 3% to 5% of the weight of the transparent conductive composition; and

the conductive powder is present in an amount of about 30% to 40% of the weight of the transparent conductive composition.

15 6. The photocurable transparent conductive composition of claim 3 wherein:

the acrylated aliphatic oligomer mixture is about 27% of the weight of the transparent conductive composition;

20 the acrylated epoxy oligomer is about 7% of the weight of the transparent conductive composition;

the isobornyl acrylate monomer is about 28% of the weight of the transparent conductive composition;

the photoinitiator is about 5% of the weight of the metallic composition;

25 the flow promoting agent is present in an amount of about 3.5% of the weight of the transparent conductive composition; and

the conductive powder is present in an amount of about 33% of the weight of the transparent conductive composition.

30 7. A method for coating a substrate with a photocurable transparent conductive composition, the method comprising:

applying the transparent conductive composition to the substrate, wherein the transparent conductive composition includes:

the acrylated aliphatic oligomer mixture is about 10% to 40% of the weight of the transparent conductive composition;

5 the acrylated epoxy oligomer is about 3% to 11% of the weight of the transparent conductive composition;

the isobornyl acrylate monomer is about 10% to 40% of the weight of the transparent conductive composition;

10 the photoinitiator is about 2% to 10% of the weight of the metallic composition;

the flow promoting agent is present in an amount of about 0.1% to 8% of the weight of the transparent conductive composition; and

the conductive powder is present in an amount of about 20% to 50% of the weight of the transparent conductive composition and

15 illuminating the transparent conductive composition with an UV light sufficient to cause the transparent conductive composition to be incorporated into the transparent conductive coating by the time the composition is cured.

8. The method of claim 7, wherein UV light used in illuminating impinges upon the transparent conductive composition so that the transparent
20 conductive composition is caused to form a coating as it cures.

9. The method of claim 7, wherein the method of applying the transparent conductive composition is spraying.

10. The method of claim 7, wherein the method of applying the transparent conductive composition is screen printing.

25 11. The method of claim 7, wherein the method of applying the transparent conductive composition is dipping the substrate into the composition sufficiently to cause the composition to uniformly coat the substrate.

12. The method of claim 7, wherein the method of applying the transparent conductive composition is brushing.

13. The method of claim 7, wherein the method of applying the transparent conductive composition is selectively depositing to the substrate at
5 predetermined locations.

14. A method for coating a substrate with a photocurable transparent conductive composition, the method comprising:

applying the transparent conductive composition to the substrate, wherein the transparent conductive composition includes:

10 the acrylated aliphatic oligomer mixture is about 20% to 30% of the weight of the transparent conductive composition;

the acrylated epoxy oligomer is about 5% to 9% of the weight of the transparent conductive composition;

15 the isobornyl acrylate monomer is about 20% to 35% of the weight of the transparent conductive composition;

the photoinitiator is about 4% to 6% of the weight of the metallic composition;

the flow promoting agent is present in an amount of about 3% to 5% of the weight of the transparent conductive composition; and

20 the conductive powder is present in an amount of about 30% to 40% of the weight of the transparent conductive composition; and

illuminating the transparent conductive composition with an UV light sufficient to cause the transparent conductive composition to be incorporated into the transparent conductive coating by the time the composition is cured.

25 15 The method of claim 14, wherein:

the acrylated aliphatic oligomer mixture is about 27% of the weight of the transparent conductive composition;

the acrylated epoxy oligomer is about 7% of the weight of the transparent conductive composition;

the isobornyl acrylate monomer is about 28% of the weight of the transparent conductive composition;

the photoinitiator is about 5% of the weight of the metallic composition;

5 the flow promoting agent is present in an amount of about 3.5% of the weight of the transparent conductive composition; and

the conductive powder is present in an amount of about 33% of the weight of the transparent conductive composition.

16. The transparent conductive composition of claim 2 wherein the
10 isobornyl acrylate monomer is selected from the group consisting of isobornyl acrylate, isobornyl methacrylate, and mixtures thereof.

17. The transparent conductive composition of claim 2 wherein the photoinitiator is selected from the group consisting of:

1-hydroxycyclohexyl phenyl ketone;
15 2-methyl-1-[4-(methylthio)phenyl]-2-morpholino propan-1-;
the combination of 50% 1-hydroxy cyclohexyl phenyl ketone and 50% benzophenone;

2,2-dimethoxy-1,2-diphenylethan-1-one;
the combination of 25% bis(2,6-dimethoxybenzoyl-2,4-, 4-trimethyl
20 pentyl phosphine oxide and 75% 2-hydroxy-2-methyl-1-phenyl-propan-1-one;

2-hydroxy-2-methyl-1-phenyl-1-propane;
the combination of 50% 2,4,6-trimethylbenzoyldiphenyl-phosphine
oxide and 50% 2-hydroxy 2-methyl-1-phenyl-propan-1-one;

25 mixed triaryl sulfonium hexafluoroantimonate salts, mixed triaryl sulfonium hexafluorophosphate salts; and
mixtures thereof.

18. The transparent conductive composition of claim 2 wherein the acrylated epoxy oligomer is selected from the group consisting of:

novolac epoxy acrylate diluted 20% by weight with tripropylene
30 glycol diacrylate;

difunctional bisphenol based epoxy acrylate; and mixtures thereof.

19. A method of making a photocurable transparent conductive composition comprising:

- 5 mixing an isobornyl acrylate monomer and a photoinitiator in a pan; introducing a flow promoting agent and an acrylated epoxy oligomer into the pan;
- mixing the flow promoting agent and the acrylated epoxy oligomer in the pan;
- 10 introducing the conductive powder into the pan; and mixing the conductive powder in the pan.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 01/00976

A. CLASSIFICATION OF SUBJECT MATTER
 IPC 7 C09D4/06 C08F290/06 C08F290/14 C09D4/00 C08F220/18
 C09D5/24

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED
 Minimum documentation searched (classification system followed by classification symbols)
 IPC 7 C09D C08F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
 EPO-Internal, WPI Data, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X,P	WO 00 62586 A (ALLIED PHOTOCHEMICAL INC) 19 October 2000 (2000-10-19) whole document	1-19
X	US 5 180 757 A (LUCEY MICHAEL) 19 January 1993 (1993-01-19) column 5, line 51 -column 8, line 39 column 12, line 58 - line 65 column 17, line 46 -column 18, line 38 column 20, line 37 -column 21, line 18 examples	1
Y	idem. claims 1-4,12,13,37,39,40,45-47,51,54,55	2,3
A	idem.	4,7,14, 19
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Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

° Special categories of cited documents :

<p>*A* document defining the general state of the art which is not considered to be of particular relevance</p> <p>*E* earlier document but published on or after the international filing date</p> <p>*L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>*O* document referring to an oral disclosure, use, exhibition or other means</p> <p>*P* document published prior to the international filing date but later than the priority date claimed</p>	<p>*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>*&* document member of the same patent family</p>
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Date of the actual completion of the international search 24 April 2001	Date of mailing of the international search report 10/05/2001
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Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer <p style="text-align: center;">Bettels, B</p>
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INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 01/00976

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 959 178 A (FRENTZEL RICHARD L ET AL) 25 September 1990 (1990-09-25) column 3, line 32 - line 36 column 3, line 54 - line 63 column 4, line 18 - line 47 examples: comparisons 5-7,9,11 claims 1,5,6,11-15	1
Y	idem.	2,3
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