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(54) Title: METAL NANOPARTICLE PASTE, BONDING METHOD, BONDED ELEMENT AND ELECTRONIC SUBSTRATE

(57) Abstract: A metal nanoparticle paste includes: metal nanoparticles; a phosphate dispersant that has a hydrophilic portion; and a polar solvent, wherein the content of the metal nanoparticles is higher than or equal to 70 percent by weight and lower than 100 percent by weight.

METAL NANOPARTICLE PASTE, BONDING METHOD, BONDED ELEMENT AND ELECTRONIC SUBSTRATE

BACKGROUND OF THE INVENTION

1. Field of the Invention

[0001] The invention relates to a metal nanoparticle paste, a bonding method using the metal nanoparticle paste, a bonded element bonded by the metal nanoparticle paste, and an electronic substrate having a wire formed of the metal nanoparticle paste.

2. Description of Related Art

[0002] In an existing art, solder is used as a bonding material; however, it is difficult to use solder for a power device element, such as silicon carbide and gallium nitride having a high operating temperature. Therefore, currently, a metal fine particle paste having a high resistance is increasingly used as a bonding material. For example, Japanese Patent Application Publication No. 2009-279649 (JP 2009-279649 A) describes a bonding material that contains silver nanoparticles, any one of silver carbonate and silver oxide, and carboxylic acid. In addition, Japanese Patent Application Publication No. 2011-21255 (JP 2011-21255 A) describes a composite nanometal paste that contains composite metal nanoparticles each having an organic coating layer, metal nanofiller particles, and metal filler particles. Japanese Patent Application Publication No. 6-119808 (JP 6-119808 A) describes a metal fine powder, an organic binder, an organic solvent and a phosphorus oxide.

[0003] When metal fine particles, particularly, metal nanoparticles, are used in a paste, metal fine particles flocculate, and the viscosity of the paste excessively increases. As a result, there is inconvenience in handleability, so it has been reported that a dispersant is used to prevent flocculation of metal fine particles (for example, Japanese Patent Application Publication No. 2007-21475 (JP 2007-21475 A)).

[0004] As described above, when metal fine particles are used in a paste, the viscosity of the paste increases because of flocculation of metal fine particles. In this case, by increasing the amount of organic solvent, the viscosity may be decreased; while, on the other hand, bonding strength and electrical/thermal conductivity decrease. In order to prevent flocculation of metal fine particles, for example, a dispersant is used in JP 2007-21475 A; however, a large amount of organic solvent is also used, and the content of metal in the paste remains about 60% (example and Table 1).

[0005] Therefore, there is a need for a metal fine particle paste having excellent handleability, bonding strength and electrical/thermal conductivity, the metal fine particle paste having a low viscosity while containing metal fine particles at a high rate.

SUMMARY OF THE INVENTION

[0006] The inventors diligently studied and finally found that a low-viscosity metal nanoparticle paste that contains metal nanoparticles at a high rate may be manufactured using a phosphate dispersant.

[0007] A first aspect of the invention relates to (1) a metal nanoparticle paste that includes: metal nanoparticles; a phosphate dispersant that has a hydrophilic portion; and a polar solvent, wherein the content of the metal nanoparticles is higher than or equal to 70 percent by weight and lower than 100 percent by weight.

[0008] (2) In the metal nanoparticle paste described in the above (1), the content of the metal nanoparticles may be higher than or equal to 90 percent by weight and lower than 99 percent by weight.

[0009] (3) In the metal nanoparticle paste described in the above (2), the content of the metal nanoparticles may be higher than or equal to 95 percent by weight and lower than 99 percent by weight.

[0010] (4) In the metal nanoparticle paste according to any one of the above (1) to (3), the viscosity of the metal nanoparticle paste may be lower than or equal to 100 Pa•s.

[0011] (5) In the metal nanoparticle paste according to any one of claims (1) to (4), the hydrophilic portion may be polyethylene glycol.

[0012] (6) In the metal nanoparticle paste according to any one of the above (1) to (5), the polar solvent may be an alcohol solvent.

[0013] A second aspect of the invention relates to (7) a bonding method that includes applying the metal nanoparticle paste according to any one of the above (1) to (6) to a bonded member.

[0014] (8) In the bonding method according to the above (7), the bonded member may be bonded under no pressure.

[0015] A third aspect of the invention relates to (9) a bonded element that is obtained by the bonding method described in the above (7) or (8).

[0016] (10) In the bonded element according to the above (10), the shear

strength of the bonded element may be 80 to 110 MPa.

[0017] A fourth aspect of the invention relates to (11) an electronic substrate that has a wire formed from the metal nanoparticle paste according to any one of the above (1) to (6).

[0018] According to the aspects of the invention, it is possible to provide a low-viscosity metal nanoparticle paste that contains metal nanoparticles at a high rate.

