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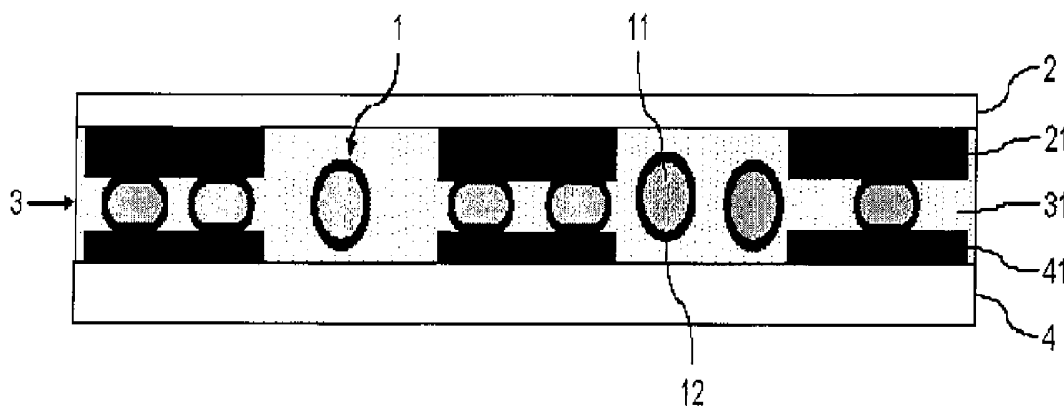
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(54) Title: POLYMER PARTICLES, CONDUCTIVE PARTICLES, AND AN ANISOTROPIC CONDUCTIVE PACKAGING MATERIALS CONTAINING THE SAME



(57) Abstract: Disclosed herein are anisotropic conductive particles contained in anisotropic conductive adhesive films which are used in the mounting field of circuit boards. The conductive particles have a uniform shape, a narrow particle diameter distribution, and appropriate compressive de-formability and recoverability from deformation. In addition, the conductive particles exhibit enhanced conducting properties without being ruptured when interposed and compressed between connection substrates, thereby achieving a sufficient contact area between the particles and the connection substrates. Further disclosed are polymer-based particles used in the conductive particles.

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Description

POLYMER PARTICLES, CONDUCTIVE PARTICLES, AND AN ANISOTROPIC CONDUCTIVE PACKAGING MATERIALS CONTAINING THE SAME

Technical Field

- [1] The present invention relates to conductive particles for use in conductive adhesives, anisotropic conductive adhesive films, conductive connection structures and the like for mounting circuit boards, and polymer particles used in the conductive particles. More specifically, the present invention relates to polymer particles having a 10% K value (i.e. K value when deformation in particle diameter is 10%) of 250 to 700 kgf/mm², a compression recovery factor of 30% or less and a compressive rupture deformation of 30% or higher wherein the conductive particles have K values at 20% and 30% compressive deformation not higher than 70% of the 10% K value, and conductive particles using the polymer particles.

[2]

Background Art

- [3] In general, anisotropic conductive connection is required to electrically connect connection electrodes of an IC circuit board to terminals of a substrate mounted on a circuit board, such as a liquid crystal display (LCD) panel. As such anisotropic conductive packaging materials, there are widely used film-type adhesives in which conductive particles, such as metal-coated resin particles or metal particles, are dispersed in an insulating resin, e.g., epoxy, urethane, or acrylic resin.
- [4] Conductive particles are interposed between electrodes and terminals by disposing an anisotropic conductive packaging material containing the conductive particles between the electrodes and the terminals, and pressing under heating to adhere the packaging material therebetween. At this time, electrical connection occurs in a pressing direction, and an insulation state is maintained in a direction perpendicular to the pressing direction due to the presence of insulating components contained in an insulating adhesive.
- [5] In LCD packaging where anisotropic conductive connection is required, recent advances in LCD technologies have brought about compactness of connection pitches, minuteness of IC bumps and increased number of leads printed on substrates. Further, there has been a continued need for improved electrical connection reliability. In order to satisfy such technical needs, conductive particles contained in anisotropic conductive films are largely required to have a uniform and small particle diameter. Further, conductive particles are critically required to have enhanced conducting

properties without being ruptured, together with appropriate compressive deformability and recoverability from deformation, in terms of increased contact area with connection substrates when the conductive particles are interposed and compressed between the connection substrates. Metal particles, such as nickel, gold and silver particles, and metal-coated base particles can be used as the conductive particles. However, since metal particles have a non-uniform shape and a much higher specific gravity than an adhesive resin, they have the problem of poor dispersibility in the adhesive resin.

