DESCRIPTION

Title of Invention: BONDING PASTE, BONDED BODY, AND METHOD FOR PRODUCING BONDED BODY

Technical Field

[0001] The disclosure relates to a bonding paste, a bonding body, and a method for producing a bonding body.

Background Art

[0002] Conventionally, solder has been used as a bonding material for adhering metal members to each other, metal members to semiconductor elements, metal members to light-emitting diode (LED) elements, etc. In recent years, in the field of next-generation power electronics technology, there has been a demand for devices such as SiC capable of operating at high temperatures. As bonding materials for producing such devices, materials alternative to the solder are required from the viewpoint of high-temperature driving reliability. For example, as shown in Patent Document 1 to Patent Document 3, bonding materials such as bonding pastes using metal particles with sinterability have been proposed.

[0003] Patent Document 1 discloses a bonding material containing a metal nanoparticle coated with amine and amine that is straight-chain alkylamine or alkanolamine. Patent Document 2 discloses the use of a conductive adhesive containing a silver particle having an average particle diameter of 20 to 500 nm, alkanolamine, and a solvent as a bonding material. Patent Document 3 discloses a bonding composition containing a copper powder, a liquid medium, and a reducing agent, and the reducing agent has at least one amino group and multiple hydroxyl groups.

Related Art Documents

Patent Documents

[0004] Patent Document 1: Japanese Patent Application Laid-Open No. 2014-214357

Patent Document 2: Japanese Patent Application Laid-Open No. 2022-117824

Patent Document 3: International Patent Publication No. 2020-032161

SUMMARY OF INVENTION

Problem to Be Solved by Invention

[0005] As a bonding process using such bonding materials, there are methods performed without pressurization (hereinafter referred to as non-pressurized bonding) and methods performed with pressurization (hereinafter referred to as pressurized bonding). Pressurized bonding is effective in reducing voids, and since cracks originating from voids are suppressed, it has the advantage of excellent bonding strength and thermal cycle properties. However, pressurized bonding requires specialized equipment, and furthermore, the target to be bonded must be able to withstand the pressurized environment, which limits the bonding targets and applications. Thus, there is a demand to reduce voids and improve the bonding strength and thermal cycle properties in non-pressurized bonding.

On the other hand, in recent years, semiconductor elements have been increasing in size, but it is known that the larger area the element becomes, the more difficult it is for internal gases to escape, leading to a tendency for voids to occur more easily. In addition, the difference between the linear expansion coefficient of Si or SiC elements and the linear expansion coefficient of silver particles and copper used as substrates is very large, and during thermal cycles, strain occurs between layers of the copper substrate/the bonding layer containing silver particles/the Si element, leading to a tendency for cracks to occur and thermal cycle properties to deteriorate easily. Furthermore, although SiC elements have linear expansion coefficients similar to Si elements, SiC elements have higher hardness compared to Si elements, so the strain during thermal cycles is larger compared to Si elements, leading to a tendency for cracks to occur more easily in the bonding layer. In other words, the level of requirements regarding improvement in the bonding strength and thermal cycle properties of SiC semiconductor elements has been rising in recent

years.

However, the invention described in Patent Document 1 involves pressurized bonding of a copper element with a small area using a bonding material containing a silver particle and alkanolamine such as triethanolamine, and cannot solve the problem of the bonding strength and thermal cycle properties in non-pressurized bonding and large-area SiC elements. In addition, the invention described in Patent Document 2 involves non-pressurized bonding of a Si element with a small area using a bonding material containing a silver particle and alkanolamine such as diglycolamine, and cannot solve the problem of the bonding strength and thermal cycle properties in large-area SiC elements. Furthermore, the invention described in Patent Document 3 involves pressurized bonding of a copper element with a small area using a bonding material containing a copper particle and alkanolamine, but a copper particle is more easily oxidized compared to a silver particle, and upon oxidization, the function of the copper particle cannot be exhibited, so there is a problem in handling. Especially in bonding applications heated to about 200°C or higher, oxidation progresses even with the presence of a small amount of oxygen during the heating process, which makes oxidation suppression difficult, and cannot solve the problem of the bonding strength and thermal cycle properties in non-pressurized bonding and large-area SiC elements. Also, Patent Document 3 does not describe the use of silver particles.

[0006] Thus, the problem to be solved by the disclosure is to provide a bonding paste that uses a silver particle, is excellent in bonding strength, suppresses a decrease in bonding strength accompanying thermal cycles, and enables bonding even without pressurization, and a bonding body using the bonding paste.

Means for Solving Problem

[0007] The present inventors have conducted intensive research to solve the above problem, thus leading to the disclosure.

A bonding paste according to an aspect of the disclosure includes: a silver particle (A) having an average particle diameter of 100 nm to 500 nm; a compound (B) having at least one

nitrogen atom selected from the group consisting of a secondary nitrogen atom and a tertiary nitrogen atom and having a number of hydroxyl groups of 4 or more; and a dispersion medium (C).

[0008] In the bonding paste according to an aspect of the disclosure, in a case where a sintering temperature of the silver particle (A) is T1°C, a 50% weight reduction temperature of the compound (B) is T2°C, and a 50% weight reduction temperature of the dispersion medium (C) is T3°C, the bonding paste satisfies $T3 < T1 < (T2 + 10)$.

[0009] In the bonding paste according to an aspect of the disclosure, the T2 is 200 to 300°C.

[0010] In the bonding paste according to an aspect of the disclosure, the compound (B) has a tertiary nitrogen atom.

[0011] In the bonding paste according to an aspect of the disclosure, the number of tertiary nitrogen atoms in the compound (B) is 1 to 3.

[0012] In the bonding paste according to an aspect of the disclosure, the number of hydroxyl groups in the compound (B) is 4 to 6.

[0013] In the bonding paste according to an aspect of the disclosure, the compound (B) is liquid at 25°C.

[0014] In the bonding paste according to an aspect of the disclosure, a content ratio of the compound (B) is 0.05 to 1.0 mass% based on a mass of the silver particle (A).

[0015] In the bonding paste according to an aspect of the disclosure, the silver particle (A) has an average particle diameter of 150 nm to 400 nm.

[0016] In the bonding paste according to an aspect of the disclosure, the dispersion medium (C) has a boiling point of 250°C or higher.

[0017] The bonding paste according to an aspect of the disclosure further includes a compound (D) with a carbon number of 20 to 80 having, in a quantity of two or more, at least one functional group selected from the group consisting of a hydroxyl group, a carboxy group, and an amino group.

[0018] The bonding paste according to an aspect of the disclosure further includes a compound (E) with a carbon number of 14 to 20 having one carboxy group.