BRIEF DESCRIPTION OF THE DRAWINGS

[0019] Features, advantages, and technical and industrial significance of exemplary embodiments of the invention will be described below with reference to the accompanying drawings, in which like numerals denote like elements, and wherein:

FIG. 1 shows the results of the viscosity of a silver nanoparticle paste according to an embodiment of the invention;

FIG. 2A shows a schematic view of a specimen used in a shear strength test, in which the left view is a side view of the specimen and the right view is a plan view of the specimen;

FIG. 2B shows a schematic view of the shear strength test;

FIG. 3 shows the results of the shear strength test;

FIG. 4 shows the viscosity of a metal nanoparticle paste and the bonding strength of a specimen in a comparative example;

FIG. 5A shows a TEM image at a bonded interface of a specimen; and

FIG. 5B shows the results of elemental analysis at bonded interfaces of specimens.

DETAILED DESCRIPTION OF EMBODIMENTS

1. Metal Nanoparticle Paste

[0020] A first embodiment of the invention relates to a metal nanoparticle paste that contains metal nanoparticles, a phosphate dispersant having a hydrophilic portion, and a polar solvent, wherein the content of the metal nanoparticles is higher than or equal to 70 percent by weight and lower than 100 percent by weight. The metal nanoparticle paste according to the first embodiment of the invention may contain metal nanoparticles at a high rate although it has a low viscosity, so the metal nanoparticle paste has excellent handleability, bonding strength and electrical/thermal conductivity.

[0021] The type of metal used as metal nanoparticles is not specifically

limited, and may be any precious metal or any base metal. The precious metal may be, for example, gold, silver, ruthenium, rhodium, palladium, iridium, platinum, or the like. The base metal may be, for example, copper, aluminum, iron, nickel, or the like. The number of types of metal used may be one or may be two or more in combination. As long as metal nanoparticles maintain electrical/thermal conductivity, the metal nanoparticles may be in form of metal oxide or metal salt. In the first embodiment of the invention, although not specifically limited, silver is desirably used in terms of electrical conductivity and thermal conductivity.

[0022] The particle diameter of the metal nanoparticles is not specifically limited as long as the particle diameter is a nano-level diameter. However, as the particle diameter reduces, the contact area between particles increases and the bonding strength and the electrical/thermal conductivity improves, so the particle diameter is desirably smaller. For example, metal nanoparticles having a particle diameter of 1 to 500 nm, desirably 5 to 300 nm, more desirably 10 to 200 nm, and particularly desirably 20 to 100 nm, are used. Metal nanoparticles having a specific particle diameter range may be used solely or metal nanoparticles having different particle diameter ranges may be used in combination.

[0023] The content of metal nanoparticles in the metal nanoparticle paste is higher than or equal to 70 percent by weight and lower than 100 percent by weight (for example, lower than 99 percent by weight), desirably higher than or equal to 80 percent by weight, more desirably higher than or equal to 85 percent by weight, further desirably higher than or equal to 90 percent by weight, particularly desirably higher than or equal to 93 percent by weight, and especially desirably higher than or equal to 95 percent by weight.

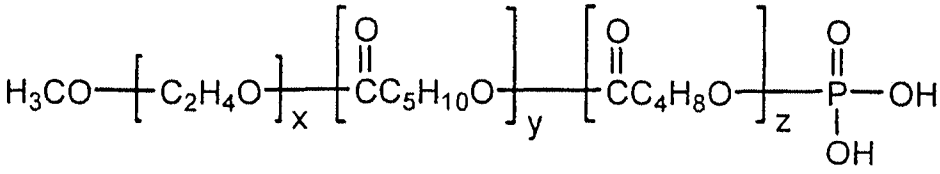
[0024] The phosphate dispersant has a phosphate group and a hydrophilic portion. Generally, metal nanoparticles each are coated with an organic protection film (for example, a fatty acid) in order to prevent flocculation. The organic protection film may be replaced with the phosphate group having strong coordination force toward a metal. Furthermore, the dispersibility of the metal nanoparticles to a polar solvent is improved by the hydrophilic portion. By so doing, it is possible to prevent flocculation of the metal nanoparticles.

[0025] The phosphate dispersant is not specifically limited as long as the phosphate dispersant has a phosphate group and a hydrophilic portion. For example,

the phosphate dispersant may be a phosphate ester dispersant, a polyoxyalkylene alkyl ether phosphate dispersant, a polyoxyalkylene alkyl phenyl ether phosphate dispersant, or the like. The phosphate group may be in form of salt. The hydrophilic portion may be, for example, polyalkylene glycol (polyethylene glycol, polytetraethylene glycol, polypropylene glycol, or the like), polyglycerol, or the like. Although not specifically limited, the phosphate dispersant desirably has polyethylene glycol as the hydrophilic portion.