- [6] For these reasons, in the mounting field where superior connection of micro-electrodes and improved connection reliability are required, there are widely used conductive particles with a uniform shape, a relatively narrow particle diameter distribution and enhanced conducting properties in which a plated layer is formed on base polymer particles.
- [7] Extensive research has hitherto been made on conductive particles in which polymer particles are plated, and particularly on the characteristics of the particles after compressive deformation in terms of improved contact with electrodes and connection reliability.
- [8] For example, Japanese Patent Laid-open No. S63-107188 discloses the use of high-strength highly elastic conductive particles with a compressive strength of 500 kg/cm^2 and a high compressive elastic modulus of $80 \times 10^3 \text{ kg/cm}^2$ or more. Further, PCT Publication WO 92/06402 discloses a spacer for an LCD and conductive particles using monodisperse resin particles as base particles. According to this publication, in order to readily control a gap between electrodes facing each other when the electrodes are connected to each other by compression using the conductive particles, the resin particles preferably have a compression hardness at 10% compressive deformation (10% K value) of 250 to 700 kgf/mm^2 . In addition, in order to increase the contact area between the conductive particles and the electrodes after compression, the resin particles preferably have a recovery factor after compressive deformation of 30 to 80%.
- [9] Further, Japanese Patent Laid-open No. H07-256231 discloses conductive particles having a K value at 10% compressive deformation of 700 to $1,000 \text{ kgf/mm}^2$ and a recovery factor after compressive deformation of 65% to 95% at 20°C in order to improve poor conductivity caused by changes in the temperature between electrodes, folding, mechanical impact, etc.
- [10] Moreover, Japanese Patent Laid-open No. H11-125953 and No. 2003-313304 disclose conductive particles having a K value at 10% compressive deformation of 250 kgf/mm^2 or lower and a recovery factor after compressive deformation of 30% or greater for better connection reliability.

- [11] These patent publications describe that as the recovery factor after compressive deformation of the conductive particles increases in a broad range of hardness, the conducting properties of the conductive particles, e.g., increased contact area with the electrodes, are enhanced.
- [12] However, when such conductive particles are dispersed in a cure-type binder resin and are pressed under heating to be connected to electrodes, the adhesive force of the curing binder resin and the contact of the particles with the electrodes are often insufficient. Recently, fast curing processes of anisotropic conductive adhesive films under low temperatures within a short period of time have been increasingly employed. These short-term connection conditions enable rapid curing of a binder resin, but cause insufficient contact of conductive particles with electrodes.
- [13] In addition, in anisotropic conductive materials, e.g., e.g., connection films for chip on glass (COG), having a greatly increased content (by several tens of %) of conductive particles, a large amount of highly elastic conductive particles interposed between electrodes partly or wholly deteriorate the adhesion of the connection films, resulting in poor connection reliability over a long period of time.
- [14] In addition to minuteness of electrode patterns and compactness of connection pitches, process for connecting relatively weak wiring patterns, e.g., ITO electrodes, under low pressure are required in order not to impair the wiring patterns. However, the connection condition of low pressures markedly deteriorates the adhesive force and connection reliability of highly elastic conductive particles.

[15]

Disclosure of Invention

Technical Problem

- [16] Therefore, the present invention has been made in view of the above problems, and it is an object of the present invention to provide conductive particles having a uniform shape, a narrow particle diameter distribution, and appropriate compressive deformability and recoverability from deformation, and to provide conductive particles having enhanced conducting properties without being ruptured when interposed and compressed between connection substrates, thereby achieving a sufficient contact area between the particles and the connection substrates.
- [17] It is another object of the present invention to provide polymer particles used in the conductive particles.
- [18] It is yet another object of the present invention to provide conductive particles having improved electrical connection reliability and anisotropic conductive packaging materials containing the conductive particles.
- [19]

Technical Solution

- [20] According to the present invention for achieving the objects, there are provided polymer particles having a 10% K value (i.e. K value when deformation in particle diameter is 10%) of 250 to 700 kgf/mm², a compression recovery factor of 30% or less, and a compressive rupture deformation of 30% or higher.
- [21] In a preferred embodiment of the present invention, the 10% K value of the polymer particles is in the range of 350 to 600 kgf/mm².
- [22] In a preferred embodiment of the present invention, the 20% and 30% K values of the polymer particles are not higher than 70% of the 10% K value.
- [23] In a preferred embodiment of the present invention, the polymer particles have an average particle diameter of 0.1~200 μ m, an aspect ratio lower than 1.5, and a coefficient of variation (CV) not higher than 20%.
- [24] In a preferred embodiment of the present invention, the polymer particles are made of a polymer resin of at least one crosslinking polymerizable monomer selected from the group consisting of: allyl compounds, e.g., divinylbenzene, 1,4-butanediol di(meth)acrylate, 1,6-hexanediol di(meth)acrylate, 1,9-nonanediol di(meth)acrylate, allyl (meth)acrylate, divinylsulfone, diallyl phthalate, diallylacrylamide, triallyl (iso)cyanurate, and triallyl trimellitate; (poly)alkylene glycol di(meth)acrylate, e.g., (poly)ethylene glycol di(meth)acrylate, and (poly)propylene glycol di(meth)acrylate; and pentaerythritol tetra(meth)acrylate, pentaerythritol tri(meth)acrylate, pentaerythritol di(meth)acrylate, trimethylolpropane tri(meth)acrylate, ditrimethylolpropane tetra(meth)acrylate, tetramethylolpropane tetra(meth)acrylate, dipentaerythritol hexa(meth)acrylate, dipentaerythritol penta(meth)acrylate, and glycerol tri(meth)acrylate.
- [25] In a preferred embodiment of the present invention, the polymer particles are prepared by copolymerizing at least one polymerizable unsaturated monomer selected from the group consisting of styrene-based monomers, e.g., styrene, ethyl vinyl benzene, α -methyl styrene, and m-chloromethyl styrene; acrylate-based monomers, e.g., methyl(meth)acrylate, ethyl(meth)acrylate, propyl(meth)acrylate, n-butyl(meth)acrylate, isobutyl(meth)acrylate, t-butyl(meth)acrylate, 2-ethylhexyl(meth)acrylate, n-octyl(meth)acrylate, lauryl(meth)acrylate, stearyl(meth)acrylate, ethyleneglycol (meth)acrylate, and glycidyl (meth)acrylate; chlorovinyl, acrylic acid esters, acrylonitrile, vinyl acetate, vinyl propionate, vinyl butyrate, vinyl ether, allyl butyl ether, butadiene, and isoprene.
- [26] In a preferred embodiment of the present invention, the polymer particles are prepared by seeded polymerization in which polymer seed particles have a molecular weight of from 1,000 to 30,000.