[0019] In the bonding paste according to an aspect of the disclosure, a content ratio of the silver particle (A) is 80 to 95 mass% based on a mass of the bonding paste.

[0020] A bonding body according to an aspect of the disclosure is obtained by bonding a first bonded part and a second bonded part with the above bonding paste.

[0021] In the bonding body according to an aspect of the disclosure, the first bonded part is a non-plated substrate.

[0022] In the bonding body according to an aspect of the disclosure, the second bonded part is SiC.

[0023] A method for producing a bonding body according to an aspect of the disclosure is a method for producing a bonding body obtained by bonding a first bonded part and a second bonded part with the above bonding paste, and includes processes below:

(1) a process of applying the bonding paste to the first bonded part;

(2) a process of placing the second bonded part on an applied part of the first bonded part to which the bonding paste has been applied;

(2a) a process of heating a placed laminate for preliminary drying; and

(3) a process of sintering the placed laminate under a non-pressurized environment.

Effects of Invention

[0024] The present invention is capable of providing a bonding paste that uses a silver particle, has a high bonding strength at the bonded site, suppresses a decrease in bonding strength accompanying thermal cycles, and enables bonding even without pressurization, and a bonding body using the bonding paste. Accordingly, even in the case of bonding a large-area SiC element without pressurization, excellent bonding strength and thermal cycle properties can be exhibited.

EMBODIMENTS FOR IMPLEMENTING INVENTION

[0025] In this specification, “a silver particle (A) having an average particle diameter of 100 nm to 500 nm” may be abbreviated as “a silver particle (A)”, and “a compound (B) having at least one nitrogen atom selected from the group consisting of a secondary nitrogen atom and a tertiary nitrogen atom, and having a number of hydroxyl groups of 4 or more” may be abbreviated as “a compound (B)”.

[0026] A bonding paste of the disclosure is characterized by including a silver particle (A) having an average particle diameter of 100 nm to 500 nm, a compound (B) having at least one nitrogen atom selected from the group consisting of a secondary nitrogen atom and a tertiary nitrogen atom and having a number of hydroxyl groups of 4 or more, and a dispersion medium (C).

By combining the silver particle (A) with the compound (B) having a specific structure, the bonding paste of the disclosure achieves improved wettability to a substrate during a sintering stage of the silver particle (A) having a specific particle diameter due to the high non-volatility of the compound (B), and suppresses formation of voids in a coating film, thus forming a dense coating film even in non-pressurized bonding.

Furthermore, in the case where a bonded part is a substrate that has not been subjected to a plating treatment (non-plated substrate) such as non-plated copper, since the surface of the non-plated substrate becomes oxidized and forms oxides due to heating during sintering, bonding between the silver particle and the non-plated substrate covered with oxides becomes insufficient, and the bonding strength tends to decrease and the thermal cycle properties tend to deteriorate. However, by including the compound (B), oxidation of the surface of the non-plated substrate is suppressed and/or the oxides are reduced by the reduction function of the compound (B), and strong bonding becomes possible between the silver particle (A) and the non-plated substrate. Thus, not only can improved bonding strength and good thermal cycle properties be obtained, but it is also possible to eliminate the need for performing a plating treatment on the substrate or cleaning the substrate in advance.

According to the above, even in non-pressurized bonding and in bonding of a bonded part that is difficult to bond stably, such as a non-plated substrate and a large-area SiC semiconductor element, void formation is suppressed, and excellent bonding strength and bonding strength after thermal cycles can be exhibited.

[0027] <Silver particle (A)>

The silver particle (A) serves a role of expressing electrical conductivity and thermal conductivity of a bonding body and bonding bonded bodies during the sintering process, and also includes an alloy containing silver, silver oxide, and a silver-coated particle having a metal (excluding silver) as a core body with a surface thereof coated with silver. By using the silver particle (A), a bonding body having excellent strength can be obtained. In addition, it is possible to accommodate a wide range of sintering temperatures, and it is also possible to accommodate various sintering environments such as an atmospheric pressure, a nitrogen atmosphere, vacuum, a reducing atmosphere, etc.

[0028] By having an average particle diameter in the range of 100 nm to 500 nm, the silver particle (A) has a function of melting or bonding (hereinafter also referred to as sintering) between particles in the temperature range of 200°C to 350°C in which the bonding paste is heated and sintered, and it becomes possible to change into bulk metal. As a result, the bonded bodies are bonded. Hereinafter, the portion formed by sintering of the silver particle (A) present between the bonded bodies will be called a bonding layer.

[0029] In the disclosure, it is important to use a silver particle (A) having a specific average particle diameter. The “average particle diameter” referred to in this specification means a 50% particle diameter (d50) in a cumulative particle diameter distribution on a volume basis obtained according to a measurement method described in Examples. The d50 of the silver particle (A) is 100 to 500 nm, preferably 150 nm or more, more preferably 180 nm or more, and even more preferably 200 nm or more. In addition, the d50 of the silver particle (A) is preferably 450 nm or less, more preferably 400 nm or less, even more preferably 350 nm or less, and particularly

preferably 300 nm or less.

[0030] The silver particle (A) is preferably coated with an organic component (a) on the surface. That is, the silver particle (A) is preferably a particle coated with an organic component (a) on the surface of the silver particle, and the coated organic component (a) is also referred to as a protective agent. When coated with the organic component (a), storage stability of the bonding paste can be expected to increase. Examples of the organic component (a) include fatty acids, aliphatic amines, aliphatic alcohols, etc., and the organic component (a) is preferably a saturated or unsaturated fatty acid, more preferably a saturated or unsaturated fatty acid having a carbon number of 3 to 18, and even more preferably a saturated or unsaturated fatty acid having a carbon number of 6 to 18. The organic component (a) may include one or two or more types.

[0031] The silver particle (A) may be used alone or may be used in a combination of multiple types. In addition, as necessary, a metal particle other than the silver particle (A) may also be used in combination. In the case of using a metal particle other than the silver particle (A) in combination, a metal particle with a particle diameter exceeding an average particle diameter of 500 nm may be combined, or a metal particle with a particle diameter exceeding an average particle diameter of 1000 nm may be combined.

[0032] <Compound (B)>

The bonding paste of the disclosure includes a compound (B) having at least one nitrogen atom selected from the group consisting of a secondary nitrogen atom and a tertiary nitrogen atom and having a number of hydroxyl groups of 4 or more.