[0026] In addition, the phosphate dispersant may have the following structure expressed by:

[Structural Formula 1]



In the formula, x is an integer of 6 to 20 (desirably, an integer of 6 to 14), y is an integer of 0 to 5 (desirably, an integer of 0 to 2), z is an integer of 0 to 5 (desirably, an integer of 0 to 2), and x + y + z is an integer of 6 to 30 (desirably, an integer of 6 to 18).

[0027] The content of the phosphate dispersant in the metal nanoparticle paste is not specifically limited; however, in order to increase the content of metal nanoparticles, the content of the phosphate dispersant is desirably as low as possible. For example, the content of the phosphate dispersant is desirably 0.1 to 10 percent by weight, more desirably 0.3 to 5 percent by weight, and particularly desirably 0.5 to 2 percent by weight.

[0028] The polar solvent is not specifically limited as long as the polar solvent has an affinity for the hydrophilic portion of the phosphate dispersant. For example, the polar solvent may be a protic polar solvent, such as water and an alcohol, an aprotic polar solvent, such as an amide (for example, dimethylacetamide), a nitrile (for example, acetonitrile), a ketone (for example, acetone) and a cyclic ether (for example, tetrahydrofuran), or the like. Although not specifically limited, the polar solvent is desirably an alcohol (for example, C1 to C18 alcohols, or the like). Specifically, the alcohol may be butanol, pentanol, hexanol, heptanol, octanol,

isobornyl cyclohexanol, terpineol, octanediol, decanol, nonanol, undecanol, or the like.

[0029] The content of the polar solvent in the metal nanoparticle paste is not specifically limited; however, in order to increase the content of metal nanoparticles, the content of the polar solvent is desirably as low as possible. For example, the content of the polar solvent is desirably 0.5 to 10 percent by weight, more desirably 1 to 7 percent by weight, and particularly desirably 3 to 5 percent by weight.

[0030] The metal nanoparticle paste may be prepared by mixing the metal nanoparticles, the phosphate dispersant and the polar solvent with one another. The metal nanoparticles tend to flocculate, so the metal nanoparticles each are desirably coated with an organic protection film, such as a fatty acid. The mixing sequence is not specifically limited; however, it is desirable that the phosphate dispersant and the polar solvent are mixed and then the metal nanoparticles are added.

[0031] The metal nanoparticle paste according to the first embodiment of the invention is able to maintain a low viscosity while containing the metal nanoparticles at a high rate. For example, the viscosity of the metal nanoparticle paste is lower than or equal to 300 Pa•s, desirably lower than or equal to 200 Pa•s, and particularly desirably lower than or equal to 100 Pa•s (for example, 1 to 100 Pa•s). Note that, unless otherwise specified in the specification, the value of viscosity means a value that is measured using a cone-plate viscometer under the condition of a rotation speed of 10 rpm and a temperature of 20°C as in the case of the examples described later.

[0032] In addition, the relationship expressed by the inequality (1) desirably holds between the content X (percent by weight) and viscosity Y (Pa•s) of the metal nanoparticles, the relationship expressed by the inequality (2) more desirably holds, and the relationship expressed by the inequality (3) particularly desirably holds.

$0 < Y \leq 3X$ (1)

$0 < Y \leq 2X$ (2)

$0 < Y \leq 1X$ (3)

2. Bonding Method

[0033] Second and third embodiments of the invention respectively relate to a method of bonding a bonded member using the metal nanoparticle paste and a bonded element obtained by the method. The metal nanoparticle paste according to the first embodiment of the invention contains the metal nanoparticles at a high rate, so the

bonded element having an excellent shear strength may be manufactured.

[0034] The bonded member is not specifically limited. The bonded member may be a metal material, a plastic material, a ceramic material, or the like. The metal material may be, for example, a copper substrate, a gold substrate, an aluminum substrate, or the like. The plastic material may be, for example, polyimide, polyethylene, polypropylene, polyethylene terephthalate, polycarbonate, polyethylene naphthalate, or the like. The ceramic material may be, for example, glass, silicon, or the like.

[0035] In addition, the bonded member may be an electronic element. Particularly, when a metal having a high thermal resistance is used as the metal nanoparticles, a power device element, such as silicon carbide and gallium nitride, may be used as the bonded member. Self-cooling may be performed using a power device element, so a cooler is not required, and cost may be significantly reduced.

[0036] The bonded member may be bonded as follows. The metal nanoparticle paste is put on (for example, applied to) the bonded member and is fired. An existing metal paste has a low content of metal. Therefore, unless the existing metal paste is fired under pressure, voids are formed in the metal paste, and it has been difficult to firmly bond a bonded member. On the other hand, the metal nanoparticle paste according to the first embodiment of the invention has an extremely high content of metal nanoparticles. Therefore, the bonded member may be firmly bonded under no pressure. Here, the "no pressure" condition means that it is not required to apply high pressure using a machine, or the like, and does not exclude pressure applied by pressing the bonded member by hand. Because the bonded member may be bonded under no pressure, manufacturing cost of the bonded element may be significantly reduced.