- [27] In the seeded polymerization, the total content of the polymerizable monomers is preferably 10~300 parts by weight, based on one part by weight of the polymer seed particles.
- [28] The present invention also provides conductive particles consisting of the polymer particles as base particles and at least one conductive metal layer formed on the surface of the polymer particles.
- [29] The conductive metal layer of the conductive particles is composed of at least one metal selected from the group consisting of nickel (Ni), gold (Au), silver (Ag), copper (Cu), platinum (Pt), palladium (Pd), cobalt (Co), tin (Sn), indium (In), and indium tin oxide (ITO). The conductive metal layer preferably has a thickness of 0.01 μ to 5 μ .
- [30] It is preferable that the conductive metal layer of the conductive particles is at least one double layer selected from the group consisting of nickel/gold, nickel/platinum, and nickel/silver.
- [31] The present invention also provides an anisotropic conductive packaging material containing the conductive particles.

[32]

Advantageous Effects

- [33] The conductive particles of the present invention use the polymer particles, as base particles of the conductive particles, having a 10% K value (i.e. K value when deformation in particle diameter is 10%) of 250 to 700 kgf/mm² and a compression recovery factor of 30% or less and a compressive rupture deformation of 30% or higher wherein the 20% and 30% K values are 70% or less of the 10% K value. Accordingly, the conductive particles of the present invention have appropriate compressive deformability and recoverability. In addition, when the conductive particles are interposed between electrodes of a circuit board and the like, they guarantee increased contact area while maintaining a uniform gap size, thus showing superior electrical connection and improved connection reliability.

[34]

Brief Description of the Drawings

- [35] The above and other objects, features and other advantages of the present invention will be more clearly understood from the following detailed description taken in conjunction with the accompanying drawing, in which:
- [36] Fig. 1 is a cross-sectional view of an electrical connection structure of an anisotropic conductive film using conductive particles of the present invention.

[37]

Best Mode for Carrying Out the Invention

- [38] Hereinafter, the present invention will be explained in more detail.

- [39] The present invention provides conductive particles composed of polymer particles and at least one metal layer coated on the surface of the polymer particles. The conductive particles of the present invention can be used in electrical connection structures of microelectrodes, anisotropic conductive adhesive films, and the like, in the mounting field of circuit boards.
- [40] In order to show enhanced conducting properties when the polymer particles used in the conductive particles of the present invention are used in the above-mentioned electrical packaging materials, the polymer particles must have K values, recovery factor after compressive deformation, and compressive rupture deformation in optimum ranges.
- [41] K value is measured using a micro-compression tester (MCT-W series, manufactured by Shimadzu Corporation Ltd., Japan). Specifically, the K value is measured by fixing a single particle between a smooth upper pressure indenter (diameter: 50 μ m) and a lower pressure plate, compressing the single particle at a compression speed of 0.2275 gf/sec and a maximum test load of 5 gf to obtain a load value and a compression displacement, and substituting the obtained values into the following Equation 1:
- [42] Equation 1
- [43]
- $$K = \left(\frac{3}{\sqrt{2}} \right) \cdot F \cdot S^{-\frac{3}{2}} \cdot R^{-\frac{1}{2}}$$
- [44] wherein F is a load value (kg) at x% compressive deformation, S is a compression displacement (mm) at x% compressive deformation, and R is a radius of the particles (mm).
- [45] As described above, it is preferred that the polymer particles have a K value at 10% compressive deformation of 250 to 700 kgf/mm². The use of the polymer particles in the K value range defined above enables the connection between facing electrodes in a constant gap size when interposed between the electrodes, without any damage to the electrodes. It is more preferable that the 10% K value of the conductive particles is in the range of 350 to 600 kgf/mm². When the 10% K value exceeds 700 kgf/mm², the conductive particles interposed between the electrodes are hardly deformed and hence the contact area between the electrode surface and the conductive particles is not sufficiently increased, making it difficult to lower the connection resistance. Further, the polymer particles within the above range are so hard that they may damage the electrode surface when the applied compressive force is increased for the purpose of lowering the connection resistance. On the other hand, when the 10% K value is lower than 250 kgf/mm², excessive deformation is likely to take place by the applied pressure

and thus the conductive layer may be peeled off from the particles, or excessive flatness occurs and thus the distance between the upper and lower electrodes is insufficiently ensured, which may cause problems of poor adhesive force and connection reliability.