The compound (B) serves the function of uniformly dispersing the silver particle (A), suppresses aggregation of the silver particle (A) over time, and further enables good viscosity properties and coating properties. Specifically, the secondary nitrogen atom and the tertiary nitrogen atom are excellent in the function of reducing the silver particle (A) and the bonded part. In addition, the secondary nitrogen atom and the tertiary nitrogen atom are excellent in bondability with the silver particle (A) and thus improve dispersibility. In particular, the tertiary nitrogen

atom has a branched structure and thus is excellent in dispersibility due to steric hindrance effects.

In addition, since the secondary nitrogen atom and the tertiary nitrogen atom are excellent in the function of reducing the silver particle (A) and the bonded part, by including the compound (B), the silver particle (A) is reduced, and the silver layer after sintering becomes firm. In addition, oxidation of the substrate surface, which is the bonded part, is suppressed, and the bonding force at the interface is enhanced.

Furthermore, the compound (B) serves the function of imparting fluidity to the silver particle (A) during firing and densifying the silver layer after sintering.

From the viewpoints of the bonding strength and the thermal cycle properties, the compound (B) preferably has a tertiary nitrogen atom, and the number of tertiary nitrogen atoms is preferably 1 to 3, and more preferably 2 to 3. With the number of tertiary nitrogen atoms being 2 or more, bondability with the silver particle (A) is enhanced, and dispersibility is significantly improved. In addition, the tertiary nitrogen atom has the function of reducing the silver particle (A) and the bonded part, and with the number of tertiary nitrogen atoms being 2 or more, the reduction function is enhanced.

In addition, from the viewpoints of dispersibility and the reduction function, the number of hydroxyl groups of the compound (B) is preferably 4 to 6. In particular, the hydroxyl group is excellent in bondability with the silver particle (A) and thus tends to improve dispersibility, and with the number of hydroxyl groups being 4 or more, bondability with the silver particle (A) is enhanced, and dispersibility is significantly improved. In addition, the hydroxyl group has the function of reducing the silver particle (A) and the bonded part, and with the number of hydroxyl groups being 4 or more, the reduction function is greatly improved.

[0033] The compound (B) is preferably a compound which is liquid at 25°C. In this specification, “liquid” means having fluidity without being in a solid state, with a viscosity of 1000 Pa·s or less measured using an E-type viscometer at 25°C and 2.5 rpm.

By being liquid at 25°C, wettability at the bonding interface with the bonded part

increases during the process from solvent drying to sintering, and the contact area increases. As a result, it becomes possible to produce a bonding body having a firm bonding interface. In addition, during the process from solvent drying to sintering, even if voids are formed in the bonding layer, since the fluidity of the bonding paste increases, the liquid compound (B) flows into defect parts such as the voids, and it becomes possible to obtain a bonding layer and a bonding body with few defects.

[0034] Generally, since the silver particle (A) is a powder, in the case where the compound (B) is not included, it becomes difficult to form an interface with the bonded part as the dispersion medium (C) (to be described later) volatilizes from the bonding paste. However, by including the compound (B) of the disclosure, even after a part or all of the dispersion medium (C) has volatilized, the composition can be present in a liquid form, and a good bonding interface integrated with the bonded part can be formed.

In addition, one of the factors for defects (voids) in the bonding layer is the rapid volatilization of the dispersion medium (C) in the firing temperature range in the case where volatilization of the dispersion medium (C) has been insufficient. However, by including the compound (B), even after the dispersion medium (C) has dried and been removed, the coating film can flow and form a bonding interface. Accordingly, it is possible to provide a preliminary drying process of drying the dispersion medium (C) in advance, and it becomes possible to form a bonding layer with fewer defects.

In addition, in the case where the compound (B) has the property of being liquid in the sintering temperature range of the silver particle (A), since wettability at the bonding interface with the bonded part is improved and the contact area increases, it is possible to obtain a bonding body having a firmer bonding interface. Furthermore, even if voids occur in the bonding layer during sintering, since the bonding paste has fluidity, the liquid compound (B) can flow into the void parts, and voids can be reduced.

[0035] Specific examples of the compound (B) include, for example, bis(2-

hydroxyethyl)aminotris(hydroxymethyl)methane (number of tertiary nitrogen atoms: 1, number of hydroxyl groups: 5), N,N,N',N'-tetrakis(2-hydroxyethyl)ethylenediamine (number of tertiary nitrogen atoms: 2, number of hydroxyl groups: 4), N,N,N',N",N"-pentakis(2-hydroxyethyl)diethylenetriamine (number of tertiary nitrogen atoms: 3, number of hydroxyl groups: 5), N,N,N',N'-tetrakis(2-hydroxypropyl)ethylenediamine (number of tertiary nitrogen atoms: 2, number of hydroxyl groups: 4), 1,3-bis[tris(hydroxymethyl)methylamino]propane (number of secondary nitrogen atoms: 2, number of hydroxyl groups: 6), N,N,N',N'-tetrakis(2-hydroxyethyl)adipamide (number of tertiary nitrogen atoms: 2, number of hydroxyl groups: 4), bis(2-hydroxypropyl)aminotris(hydroxymethyl)methane (number of tertiary nitrogen atoms: 1, number of hydroxyl groups: 5), and N,N,N',N",N"-pentakis(2-hydroxypropyl)diethylenetriamine (number of tertiary nitrogen atoms: 3, number of hydroxyl groups: 5).

Particularly, bis(2-hydroxyethyl)aminotris(hydroxymethyl)methane, N,N,N',N'-tetrakis(2-hydroxyethyl)ethylenediamine, and N,N,N',N'-tetrakis(2-hydroxypropyl)ethylenediamine are preferably used. These compounds (B) may be used alone or may be used in a combination of multiple types.

[0036] The content ratio of the compound (B) is preferably 0.02 mass% or more based on the mass of the silver particle (A). With the content ratio being 0.02 mass% or more, dispersibility of the silver particle (A) is improved, and sintering progresses with the compound (B) remaining in the coating film. Since sintering progresses with the coating film having fluidity, adhesion to the substrate is improved and defects in the bonding layer are reduced. From the viewpoints of dispersibility, adhesion to the substrate, and reduction of defects in the bonding layer, the content ratio is more preferably 0.05 mass% or more, and even more preferably 0.10 mass% or more.

In addition, the content ratio of the compound (B) is preferably 2.00 mass% or less based on the mass of the silver particle (A). With the content ratio being 2.00 mass% or less, the amount remaining in the coating film after sintering can be reduced, and the bonding strength and the thermal cycle properties are excellent. From the viewpoint of reducing the remaining amount,

the content ratio is more preferably 1.0 mass% or less, and even more preferably 0.5 mass% or less.