[0037] The bonded element bonded using the metal nanoparticle paste according to the first embodiment of the invention has a high shear strength. The shear strength in the specification means a shear strength obtained in the shear strength test described in the examples. For example, the bonded element according to the third embodiment of the invention has a shear strength of 20 to 120 MPa, desirably 60 to 115 MPa and particularly desirably 80 to 110 MPa.

[0038] In the bonded element according to the third embodiment of the invention, the component composition of the metal nanoparticle paste varies. The

polar solvent that is the component of the paste reduces because of natural evaporation and firing at the time of bonding, so the content of the metal nanoparticles relatively increases. In addition, the phosphate dispersant may also possibly decompose because of firing at the time of bonding. This may influence the content of the metal nanoparticles.

[0039] In the bonded element according to the third embodiment of the invention, phosphorus that originates in the phosphate dispersant tends to segregate near the bonded interface between a portion of the fired metal nanoparticle paste and the bonded member (FIG. 5A and FIG. 5B). Thus, in the bonded element according to the third embodiment of the invention, the concentration of phosphorus is relatively high near the bonded interface, and the concentration of phosphorus is relatively low at the other portions.

[0040] The metal nanoparticle paste according to the first embodiment of the invention may be used to form a wire of an electronic substrate. Thus, a fourth embodiment of the invention relates to an electronic substrate having a wire formed from the metal nanoparticle paste. The metal nanoparticle paste according to the first embodiment of the invention has a low viscosity and contains a large amount of metal nanoparticles having a small particle diameter. Therefore, it is possible to form a fine wire.

Examples

[0041] Hereinafter, the embodiments of the invention will be described in more detail using examples and a comparative example; however, the technical scope of the invention is not limited to these examples.

1. Preparation of Metal Nanoparticle Paste

[0042] Silver nanoparticles (up to 50 nm), silver nanofillers (100 to 200 nm) and silver fillers (300 nm) that serve as metal nanoparticles, DISPER-BYK111 (produced by BYK) that serves as a phosphate dispersant and isobornyl cyclohexanol (product name: Terusolve MTPH, produced by Nippon Terpene Chemicals, Inc.) and octanol that serve as a polar solvent were mixed at the ratios shown in the following table to thereby prepare silver nanoparticle pastes respectively having silver contents of 90 percent by weight, 91 percent by weight, 92 percent by weight, 93 percent by weight, 94 percent by weight and 95 percent by weight.

Table 1

	Silver Content (wt%)	Silver Nano-particles (wt%)	Silver Nano-fillers (wt%)	Silver Fillers (wt%)	DISPER-BYK111 (wt%)	Isobornyl cyclo-hexanol (wt%)	Octanol (wt%)
Example	90	34.1	40.7	15.2	2	3	5
	91	34.5	41.2	15.3	1.8	2.7	4.5
	92	34.9	41.6	15.5	1.6	2.4	4
	93	35.2	42.1	15.7	1.4	2.1	3.5
	94	35.6	42.5	15.9	1.2	1.8	3
	95	36	43	16	1	1.5	2.5
Comparative Example	88.8	32.7	42.1	14	0	4.7	6.5

2. Measurement of Viscosity

[0043]

A cone-plate viscometer (cone rotor: 3°×R9.7) (TV-25 viscometer, produced by Toki Sangyo Co., Ltd.) was used to measure the viscosity of each of the silver nanoparticle pastes at various rotation speeds (rpm) and a temperature of 20°C. The results are shown in FIG. 1.

3. Shear Strength Test

[0044] Each of the above silver nanoparticle pastes was applied to a 3mm × 3mm copper plate and was stuck to a 50mm × 10mm copper plate (FIG. 2A). After that, the 50mm × 10mm copper plate having the stuck 3mm × 3mm copper plate was fired at 250°C under no pressure for an hour to bond the 3mm × 3mm copper plate. Thus, a specimen was prepared. Subsequently, a push-pull gauge RX-100 (produced by Aikoh Engineering, Co., Ltd.) was used to measure the shear strength of each specimen (FIG. 2B). The test was conducted five times for each silver content. The results are shown in FIG. 3.

[0045] For comparison, the shear strength of a specimen obtained by using a metal nanoparticle paste that contains silver nanoparticles (32.7 percent by weight), silver nanofillers (42.1 percent by weight), silver fillers (14 percent by weight), isobornyl cyclohexanol (4.7 percent by weight) and octanol (6.5 percent by weight), which was fired at 350°C under no pressure for an hour, was also measured. The

viscosity of the paste and the bonding strength of the specimen are shown in FIG. 4.

4. Elemental Analysis

[0046] The ratio of elements near the bonded interface between the silver portion and the copper portion in each of the specimens created using the silver nanoparticle paste according to the embodiments of the invention was analyzed by TEM-EDS. As shown in FIG. 5B, it appeared that phosphorus that originates in the phosphate dispersant segregated around the bonded interface.