- [46] In general, the 10% K value universally and quantitatively represents the hardness of the particles, but the compressive deformability of the particles cannot be exactly evaluated by the 10% K value. Accordingly, K values at both 20% and 30% compressive deformation should simultaneously be taken into consideration.
- [47] Fig. 1 is a cross-sectional view of an electrical connection structure in which the conductive particles 1 of the present invention are dispersed in an anisotropic conductive film 3 and are interposed between respective electrodes of a circuit board 2 and a glass substrate 4. As shown in Fig. 1, in order to impart adhesion of the conductive particles 1 to the electrodes and to stably maximize the contact area between the conductive particles 1 and the electrodes after deformation while uniformly and sufficiently maintaining a constant gap between the conductive particles 1 and the electrodes, the particles 1 should be hard to some extent in the initial stage of compression and must be continuously deformed along the compression.
- [48] Therefore, it is preferred that 20% and 30% K values of the polymer particles 11 are 70% or less of the 10% K value, which is a representative value of initial hardness upon compression. It is more preferable that the 20% and 30% K values of the particles are maintained at 60% or less of the 10% K value in terms of sufficient compressive deformability.
- [49] The compression recovery factor is measured by obtaining the relationship between the applied loads and the compression displacements while compressing the particles to a peak load of 1.0 gf in a micro-compression tester and decompressing to an initial load of 0.1 gf. Specifically, the compression recovery factor of the particles is defined as the ratio $L2/L1$ (%) wherein $L1$ is a displacement to the peak load upon loading and $L2$ is a displacement from the peak load to the initial load upon unloading. The loading and unloading is carried out at a compression speed of 0.1517 gf/sec.
- [50] In the present invention, the compression recovery factor of the polymer particles 11 is preferably limited to 30% or less and preferably 5%~30% in terms of stabilized adhesion, maximized contact area with the electrodes, and improved connection reliability. If the recovery factor of the polymer particles 11 is close to zero, the difference between the elasticity of the particles and an adhesive resin according to changes in temperature is excessively large, thus risking the danger of poor conducting properties. This limitation to the recoverability from deformation of the particles, although not general, is very significant when the compressive deformability of the particles is taken into consideration along with the recoverability.

- [51] The compressive rupture deformation is measured using the same micro-compression tester as in the measurement of K value. Specifically, the compressive rupture deformation of the particles is defined as the ratio L_d/D (%) wherein L_d is a displacement at a time point when the particles are ruptured and D is a diameter of the particles. Since the conductive particles 1 of the present invention should not be easily ruptured by compression in order to achieve sufficient deformation and low connection resistance, the compressive rupture deformation of the polymer particles 11 is limited to 30% or greater. More preferably, the compressive rupture deformation of the polymer particles 11 is 40% or greater.
- [52] The polymer particles 11 of the present invention preferably have a particle diameter of 0.1 to 200 μ m, and more preferably 1 to 20 μ m. When the particles have a particle diameter smaller than 0.1 μ m, they tend to aggregate. Meanwhile, when the particles have a particle diameter exceeding 200 μ m, they are not of interest in their application to materials for recent micromounting techniques.
- [53] It is preferable that the polymer particles 11 have an aspect ratio of less than 1.5 and a coefficient of variation (CV) in particle diameter of 20% or less in order not to degrade the connection reliability. The aspect ratio used herein refers to a ratio of the longest axis to the shortest axis in the diameter of a single particle, and the CV value refers to a percentage (%) obtained by dividing the standard deviation of the particle diameter by the average particle diameter. It is more preferred that the particles have an aspect ratio of less than 1.3 and a CV value not greater than 10%.
- [54] As mentioned earlier, the conductive particles of the present invention have a structure wherein a metal layer 12 is coated on the surface of the base polymer particles 11. Accordingly, the compressive deformability and compression recoverability of the conductive particles 1 are largely dependent on the base polymer particles 11 of the conductive particles.
- [55] The polymer resin-based particles 11 are preferably made of at least one material selected from the group consisting of polyethylene, polypropylene, polyvinyl chloride, polystyrene, polytetrafluoroethylene, polyethylene terephthalate, polybutylene terephthalate, polyamide, polyimide, polysulfone, polyphenylene oxide, polyacetal, urethane resin, unsaturated polyester resin, (meth)acrylate resin, styrene-based resin, butadiene resin, epoxy resin, phenol resin, and melamine resin.
- [56] Of these, styrene-based resin and (meth)acrylate resin are preferred, and polymer resins containing at least one crosslinking polymerizable monomer are more preferred.
- [57] The crosslinking polymerizable monomer is preferably selected from the group consisting of: allyl compounds, e.g., divinylbenzene, 1,4-butanediol di(meth)acrylate, 1,6-hexanediol di(meth)acrylate, 1,9-nonanediol di(meth)acrylate, allyl (meth)acrylate, divinyldisulfone, diallyl phthalate, diallylacrylamide, triallyl (iso)cyanurate, and triallyl

trimellitate; (poly)alkylene glycol di(meth)acrylate, e.g., (poly)ethylene glycol di(meth)acrylate, and (poly)propylene glycol di(meth)acrylate; and pentaerythritol tetra(meth)acrylate, pentaerythritol tri(meth)acrylate, pentaerythritol di(meth)acrylate, trimethylolpropane tri(meth)acrylate, ditrimethylolpropane tetra(meth)acrylate, tetramethylolpropane tetra(meth)acrylate, dipentaerythritol hexa(meth)acrylate, dipentaerythritol penta(meth)acrylate, and glycerol tri(meth)acrylate.