[0037] <Dispersion medium (C)>

The bonding paste of the disclosure includes a dispersion medium (C) (excluding the compound (B) and a compound (D) to be described later). The dispersion medium (C) serves the function of dispersing the silver particle (A) and the compound (B). In addition, the dispersion medium (C) serves the role of imparting fluidity to the coating film during the sintering process of silver. The carbon number of the dispersion medium (C) is preferably less than 20.

[0038] The dispersion medium (C) may be any dispersion medium as long as it is capable of uniformly dispersing the silver particle (A) and the compound (B), and specific examples include, for example, terpineol, dihydroterpineol, dihydroterpinyl acetate, Terusolve TOE-100, Terusolve MTPH (manufactured by Nippon Terpene Chemicals, Inc.), Texanol (2,2,4-trimethylpentane-1,3-diol monoisobutyrate), Carbitol, Carbitol acetate, butyl Carbitol, isophorone, γ-butyrolactone, dipropylene glycol monomethyl ether, dipropylene glycol monoethyl ether, dipropylene glycol methyl-n-propyl ether, 3-methoxy-3-methylbutyl acetate, ethylene glycol, propylene glycol diacetate, dipropylene glycol methyl ether acetate, 1,3-butylene glycol, 1,3-butanediol, 1,4-butanediol, 2-ethyl-1,3-hexanediol, diethylene glycol monoethyl ether, diethylene glycol monobutyl ether, diethylene glycol monohexyl ether, triethylene glycol monoethyl ether, triethylene glycol monobutyl ether, tetraethylene glycol monobutyl ether, polyethylene glycol monobutyl ether, diethylene glycol monoethyl ether acetate, diethylene glycol monobutyl ether acetate, and isoparaffin-based solvents included in hydrocarbon-based solvents, but are not limited thereto.

[0039] The dispersion medium (C) preferably includes a dispersion medium having a boiling point of 200°C or higher. When including a dispersion medium having a boiling point of 200°C or higher, during the sintering process of silver, the dispersion medium in the coating film dries and decreases relatively gradually, and the coating film can sinter while maintaining high fluidity.

Accordingly, it becomes possible to improve adhesion with the bonded part and obtain a firm bonding body with fewer defects. The proportion of the dispersion medium having a boiling point of 200°C or higher in the mass of the dispersion medium (C) is preferably 70 mass% or more.

[0040] The boiling point of the dispersion medium having a boiling point of 200°C or higher is preferably 240°C or higher, and more preferably 250°C or higher. With the boiling point being 250°C or higher, the coating film can sinter while maintaining high fluidity for a longer time. Thus, a firm bonding body with fewer defects is formed and thermal cycle properties are excellent, which is preferable. In addition, from the viewpoint of suppressing residue of the dispersion medium, the boiling point is preferably 350°C or lower. In the case where multiple types of dispersion media having boiling points of 200°C or higher are included, the boiling point of the dispersion medium having a boiling point of 200°C or higher may be calculated from each boiling point and the mass ratio thereof.

[0041] Examples of the dispersion medium having a boiling point of 200°C or higher include, for example, terpineol, dihydroterpineol, dihydroterpinyl acetate, Terusolve TOE-100, Terusolve MTPH (manufactured by Nippon Terpene Chemicals, Inc.), Texanol (2,2,4-trimethylpentane-1,3-diol monoisobutyrate), isophorone, γ-butyrolactone, dipropylene, 1,3-butanediol, 1,4-butanediol, 2-ethyl-1,3-hexanediol, diethylene glycol monoethyl ether, diethylene glycol monobutyl ether, diethylene glycol monohexyl ether, triethylene glycol monoethyl ether, triethylene glycol monobutyl ether, tetraethylene glycol monobutyl ether, diethylene glycol monoethyl ether acetate, diethylene glycol monobutyl ether acetate, polyethylene glycol monobutyl ether, and hydrocarbon-based solvents having a boiling point of 200°C or higher, but are not limited thereto. These dispersion media having a boiling point of 200°C or higher may be used alone or in a combination of multiple types.

[0042] Among the dispersion media having a boiling point of 200°C or higher, diol-based dispersion media such as 1,3-butanediol, 1,4-butanediol, 2-ethyl-1,3-hexanediol, etc.; and glycol

ether-based dispersion media such as diethylene glycol monohexyl ether, triethylene glycol monoethyl ether, triethylene glycol monobutyl ether, tetraethylene glycol monobutyl ether, polyethylene glycol monobutyl ether, etc. are preferably used. Glycol ether-based dispersion media are more preferable.

As the glycol ether-based dispersion medium having a boiling point of 250°C or higher, diethylene glycol monohexyl ether, triethylene glycol monoethyl ether, triethylene glycol monobutyl ether, tetraethylene glycol monobutyl ether, and polyethylene glycol monobutyl ether are particularly preferably used.

[0043] Particularly preferably, the boiling point of the dispersion medium (C) is 250°C or higher. In the case where the dispersion medium (C) includes multiple types of dispersion media, the boiling point of the dispersion medium (C) may be calculated from the boiling point of each dispersion medium and the mass ratio thereof.

With the boiling point of the dispersion medium (C) as a whole being 250°C or higher, during the sintering process of silver, since the dispersion medium in the coating film dries and decreases relatively gradually, the coating film can sinter while maintaining high fluidity. Accordingly, it becomes possible to improve adhesion with the bonded part and obtain a firm bonding body with fewer defects, which is preferable.

[0044] <Sintering temperature, 50% weight reduction temperature>

In the bonding paste of the disclosure, in the case where the sintering temperature of the silver particle (A) is T1°C, the 50% weight reduction temperature of the compound (B) is T2°C, and the 50% weight reduction temperature of the dispersion medium (C) is T3°C, $T3 < T1 < (T2 + 10)$ is preferably satisfied. At this time, $T3 < T2$ is preferably satisfied. In this manner, by configuring the 50% weight reduction temperature T3 of the dispersion medium (C) to be lower than the 50% weight reduction temperature T1 of the silver particle (A) and the 50% weight reduction temperature T2 of the compound (B), and configuring the 50% weight reduction temperature T2 of the compound (B) to a temperature range exceeding a temperature about 10°C

lower than the 50% weight reduction temperature T1 (sintering temperature) of the silver particle (A), it is inferred that the dispersion medium (C) first volatilizes and is removed, and the compound (B) is present during and after sintering of the silver particle (A). Accordingly, it is believed that defects in the bonding layer are reduced, formation of a firm bonding interface and dense bonding become possible, and excellent bonding strength and thermal cycle properties are exhibited.

[0045] The 50% weight reduction temperatures T2 and T3 of the compound (B) and the dispersion medium (C) may be measured using thermogravimetric differential thermal analysis (hereinafter also referred to as TG-DTA analysis). The measurement method may be, for example, the following method.