CLAIMS:

1. A metal nanoparticle paste characterized by comprising:
metal nanoparticles;
a phosphate dispersant that has a hydrophilic portion; and
a polar solvent, wherein
content of the metal nanoparticles is higher than or equal to 70 percent by weight and lower than 100 percent by weight.

2. The metal nanoparticle paste according to claim 1, wherein the content of the metal nanoparticles is higher than or equal to 90 percent by weight and lower than 99 percent by weight.

3. The metal nanoparticle paste according to claim 2, wherein the content of the metal nanoparticles is higher than or equal to 95 percent by weight and lower than 99 percent by weight.

4. The metal nanoparticle paste according to any one of claims 1 to 3, wherein the metal nanoparticles are made of at least one selected from the group consisting of gold, silver, ruthenium, rhodium, palladium, iridium, platinum, copper, aluminum, iron and nickel.

5. The metal nanoparticle paste according to any one of claims 1 to 4, wherein the metal nanoparticles are made of at least one of a metal oxide and a metal salt, having electrical/thermal conductivity.

6. The metal nanoparticle paste according to any one of claims 1 to 5, wherein the viscosity of the metal nanoparticle paste is lower than or equal to 100 Pa•s.

7. The metal nanoparticle paste according to any one of claims 1 to 6, wherein the hydrophilic portion is one selected from the group consisting of polyalkylene glycol and polyglycerol.

8. The metal nanoparticle paste according to claim 7, wherein the hydrophilic portion is one selected from the group consisting of polyethylene glycol, polytetraethylene glycol and polypropylene glycol.

9. The metal nanoparticle paste according to any one of claims 1 to 8, wherein the polar solvent has an affinity for the hydrophilic portion of the phosphate dispersant.

10. The metal nanoparticle paste according to claim 9, wherein the polar solvent is any one of a protic polar solvent and an aprotic polar solvent.

11. The metal nanoparticle paste according to claim 10, wherein the protic polar solvent is one selected from the group consisting of water and alcohol.

12. The metal nanoparticle paste according to claim 10, wherein the aprotic polar solvent is one selected from the group consisting of an amide, a nitrile, a ketone and a cyclic ether.

13. A bonding method characterized by comprising:

applying the metal nanoparticle paste according to any one of claims 1 to 12 to a bonded member.

14. The bonding method according to claim 13, further comprising:

bonding the bonded member under no pressure.

15. A bonded element obtained by the bonding method according to claim 13 or 14.

16. The bonded element according to claim 15, wherein the shear strength of the bonded element is 80 to 110 MPa.

17. An electronic substrate characterized by comprising:

a wire formed from the metal nanoparticle paste according to any one of claims 1 to 12.