[58] As monomers used in combination with the crosslinking polymerizable monomers, there may be exemplified polymerizable unsaturated monomers that can be copolymerized with the crosslinking polymerizable monomers. Specific examples of polymerizable unsaturated monomers include styrene-based monomers, e.g., ethyl vinyl benzene, styrene, α -methyl styrene, and m-chloromethyl styrene; acrylate-based monomers, e.g., methyl(meth)acrylate, ethyl(meth)acrylate, propyl(meth)acrylate, n-butyl(meth)acrylate, isobutyl(meth)acrylate, t-butyl(meth)acrylate, 2-ethylhexyl(meth)acrylate, n-octyl(meth)acrylate, lauryl(meth)acrylate, stearyl(meth)acrylate, ethyleneglycol (meth)acrylate, and glycidyl (meth)acrylate; chlorovinyl, acrylic acid esters, acrylonitrile, vinyl acetate, vinyl propionate, vinyl butyrate, vinyl ether, allyl butyl ether, butadiene, and isoprene. These polymerizable unsaturated monomers may be used alone or in combination.

[59] The polymer particles can be prepared by suspension polymerization, dispersion polymerization, precipitation polymerization, seeded polymerization, and soap-free emulsion polymerization. In the present invention, seeded polymerization is used to prepare polymer particles having a uniform particle diameter distribution.

[60] The seeded polymerization is carried out by the following specific procedure. First, polymer seed particles having a uniform particle diameter are dispersed in an aqueous solution. To the dispersion was added an aqueous emulsion of a (crosslinking) polymerizable unsaturated monomer in which an oil-soluble initiator is dissolved. By this addition, the monomer is absorbed inside the seed particles. Thereafter, the (crosslinking) polymerizable unsaturated monomer containing the seed particles is polymerized to prepare polymer particles. Since the molecular weight of the polymer seed particles greatly affects the phase separation and mechanical properties of the polymer particles prepared by the seeded polymerization, it is preferably limited to the range of 1,000~30,000 and more preferably 5,000~20,000. In addition, the (crosslinking) polymerizable unsaturated monomer is preferably absorbed in an amount of 10~300 parts by weight, based on one part by weight of the polymer seed particles.

[61] The initiator used to prepare the polymer particles is a common oil-soluble radical initiator, and specific examples thereof include peroxide-based compounds, e.g., benzoyl peroxide, lauryl peroxide, o-chlorobenzoyl peroxide, o-methoxybenzoyl

peroxide, t-butylperoxy-2-ethylhexanoate, t-butyl peroxyisobutyrate, 1,1,3,3-tetramethylbutylperoxy-2-ethylhexanoate, dioctanoyl peroxide and didecanoyl peroxide, and azo compounds, e.g., 2,2'-azobisisobutyronitrile, 2,2'-azobis(2-methylbutyronitrile) and 2,2'-azobis(2,4-dimethylvaleronitrile). It is preferred that the initiator is used in an amount of 0.1~20% by weight, based on the monomers.

- [62] In the course of the polymerization of the polymer particles, if necessary, a surfactant and a dispersion stabilizer can be used to secure the stability of latex. Examples of suitable surfactants include common surfactants, such as anionic, cationic, and non-ionic surfactants.
- [63] The dispersion stabilizer is a material that can be dissolved or dispersed in polymerization media, and specific examples thereof include water-soluble polymers, e.g., gelatin, starch, methylcellulose, ethylcellulose, hydroxyethylcellulose, carboxymethylcellulose, polyvinylpyrrolidone, polyvinyl alkyl ether, polyvinyl alcohol, polyacrylic acid, polyacrylamide, polyethylene oxide and sodium polymethacrylate, barium sulfate, calcium sulfate, calcium carbonate, calcium phosphate, aluminum sulfate, talc, clay, diatomaceous earth, and metal oxide powders. These materials may be used alone or in combination. The dispersion stabilizer is used in an amount to inhibit the settlement of the polymer particles formed during polymerization due to gravity and aggregation of the particles. Preferably, the dispersion stabilizer is used in an amount of about 0.01 to about 15 parts by weight, based on 100 parts by weight of all the reactants.
- [64] The conductive particles 1 are prepared by forming the metal layer 12 on the surface of the polymer particles 11. Examples of metals that can be used to form the metal layer 12 include, but are not especially limited to, nickel (Ni), gold (Au), silver (Ag), copper (Cu), platinum (Pt), palladium (Pd), cobalt (Co), tin (Sn), indium (In), indium tin oxide (ITO), and multilayer composite metals containing one or more of these metals as main components. Particularly preferred is a double metal layer of nickel/gold in which the surface of the polymer particles 11 is sequentially plated with nickel and gold. Another conductive metal, such as platinum (Pt) or silver (Ag), can be used instead of gold.
- [65] Examples of methods for forming the metal layer on the base particles include, but are not particularly limited to, electroless plating, coating using metal powders, vacuum deposition, ion plating, and ion sputtering.
- [66] The preparation of the conductive particles by electroless plating is carried out through the following three steps: first step (pretreatment of the surface of the base particles) defatting, etching, sensitizing, catalyzing, treating with a reducing agent, etc.; second step - electroless nickel (Ni) plating and washing; and third step - gold (Au)

substitution plating.