Using a thermogravimetric differential thermal analysis apparatus (TG/DTA8122 (manufactured by RIGAKU)), heating is performed under nitrogen atmosphere at a temperature increase rate of 1°C/minute, and the temperature at which 50% weight reduction occurs is taken as the 50% weight reduction temperatures T2 and T3, respectively. In the case where the compound (B) or the dispersion medium (C) is a mixture of two or more types, a result obtained by performing the above measurement using the mixture is used.

[0046] The 50% weight reduction temperature T2 of the compound (B) is preferably 200 to 300°C, more preferably 210°C to 270°C, and even more preferably 210°C to 255°C. With the 50% weight reduction temperature T2 being 200°C or higher, the compound (B) can sufficiently remain in the coating film even in the sintering temperature range, fluidity of the coating film during sintering is improved, adhesion to the substrate is improved, and bonding strength is excellent. With the 50% weight reduction temperature T2 being 300°C or lower, the residue in the coating film after sintering can be reduced, and bonding strength and thermal cycle properties are excellent.

[0047] The sintering temperature T1 of the silver particle (A) may be measured using thermogravimetric differential thermal analysis (hereinafter also referred to as TG-DTA analysis). The measurement method may be, for example, the following method.

Regarding the sintering temperature T1 of the silver particle (A), heating is performed until 400°C under nitrogen atmosphere at a temperature increase rate of 1°C/minute, the protective agent is decomposed, and when the reduction amount thereof is taken as 100, the temperature at which 50% weight reduction occurs is taken as T1.

[0048] <Compound (D)>

The bonding paste of the disclosure may include a compound (D) with a carbon number of 20 to 80 having, in a quantity of two or more, at least one functional group (hereinafter referred to as a functional group (d)) selected from the group consisting of a hydroxyl group, a carboxy group, and an amino group. By including such a compound (D), since the composition can be present in a liquid form even after a part or all of the dispersion medium (C) has volatilized, a good bonding interface integrated with the bonded part can be formed. In addition, each functional group has high bondability with the silver particle (A) and exhibits excellent dispersibility.

[0049] The carbon number of the compound (D) represents a numerical value that also includes the carbon in the functional group (d). Thus, in the case where the functional group (d) in the compound (D) is a carboxy group, a carbon number also including the carbon in the carboxy group is regarded as the carbon number of the compound (D).

[0050] In the compound (D), the skeleton (partial structure) excluding the functional group (d) is an organic residue, but is preferably a hydrocarbon group or a group obtained by bonding multiple hydrocarbon groups by a linking group containing heteroatoms. Examples of such a linking group containing heteroatoms include, for example, -O- group (ether group), -C(=O)- group (carbonyl group), -C(=O)-O- group (ester group or oxycarbonyl group), and -C(=O)-NH- group (amide group or iminocarbonyl group). Preferably, the compound (D) does not have a functional group other than the functional group (d).

[0051] The state of matter of the compound (D) is not particularly limited, but is preferably liquid at 200°C to 350°C, which is a temperature range in which the bonding paste of the disclosure is fired. The compound (D) may be solid or liquid at room temperature (25°C), but from the

viewpoint of being uniformly dispersed in the bonding paste to act effectively, the compound (D) is more preferably liquid at room temperature.

[0052] In the case where the compound (D) has the property of being liquid in the above temperature range, it can be expected that wettability at the bonding interface with the bonded part increases, and the contact area increases. As a result, it becomes possible to produce a bonding body having a firm bonding interface. In addition, even if voids are formed in the bonding layer during sintering, since flowability of the bonding paste increases, it is believed that the liquid compound (D) flows into defect parts such as the voids, and it is possible to obtain a bonding layer and a bonding body with few defects.

[0053] The number of functional groups (d) in the compound (D) is preferably 2 or 3.

[0054] The compound (D) may have a straight-chain structure or may have a branched and/or cyclic structure, but preferably has a branched and/or cyclic structure. In the case of having a branched and/or cyclic structure, it is preferable in terms of having a low crystallinity and readily becoming a liquid with a good fluidity.

[0055] In the case where the number of functional groups (d) of the compound (D) is taken as n, the compound (D) preferably has an n-valent hydrocarbon group. In the compound (D), from the viewpoint of obtaining a firm bonding body, the skeleton excluding the functional group (d) is more preferably composed only of the n-valent hydrocarbon group.

[0056] For the above reasons, preferably, the compound (D) does not immediately evaporate at the start of sintering. However, at the end of sintering, it does not necessarily need to remain in the formed bonding layer. In the case where the carbon number of the n-valent hydrocarbon group is 30 to 60, the amount of the compound (D) remaining in the bonding layer after sintering can be further reduced, and a denser bonding layer is readily obtained, which is preferable. As a result, an initial bonding strength is excellent, and suppression of a decrease in bonding strength accompanying thermal cycles can be further expected.

[0057] Examples of the compound (D) having a straight-chain structure include eicosanedioic

acid, heneicosanedioic acid, docosanedioic acid, tetracosanedioic acid, triacontanedioic acid, dotriacontanedioic acid, tetracontanedioic acid, pentacontanedioic acid, hexacontanedioic acid, and batyl alcohol, but are not limited thereto.

[0058] Examples of the compound (D) having a branched and/or cyclic structure include dimer acid, trimer acid, tetramer acid, dimer diol, trimer triol, tetramer tetraol, dimer diamine, trimer triamine, tetramer tetramine, and phytantriol, but are not limited thereto.

[0059] Among these, for reasons described above, the compound (D) is more preferably a compound selected from the group consisting of dimer acid, trimer acid, dimer diol, trimer triol, dimer diamine, and trimer triamine.

[0060] Dimer acid, trimer acid, and tetramer acid may be produced by polymerization reactions of unsaturated fatty acids. For example, they may be produced by a radical reaction or a Diels-Alder reaction between oleic acid (carbon number 18) and linoleic acid (carbon number 18). Preferably, the carbon number of dimer acid is 36 or 44, the carbon number of trimer acid is 54, and the carbon number of tetramer acid is 72. In addition, it is possible to produce a compound (D) having a carbon number other than those described above by appropriately changing the carbon numbers of the unsaturated fatty acids serving as raw materials. In this specification, dimer, trimer, and tetramer of unsaturated fatty acids with a carbon number of 12 or more are referred to as dimer acid, trimer acid, and tetramer acid, respectively.