FIG. 1

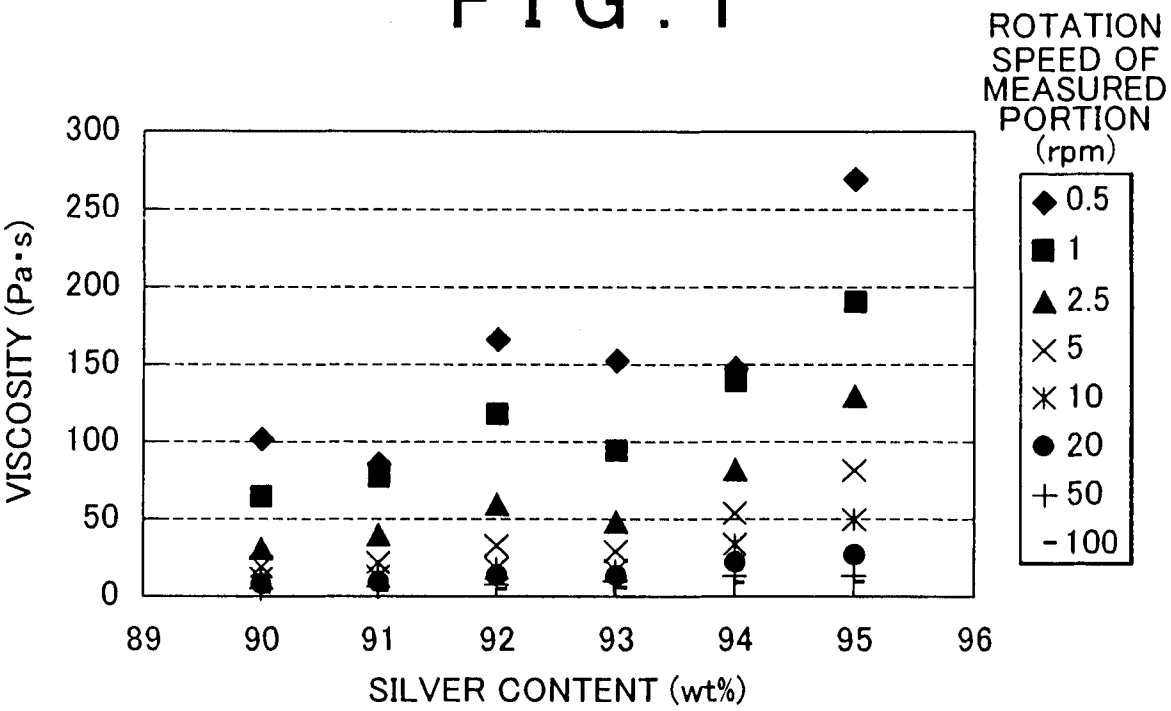


FIG. 2A

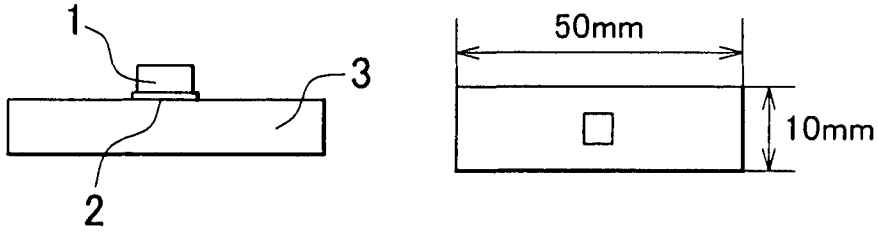


FIG. 2B