[67] The electroless plating is carried out in accordance with the following specific procedure. First, the polymer particles are dipped in a surfactant solution having a proper concentration to wash and defat the particle surface. Thereafter, etching is performed using a mixed solution of chromic acid and sulfuric acid to form anchors on the surface of the base particles. The surface-treated base particles are dipped in a solution of tin chloride and palladium chloride to catalyze and activate the particle surface. As a result, fine nuclei of the palladium catalyst are formed on the surface of the base particles. Subsequently, a reduction reaction is carried out using sodium hypophosphite, sodium borohydride, dimethyl amine borane, hydrazine, and the like, to form uniform palladium nuclei on the particles. The resulting base particles are dispersed in an electroless nickel plating solution, after which the nickel salts are reduced using sodium hypophosphite to form a nickel-plated layer on the base particles. The nickel-plated base particles are added to an electroless gold plating solution having a certain concentration to induce a gold substitution plating reaction, thereby forming a gold-deposited layer on the outermost layer.

[68] The conductive metal layer 12 of the conductive particles 1 according to the present invention preferably has a thickness of 0.01 to 5 μ m. When the thickness of the metal layer is less than 0.01 μ m, it is difficult to attain the desired conductivity. On the other hand, when the thickness of the metal layer exceeds 5 μ m, the deformability, elasticity and recoverability of the particles are not satisfactory due to the thick metal layer, and the particles tend to aggregate when used in electrode packaging materials, making it difficult to show enhanced conducting properties.

[69] The present invention will now be described in more detail with reference to the following examples. However, these examples are given for the purpose of illustration and are not to be construed as limiting the scope of the invention.

[70]

Mode for the Invention

[71] <Example 1>

[72] (1) Preparation of seed particles

[73] 30 parts by weight of a styrene monomer, 6 parts by weight of 2,2'-azobis(2,4-dimethylvaleronitrile) as an initiator, 18.7 parts by weight of polyvinylpyrrolidone (molecular weight: 40,000), and 190 parts by weight of methanol and 15 parts by weight of ultrapure water as reaction media were mixed together, quantified, and added to a reactor. Thereafter, the reaction mixture was subjected to polymerization under a nitrogen atmosphere at 70°C for 24 hours to prepare polystyrene seed particles. The seed particles were washed with ultrapure water and

methanol several times, and dried in a vacuum freeze dryer to obtain a powder. The seed particles were measured to have an average particle diameter of 1.15 μ , a CV value of 4.1%, and a molecular weight of 15,500.

[74]

[75] (2) Preparation and evaluation of polymer resin-based particles

[76] 2 parts by weight of the seed particles were homogeneously dispersed in 450 parts by weight of an aqueous sodium lauryl sulfate (SLS) solution (0.2 wt%). Separately, a monomer mixture consisting of 90 parts by weight of styrene and 10 parts by weight of divinylbenzene, wherein 1 parts by weight of benzoyl peroxide as an initiator was dissolved, was added to 300 parts by weight of an aqueous SLS solution (0.2 wt%). The resulting mixture was emulsified for 10 minutes using a homogenizer. The monomer emulsion was added to the seed dispersion to swell the monomers inside the seed particles at room temperature. After completion of the swelling, 500 parts by weight of an aqueous polyvinylalcohol solution (5 wt%) having a saponification degree of about 88% was added thereto. After the temperature of the reactor was raised to 80°C, polymerization was performed. The styrene-divinylbenzene copolymer resin particles thus prepared were washed with ultrapure water and ethanol several times, and dried in vacuo at room temperature. The K value and compression recovery factor of the conductive particles were measured, and the obtained results are shown in Table 1.

[77]

[78] (3) Preparation and evaluation of conductive particles

[79] The polymer particles were etched in an aqueous sodium hydroxide solution, dipped in a palladium chloride solution, and reduced to form fine nuclei of the palladium on the surface of the base particles. Thereafter, electroless nickel plating and gold substitution plating were sequentially performed to obtain conductive particles in which a nickel/gold metal layer was formed on the base particles.

[80]

[81] (4) Manufacture and evaluation of anisotropic conductive connection structure

[82] 15 parts by weight of a bisphenol A epoxy resin having an epoxy equivalent of 6,000 and 7 parts by weight 2-methylimidazole as a curing agent were dissolved in a mixed solvent of toluene and methyl ethyl ketone, after which 10% by weight of the conductive particles and a silane-based coupling agent were dispersed in the solution. The resulting dispersion was coated on a PET release film, and dried to produce a 25 μ thick anisotropic conductive adhesive film.

[83]

An anisotropic conductive film was produced from the anisotropic conductive adhesive film so as to have the following dimensions. Height of bump electrodes: 40 μ , IC chip size: 6 mm x 6 mm, thickness of BT resin substrate: 0.7 mm, thickness of

wiring patterns formed on the substrate by copper and gold plating: 8 μ , pitch: 100 μ . The anisotropic conductive film was interposed between the IC chip and the substrate, and then pressurized to 3 MPa while heating at 180°C for 10 seconds to manufacture an electrical connection structure.