[0061] Dimer diol has a structure in which the carboxy groups of dimer acid are reduced to hydroxyl groups; trimer triol has a structure in which the carboxy groups of trimer acid are reduced to hydroxyl groups; and tetramer tetraol has a structure in which the carboxy groups of tetramer acid are reduced to hydroxyl groups. Preferably, the carbon number of dimer diol is 36, the carbon number of trimer triol is 54, and the carbon number of tetramer tetraol is 72.

[0062] Dimer diamine has a structure in which the carboxy groups of dimer acid are functionally converted to amino groups; trimer triamine has a structure in which the carboxy groups of trimer acid are functionally converted to amino groups; and tetramer tetramine has a

structure in which the carboxy groups of tetramer acid are functionally converted to amino groups. Preferably, the carbon number of dimer diamine is 36, the carbon number of trimer triamine is 54, and the carbon number of tetramer tetramine is 72.

[0063] Due to the production process, the compound (D) may be a mixture of compounds with different degrees of polymerization, but the compound (D) may be used as a mixture of different polymers or may be used as one specific compound.

[0064] In the bonding paste of the disclosure, the content of the compound (D) is preferably 0.05 to 2.0 parts by mass, and more preferably 0.1 to 1.0 part by mass, with respect to 100 parts by mass of the silver particle (A). In this range, it is possible to obtain a bonding body that is excellent particularly in the initial bonding strength and suppresses a decrease in bonding strength accompanying thermal cycles.

[0065] [Compound (Dx)]

The bonding paste of the disclosure preferably includes a compound (Dx) with a carbon number of 20 to 80 having three carboxy groups as the compound (D). The compound (Dx) may have a straight-chain structure or may have a branched and/or cyclic structure, but preferably has a branched and/or cyclic structure. In the case of having a branched and/or cyclic structure, it is preferable in terms of having a low crystallinity and readily becoming a liquid with a good fluidity. The compound (Dx) preferably has a trivalent hydrocarbon group. In the compound (Dx), in terms of obtaining a firm bonding body, the skeleton excluding the carboxy groups is more preferably composed only of a trivalent hydrocarbon group. Specific examples of the compound (Dx) having a branched and/or cyclic structure include trimer acid, which is suitably used.

[0066] The bonding paste of the disclosure preferably contains the compound (Dx) in an amount of 0.03 to 2.0 parts by mass with respect to 100 parts by mass of the silver particle (A). With an amount in the above range, excellent bonding strength and thermal cycle properties can be exhibited even under more severe conditions, such as a case of using a bonded substrate that has not been subjected to a plating treatment.

[0067] [Compound (Dy)]

The bonding paste of the disclosure preferably further includes a compound (Dy) with a carbon number of 20 to 80 having two carboxy groups as the compound (D). The compound (Dx) described above and the compound (Dy) having a different number of carboxy groups have different volatilization or decomposition temperatures, with the compound (Dy) having a lower volatilization or decomposition temperature than the compound (Dx). Thus, using them in combination, simultaneous volatilization or decomposition at a specific temperature can be suppressed during the sintering process of silver. Thus, the coating film can be sintered while maintaining a high fluidity for a longer time. Accordingly, adhesion with the bonded part is further improved, and it becomes possible to obtain a firm bonding body with fewer defects.

[0068] The compound (Dy) may have a straight-chain structure or may have a branched and/or cyclic structure, but preferably has a branched and/or cyclic structure. In the case of having a branched and/or cyclic structure, it is preferable in terms of having a low crystallinity and readily becoming a liquid with a good fluidity. The compound (Dy) preferably has a divalent hydrocarbon group. In the compound (Dy), in terms of obtaining a firm bonding body, the skeleton excluding the carboxy groups is more preferably composed only of a divalent hydrocarbon group.

[0069] Examples of the compound (Dy) having a straight-chain structure include eicosanedioic acid, heneicosanedioic acid, docosanedioic acid, tetracosanedioic acid, triacontanedioic acid, dotriacontanedioic acid, tetracontanedioic acid, pentacontanedioic acid, and hexacontanedioic acid, but are not limited thereto. Examples of the compound (Dy) having a branched and/or cyclic structure include dimer acid, which is suitably used.

[0070] The bonding paste of the disclosure preferably contains the compound (Dy) in an amount of 0.03 to 2.0 parts by mass with respect to 100 parts by mass of the silver particle (A). With an amount in the above range, excellent bonding strength and thermal cycle properties can be exhibited even under more severe conditions, such as a case of using a bonded substrate that

has not been subjected to a plating treatment.

[0071] The bonding paste of the disclosure preferably contains a total of the compound (Dx) and the compound (Dy) in an amount of 0.05 to 2.0 parts by mass with respect to 100 parts by mass of the silver particle (A). With the total of the compound (Dx) and the compound (Dy) being in the above range, a bonding body that is excellent particularly in an initial bonding strength and suppresses a decrease in bonding strength accompanying thermal cycles can be obtained.

[0072] <Compound (E)>

The bonding paste of the disclosure may contain a compound (E) with a carbon number of 14 to 20 having one carboxy group. The carbon number of the compound (E) is a carbon number also including the carbon of the carboxy group.

The compound (E) is a fatty acid, and by including such a compound (E), since the composition can be present in a liquid form even after a part or all of the dispersion medium (C) has volatilized, a good bonding interface integrated with the bonded part can be formed. In addition, each functional group has high bondability with the silver particle (A) and exhibits excellent dispersibility.

[0073] Examples of the compound (E) include straight-chain saturated fatty acids, straight-chain unsaturated fatty acids, or branched fatty acids. Examples of the straight-chain saturated fatty acids include myristic acid, pentadecylic acid, palmitic acid, heptadecylic acid, stearic acid, nonadecanoic acid, and arachic acid. Examples of the straight-chain unsaturated fatty acids include palmitoleic acid, oleic acid, and erucic acid. Examples of the branched fatty acids include 2-hexyldecanoic acid.

The compound (E) is preferably a straight-chain unsaturated fatty acid. By including a straight-chain unsaturated fatty acid in the bonding paste of the disclosure, lipophilicity increases, and stability in a non-aqueous solvent is improved. In addition, a straight-chain unsaturated fatty acid has a low decomposition temperature and is excellent in low-temperature sinterability and is thus preferable. The compound (E) may be used as one type alone or may be

used in a combination of multiple types.

[0074] The bonding paste of the disclosure preferably contains the compound (E) in an amount of 0.1 to 2.0 parts by mass, and more preferably 0.2 to 1.0 parts by mass, with respect to 100 parts by mass of the silver particle (A). In the above range, excellent bonding strength and thermal cycle properties can be exhibited even if the bonded part is a substrate that has not been subjected to a plating treatment.