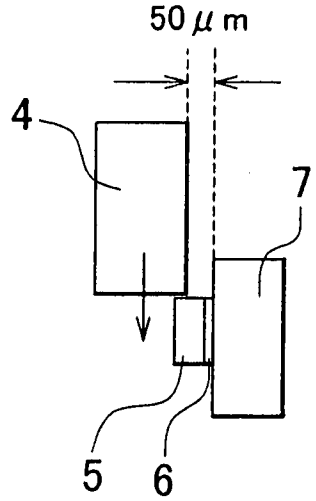


FIG. 3

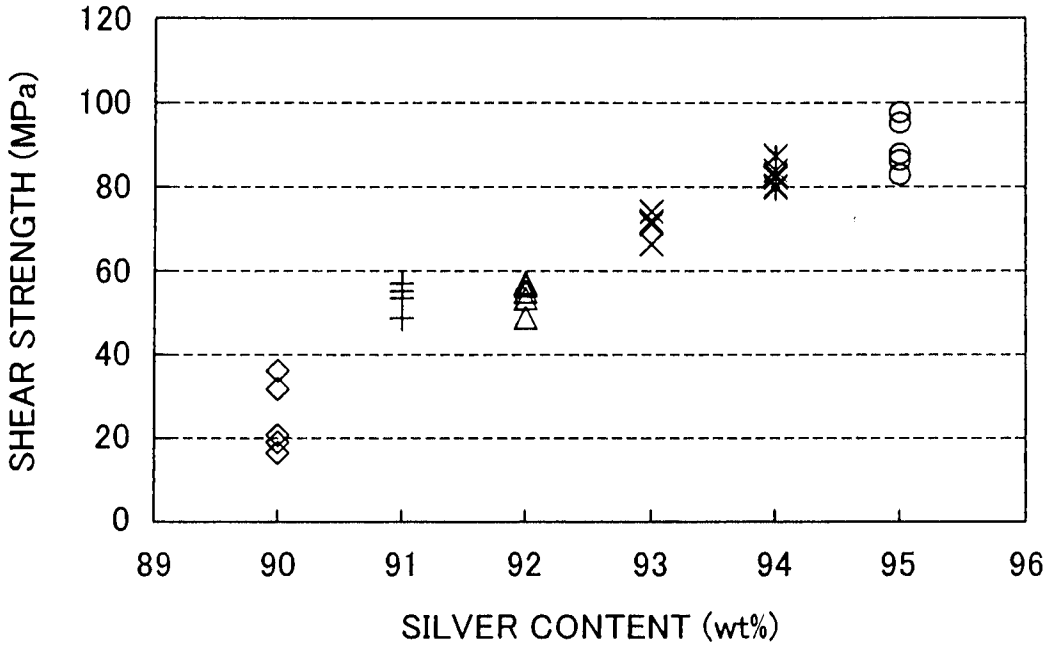
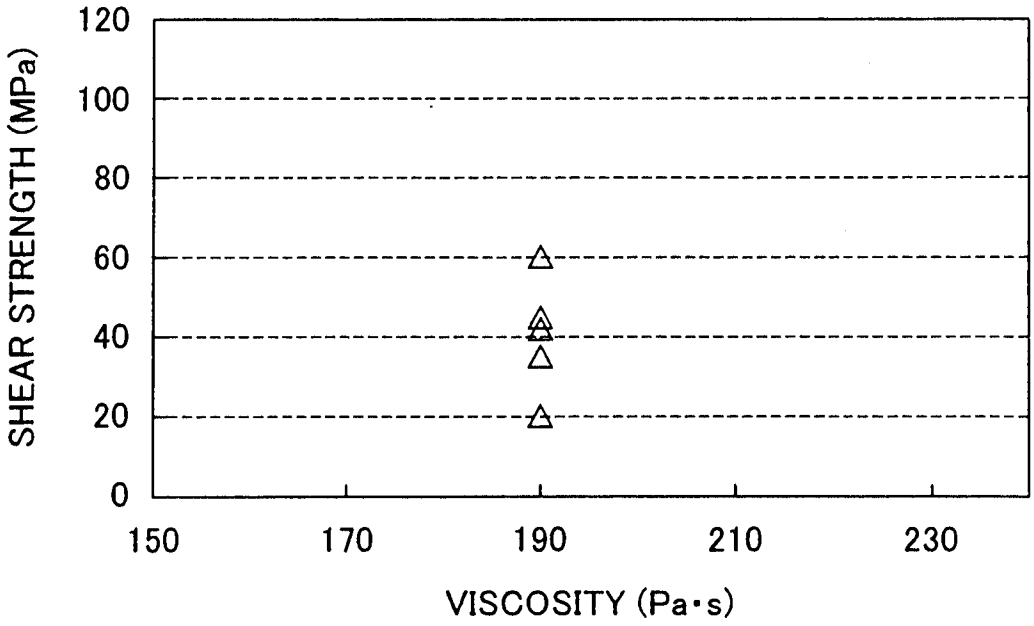