[84] To measure the electrical resistance between upper and lower electrodes of the connection sample, electrical resistance values between 20 adjacent upper and lower electrodes was measured, and averaged. Results are shown as connection resistance values in Table 1. Further, after the connection sample was aged at 85°C and 85% RH for 1,000 hours, connection reliability was evaluated according to the increment in resistance as follows:

[85] $\odot$: $\leq 0.1\Omega$, $\triangle$: $0.1\Omega\sim 0.3\Omega$, $\times$: $> 0.3\Omega$

[86]

[87] <Example 2>

[88] Polymer particles and conductive particles were prepared in the same manner as in Example 1, except that a monomer mixture consisting of 80 parts by weight of styrene and 20 parts by weight of 1,4-butanediol diacrylate was used instead of the monomer mixture consisting of 90 parts by weight of styrene and 10 parts by weight of divinylbenzene. A connection structure was manufactured using the conductive particles. The properties of the base polymer particles and the connection structure were evaluated according to the same procedure as in Example 1. The results are shown in Table 1.

[89]

[90] <Example 3>

[91] Polymer particles and conductive particles were prepared in the same manner as in Example 1, except that a monomer mixture consisting of 80 parts by weight of styrene and 20 parts by weight of 1,6-hexanediol dimethacrylate was used instead of the monomer mixture consisting of 90 parts by weight of styrene and 10 parts by weight of divinylbenzene. A connection structure was manufactured using the conductive particles. The properties of the base polymer particles and the connection structure were evaluated according to the same procedure as in Example 1. The results are shown in Table 1.

[92]

[93] <Example 4>

[94] Polymer particles and conductive particles were prepared in the same manner as in Example 1, except that a monomer mixture consisting of 80 parts by weight of styrene and 20 parts by weight of ethyleneglycol dimethacrylate was used instead of the monomer mixture consisting of 90 parts by weight of styrene and 10 parts by weight of divinylbenzene. A connection structure was manufactured using the conductive

particles. The properties of the base polymer particles and the connection structure were evaluated according to the same procedure as in Example 1. The results are shown in Table 1.

[95]

[96] <Comparative Example 1>

[97] Polymer particles and conductive particles were prepared in the same manner as in Example 1, except that 100 parts by weight of divinylbenzene was used instead of the monomer mixture consisting of 90 parts by weight of styrene and 10 parts by weight of divinylbenzene. A connection structure was manufactured using the conductive particles. The properties of the base polymer particles and the connection structure were evaluated according to the same procedure as in Example 1. The results are shown in Table 1.

[98]

[99] <Comparative Example 2>

[100] Polymer particles and conductive particles were prepared in the same manner as in Example 1, except that a monomer mixture consisting of 80 parts by weight of tetramethylolpropane tetraacrylate and 20 parts by weight of acrylonitrile was used instead of the monomer mixture consisting of 90 parts by weight of styrene and 10 parts by weight of divinylbenzene. A connection structure was manufactured using the conductive particles. The properties of the base polymer particles and the connection structure were evaluated according to the same procedure as in Example 1. The results are shown in Table 1.

[101] Table 1

		Average particle diameter	CV value	K values			Compression recovery factor	Compressive rupture deformation	Anisotropic connection structure	
				10% K value	20% K value	30% K value			Connection resistance	Connection reliability
		μm	%	Kg f/mm ²			%	%	Ω	-
Example No.	1	4.5	3.9	381	214	180	11	64	0.7	◎
	2	4.4	3.8	494	271	302	24	53	0.6	◎
	3	4.4	4.0	426	224	190	19	51	0.7	◎
	4	4.6	3.8	445	247	219	17	48	0.8	◎
Comparative Example No.	1	4.4	3.8	650	453	436	52	55	1.3	X
	2	4.5	4.0	811	605	710	75	31	1.4	X

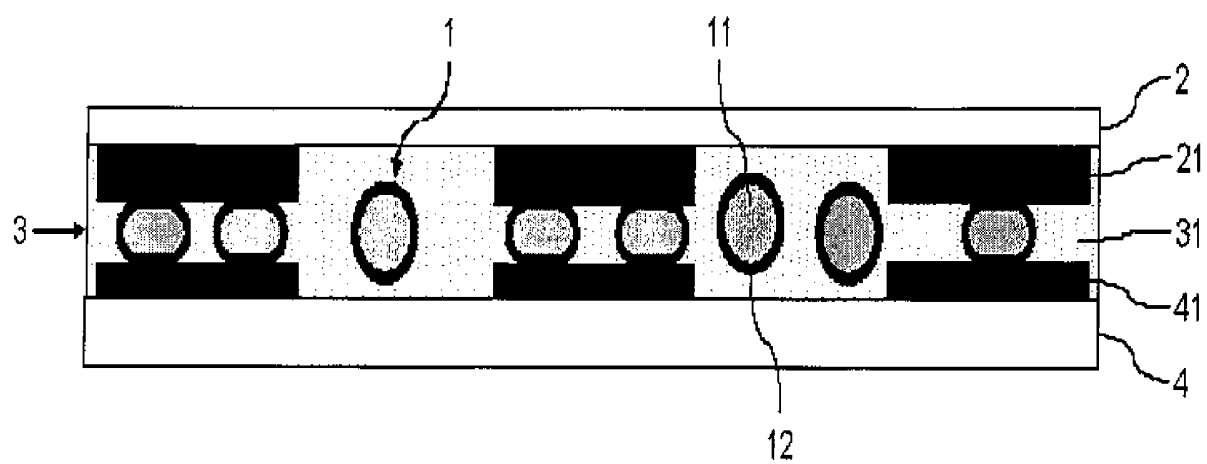
[102] As can be seen from the data shown in Table 1, the conductive particles having appropriate deformability and recoverability prepared in Examples 1~4 and the anisotropic conductive adhesive films produced using the conductive particles showed much lower connection resistance and better connection reliability than those having relatively low deformability and relatively high recoverability prepared in Comparative Examples 1 and 2 and the anisotropic conductive adhesive films produced using the conductive particles.