[0075] <Production of bonding paste>

The production method of the bonding paste of the disclosure is not particularly limited as long as the bonding paste contains at least the silver particle (A), the compound (B), and the dispersion medium (C), and the production method may be obtained by using and adjusting conventional methods. Apparatuses for preparing the bonding paste from the silver particle (A), the compound (B), and the dispersion medium (C) include a disper, a three-roll mill, a bead mill, an ultrasonic disperser, a planetary centrifugal mixer, etc.

[0076] The proportion of the mass of the silver particle (A) based on the mass of the bonding paste is preferably 80 mass% to 95 mass%, and more preferably 85 mass% to 94 mass%. By including the silver particle (A) in the above range, the bonding paste can exhibit a good printability, residue of the dispersion medium (C) in the bonding body can be suppressed, formation of voids resulting from the dispersion medium (C) can be suppressed, and a good bonding strength can be exhibited.

[0077] The bonding paste of the disclosure may include an additive, and may include, for example, a sintering promoter, a binder resin, or a resin-type dispersant.

[0078] <Bonding body and method for producing bonding body>

With the bonding paste of the disclosure, a first bonded part and a second bonded part may be bonded to obtain a bonding body. The bonding body may be produced, for example, according to Production Method (I) or Production Method (II) below.

[0079] [Production Method (I)]

Production Method (I) is a method by non-pressurized bonding, and preferably includes processes (1) to (3) below.

(1) A process of applying the bonding paste to a first bonded part.

(2) A process of placing a second bonded part on the first bonded part to which the bonding paste has been applied.

(3) A process of sintering the placed laminate under a non-pressurized environment.

[0080] (1) Application process

A method for applying the bonding paste to the bonded body is not particularly limited as long as it is a method capable of uniformly applying on members. Examples thereof include various printing methods such as screen printing, flexographic printing, offset printing, gravure printing, metal mask printing, gravure offset printing, etc., dispensing methods using a dispenser, etc. The bonding paste of the disclosure is excellent in fluidity even when containing a high concentration of metal particles, and is thus preferably used in combination with metal mask printing in particular.

[0081] (2) Placement process

Next, a second bonded part is placed on the first bonded part to which the bonding paste of the disclosure has been applied. In the case of using the bonding paste of the disclosure, placement can be performed without pressurization. In non-pressurized bonding, preferably, the placement process may also be performed without pressurization, but placement may also be performed while applying a pressure. In the case of applying a pressure, the pressure is appropriately set according to the viscosity of the bonding paste and the drying state of the paste, but is preferably 0.1 to 40 MPa, and more preferably 0.3 to 30 MPa.

[0082] (3) Sintering process

The sintering conditions for non-pressurized bonding a laminate obtained by placing the second bonded part on the first bonded part may be appropriately changed, but examples thereof may include conditions such as 200 to 350°C under atmospheric pressure, nitrogen atmosphere,

vacuum, or reducing atmosphere. Examples of a firing apparatus include a hot air oven, a firing furnace, an electric furnace, an infrared oven, a reflow oven, a microwave oven, a hot plate, a photo firing apparatus, etc. These apparatuses may be used appropriately, alone or in a combination of multiple types.

As non-pressurized sintering conditions, preferably, after raising the temperature to a set temperature at a temperature increase rate of 2°C to 30°C/minute, the temperature is maintained at the set temperature or higher for about 10 minutes to 2 hours. The set temperature is preferably 200 to 350°C, more preferably 250°C to 330°C, and even more preferably 280 to 320°C.

[0083] (2a) Preliminary drying process

In Production Method (I) using non-pressurized bonding, a preliminary drying process (2a) is preferably provided between the process (2) and the process (3) for the purpose of removing organic components from the bonding coating film. With the bonding paste of the disclosure including the compound (B), since the coating film can flow and form a bonding interface even after the dispersion medium (C) has been dried in advance and removed, the preliminary drying process can be provided. By providing the preliminary drying process, residue of the dispersion medium (C), which is one of the causes of defects (voids) in the bonding layer, can be suppressed, and density in the bonding layer is excellent, which is preferable.

The preliminary drying may be performed using an apparatus similar to the firing apparatus under conditions in a range of 60 to 220°C for 1 minute to 300 minutes. The preliminary drying is preferably performed in a range of 70 to 100°C for 30 minutes to 120 minutes.

[0084] That is, Production Method (I) particularly preferably includes processes below.

(1) A process of applying the bonding paste to the first bonded part.

(2) A process of placing the second bonded part on an applied part of the first bonded part to which the bonding paste has been applied.

(2a) A process of heating the placed laminate for preliminary drying.

(3) A process of sintering the placed laminate under a non-pressurized environment.

[0085] [Production Method (II)]

Production Method (II) is a method by pressurized bonding, and preferably includes processes (10) to (30) below.

(10) A process of applying the bonding paste to a first bonded part.

(10a) A process of heating the applied laminate for preliminary drying.

(20) A process of placing a second bonded part on the first bonded part which has been applied with the bonding paste and preliminarily dried.

(30) A process of sintering the placed laminate under a pressurized environment.

[0086] (10) Application process

A method for applying the bonding paste to the bonded body is not particularly limited as long as it is a method capable of uniformly applying on members. Examples thereof include various printing methods such as screen printing, flexographic printing, offset printing, gravure printing, metal mask printing, gravure offset printing, etc., dispensing methods using a dispenser, etc. The bonding paste of the disclosure is excellent in fluidity even when containing a high concentration of metal particles, and is thus preferably used in combination with metal mask printing in particular.

[0087] (10a) Preliminary drying process

In Production Method (II) using pressurized bonding, the preliminary drying process (10a) may be provided between the process (10) and the process (20) for the purpose of removing organic components from the bonding coating film. By providing the preliminary drying process, the preliminary drying may be performed under conditions in a range of 60 to 220°C for 1 minute to 300 minutes using, for example, an apparatus similar to the firing apparatus.

[0088] (20) Placement process

Next, a second bonded part is placed on the first bonded part which has been applied with the bonding paste of the disclosure and preliminarily dried. In the case of pressurized

bonding, the placement process may also be performed under pressurization. The pressure is appropriately set according to the viscosity of the bonding paste and the drying state of the paste, but is preferably 0.1 to 40 MPa, and more preferably 0.3 to 30 MPa.

[0089] (30) Sintering process

The sintering conditions for pressurized bonding a laminate obtained by placing the second bonded part on the first bonded part may be appropriately changed, but examples thereof may include conditions such as 200 to 350°C under atmospheric pressure, nitrogen atmosphere, vacuum, or reducing atmosphere. Examples of a firing apparatus include a hot air oven, a firing furnace, an electric furnace, an infrared oven, a reflow oven, a microwave oven, a hot plate, a photo firing apparatus, etc. These apparatuses may be used appropriately, alone or in a combination of multiple types.