SHEAR STRENGTH (MPa)
120
100
80
60
40
20
0
89
90
91
92
93
94
95
96
SILVER CONTENT (wt%)

FIG. 4


SHEAR STRENGTH (MPa)
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100
80
60
40
20
0
150
170
190
210
230
VISCOSITY (Pa·s)

FIG. 5A

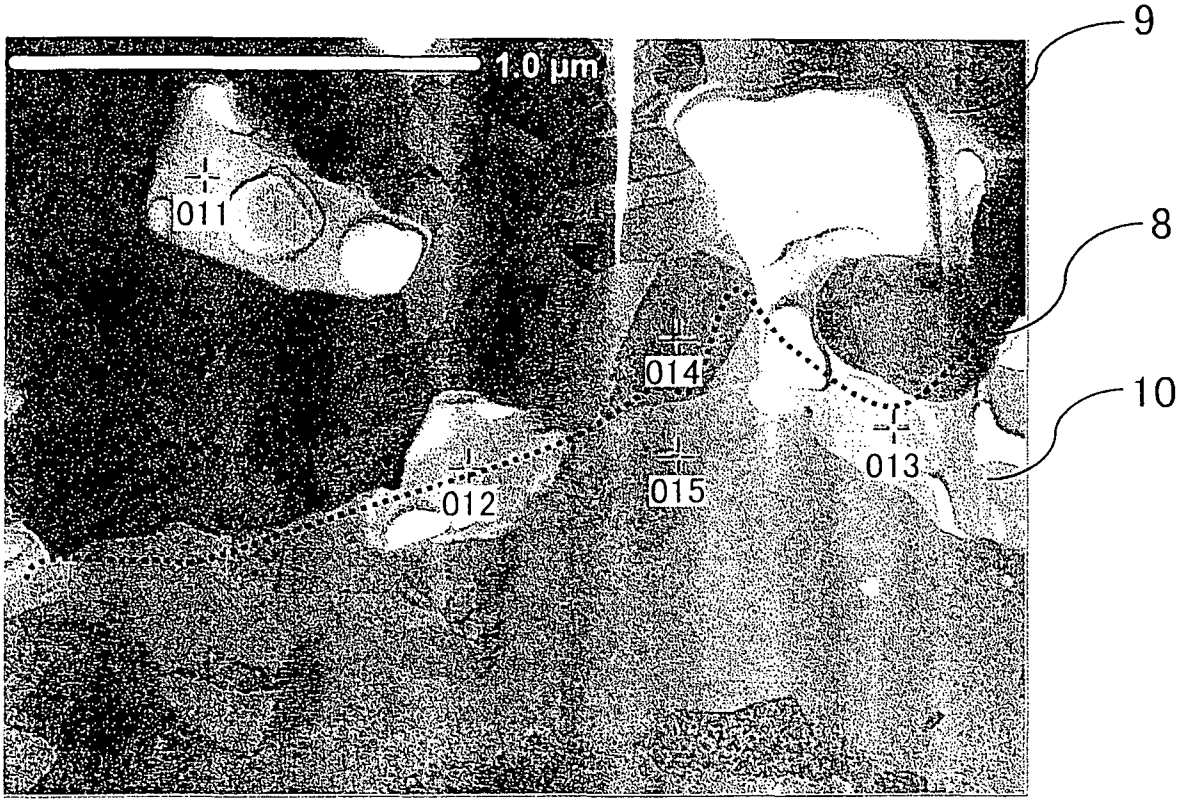
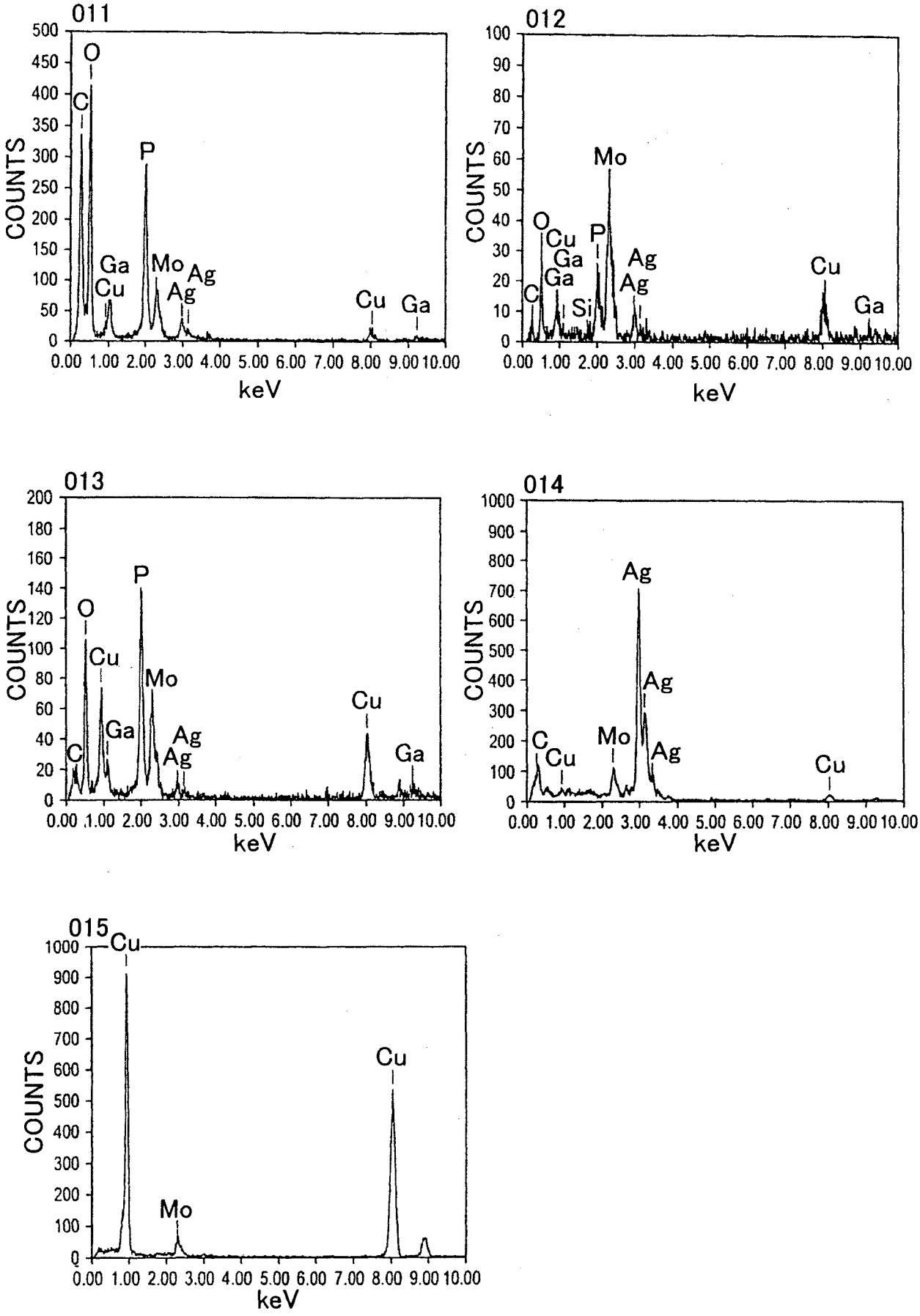


FIG. 5B



INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2012/001160

A. CLASSIFICATION OF SUBJECT MATTER
INV. B23K35/02 B23K35/365
ADD.

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)
B23K

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)
EPO-Internal, WPI Data, INSPEC

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	US 2007/012899 A1 (LEE EUN-SUNG [KR] ET AL) 18 January 2007 (2007-01-18) paragraphs [0023] - [0046]; claims 1-19; examples 1-3 -----	1-12 13-17
Y	JP 2009 279649 A (TOYOTA MOTOR CORP; TOYOTA CENTRAL RES & DEV; EBARA CORP) 3 December 2009 (2009-12-03) cited in the application abstract -----	13-17
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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
25 September 2012	02/10/2012

Name and mailing address of the ISA/	Authorized officer
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1

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INTERNATIONAL SEARCH REPORT

International application No
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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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Information on patent family members

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