Claims

- [1] A polymer particle having a 10% K value (i.e. K value when deformation in particle diameter is 10%) of 250 to 700 kgf/mm², a compression recovery factor of 30% or less, and a compressive rupture deformation of 30% or higher.
- [2] The polymer particle according to claim 1, wherein the polymer particle has a 10% K value of 350 to 600 kgf/mm².
- [3] The polymer particle according to claim 1, wherein the polymer particle has K values at 20% and 30% compressive deformation not higher than 70% of the 10% K value.
- [4] The polymer particle according to claim 1, wherein the polymer particle has an average particle diameter of 0.1~200 μ .
- [5] The polymer particle according to claim 1, wherein the polymer particle has an aspect ratio lower than 1.5 and a coefficient of variation (CV) not higher than 20%.
- [6] The polymer particle according to claim 1, wherein the polymer particle is made of a polymer resin of at least one crosslinking polymerizable monomer selected from the group consisting of: allyl compounds, including divinylbenzene, 1,4-butanediol di(meth)acrylate, 1,6-hexanediol di(meth)acrylate, 1,9-nonanediol di(meth)acrylate, allyl (meth)acrylate, divinylsulfone, diallyl phthalate, diallyl-acrylamide, triallyl (iso)cyanurate, and triallyl trimellitate; (poly)alkylene glycol di(meth)acrylate, including (poly)ethylene glycol di(meth)acrylate, and (poly)propylene glycol di(meth)acrylate; and pentaerythritol tetra(meth)acrylate, pentaerythritol tri(meth)acrylate, pentaerythritol di(meth)acrylate, trimethylolpropane tri(meth)acrylate, ditrimethylolpropane tetra(meth)acrylate, tetramethylolpropane tetra(meth)acrylate, dipentaerythritol hexa(meth)acrylate, dipentaerythritol penta(meth)acrylate, and glycerol tri(meth)acrylate.
- [7] The polymer particle according to claim 1, wherein the polymer particle is prepared by copolymerizing at least one polymerizable unsaturated monomer selected from the group consisting of styrene-based monomers, including styrene, ethyl vinyl benzene, α -methyl styrene, and m-chloromethyl styrene; acrylate-based monomers, e.g., methyl(meth)acrylate, ethyl(meth)acrylate, propyl(meth)acrylate, n-butyl(meth)acrylate, isobutyl(meth)acrylate, t-butyl(meth)acrylate, 2-ethylhexyl(meth)acrylate, n-octyl(meth)acrylate, lauryl(meth)acrylate, stearyl(meth)acrylate, ethyleneglycol (meth)acrylate, and glycidyl (meth)acrylate; chlorovinyl, acrylic acid esters, acrylonitrile, vinyl acetate, vinyl propionate, vinyl butyrate, vinyl ether, allyl butyl ether, butadiene, and isoprene.

- [8] The polymer particle according to claim 1, wherein the polymer particle is prepared by seeded polymerization in which a polymer seed particle has a molecular weight of from 1,000 to 30,000.
- [9] The polymer particle according to claim 1, wherein the total content of the polymerizable monomer used in the seeded polymerization is 10~300 parts by weight, based on one part by weight of the polymer seed particle.
- [10] A conductive particle consisting of the polymer particle according to any one of claims 1 to 9 and at least one conductive metal layer formed on the surface of the polymer particle.
- [11] The conductive particle according to claim 10, wherein the conductive metal layer is composed of at least one metal selected from the group consisting of nickel (Ni), gold (Au), silver (Ag), copper (Cu), platinum (Pt), palladium (Pd), cobalt (Co), tin (Sn), indium (In), and indium tin oxide (ITO).
- [12] The conductive particle according to claim 10 or 11, wherein the conductive metal layer is at least one double layer selected from the group consisting of nickel/gold, nickel/platinum, and nickel/silver.
- [13] The conductive particle according to any one of claims 10 to 12, wherein the conductive metal layer has a thickness of 0.01 to 5 μ m.
- [14] An anisotropic conductive packaging material containing the conductive particle according to any one of claims 10 to 13.

[Fig. 1]



INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR2005/002005

A. CLASSIFICATION OF SUBJECT MATTER**IPC7 C08J 3/12**

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)

C08J 3/12, G02F 1/1339

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 practicable, search terms used)
eKIPASS, SCOPUS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 92-6402 A (Sekisui Fine Chemical Co., LTD.) 1992-12-19 See comparative example 1, 4, 13; Claims 1, 4	1-7, 10-14
A	JP 09-185069 A (Sekisui Fine Chemical Co., LTD.) 1997-07-15 See abstract; Claims 1-6	1-14
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☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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"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

"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

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"&" document member of the same patent family

Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

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