The pressure is appropriately set according to the viscosity of the bonding paste and the drying state of the paste, but is preferably 0.1 to 40 MPa, and more preferably 0.3 to 30 MPa.

[0090] In either production method, the thickness of the bonding layer formed when bonding the bonded parts with the bonding paste is not limited, and the thickness of the bonding layer is preferably 3 μm to 500 μm, more preferably 10 μm to 200 μm, and even more preferably 20 μm to 100 μm.

[0091] [Bonded part]

A type of the bonded part is not particularly limited, and examples thereof may include metal materials, semiconductor materials, plastic materials, ceramic materials, as well as electronic elements. Examples of metals include copper, gold, and aluminum. Examples of semiconductor materials include silicon, germanium, gallium arsenide, gallium phosphide, cadmium sulfide, silicon nitride, graphite, yttrium oxide, magnesium oxide, silicon carbide, and gallium nitride. Examples of plastic materials include polyimide, polyethylene, polypropylene, polyethylene terephthalate, polycarbonate, and polyethylene naphthalate. Examples of ceramic materials include glass and silicon. Examples of electronic elements include semiconductor

elements, LED elements, and power device elements.

[0092] The first bonded part and the second bonded part may be members of the same type and may also be members of different types. To increase the bonding strength at the bonded site of the bonded part, a corona treatment, a plating treatment, etc. may be applied to the surface of the bonded part.

The bonding paste of the disclosure is excellent in bonding strength with respect to a non-plated substrate and a SiC element, and is suitably used for a bonding body in which the first bonded part is a non-plated substrate. In addition, the bonding paste is suitably used for a bonding body in which the second bonded part is SiC.

Among these, by including the compound (B), oxidation of the substrate surface is suppressed, and/or oxides are reduced, and firm bonding becomes possible between the silver particle (A) and the substrate. Since the above effect is particularly effective for easily oxidizable substrates, the bonding paste of the present invention is very effective as a bonding material for copper substrates, which are prone to oxidization, especially non-plated copper substrates.

Examples

[0093] The disclosure will be described in detail below with reference to Examples and Comparative Examples, but the technical scope of the disclosure is not limited thereto. In the examples, unless otherwise specified, “parts” and “%” represent “parts by mass” and “mass%”, respectively. Numerical values in the tables represent “parts” unless otherwise specified.

[0094] [Sintering temperature T1 of silver particle (A)]

Using a thermogravimetric differential thermal analysis apparatus (TG/DTA8122 (manufactured by RIGAKU)), heating was performed until 400°C under nitrogen atmosphere at a temperature increase rate of 1°C/minute, the protective agent was decomposed, and when the reduction amount thereof was taken as 100, the temperature at which 50% weight reduction occurred was used as the sintering temperature T1 of the silver particle (A).

[0095] [50% weight reduction temperatures T2 and T3 of compound (B) and dispersion

medium (C)]

Regarding the 50% weight reduction temperatures T2 and T3 of the compound (B) and the dispersion medium (C), using a thermogravimetric differential thermal analysis apparatus (TG/DTA8122 (manufactured by RIGAKU)), heating was performed under nitrogen atmosphere at a temperature increase rate of 1°C/minute, and the temperature at which 50% weight reduction occurred was taken as the 50% weight reduction temperatures T2 and T3, respectively. In the case where the compound (B) or the dispersion medium (C) was a mixture of two or more types, a result obtained by performing the above measurement using the mixture was used.

[0096] <Production of metal particle>

(Production Example 1) metal particle A1

Under nitrogen atmosphere, 200 parts of toluene and 22.3 parts of silver hexanoate were mixed and stirred at 25°C to form a 0.5 M solution, and then 1.6 parts of diethylaminoethanol and 0.28 parts of oleic acid were added and dissolved as dispersants. Subsequently, upon adding 73.1 parts of a 20% concentration aqueous solution of succinic acid dihydrazide (hereinafter, SUDH) as a reducing agent, the liquid color changed from pale yellow to dark brown. To further promote the reaction, the temperature was raised to 40°C and the reaction was carried out. After standing and separation, the aqueous phase was removed to eliminate excess reducing agent and impurities. Furthermore, distilled water was added to the toluene layer several times, and washing and separation were repeated. Then, toluene was added and centrifuged, then a supernatant was removed, and the process was repeated twice. The precipitate was dried to obtain a metal particle A1 in which a silver particle was coated with hexanoic acid and oleic acid. An average particle diameter (d50) of the metal particle A1 was obtained according to a method to be described later, and d50 was 210 nm.

[0097] (Production Example 2) metal particle A2

A metal particle A2 was obtained in the same manner as in Production Example 1, except that diethylaminoethanol was set to 2.4 parts and the amount of oleic acid was set to 0.42

parts. d50 was 120 nm.

(Production Example 3) metal particle A3

A metal particle A3 was obtained in the same manner as in Production Example 1, except that diethylaminoethanol was set to 2.0 parts and the amount of oleic acid was set to 0.34 parts. d50 was 155 nm.

(Production Example 4) metal particle A4

A metal particle A4 was obtained in the same manner as in Production Example 1, except that diethylaminoethanol was set to 1.8 parts and the amount of oleic acid was set to 0.31 parts. d50 was 185 nm.

(Production Example 5) metal particle A5

A metal particle A5 was obtained in the same manner as in Production Example 1, except that diethylaminoethanol was set to 1.2 parts and the amount of oleic acid was set to 0.18 parts. d50 was 290 nm.

(Production Example 6) metal particle A6

A metal particle A6 was obtained in the same manner as in Production Example 1, except that diethylaminoethanol was set to 1.0 part and the amount of oleic acid was set to 0.14 parts. d50 was 390 nm.

[0098] (Production Example 10) metal particle A10 for comparative example

A metal particle A10 was obtained in the same manner as in Production Example 1, except that diethylaminoethanol was set to 1.4 parts and the amount of oleic acid was set to 0.09 parts. d50 was 600 nm.

(Production Example 11) metal particle A11 for comparative example

A metal particle A11 was obtained in the same manner as in Production Example 1, except that diethylaminoethanol was set to 0.6 parts and the amount of oleic acid was set to 0.070 parts. d50 was 1100 nm.

(Production Example 12) metal particle A12 for comparative example

A metal particle A12 was obtained in the same manner as in Production Example 1, except that diethylaminoethanol was set to 2.3 parts and the amount of oleic acid was set to 2.8 parts. d50 was 20 nm.

(Production Example 13) metal particle A13 for comparative example

A metal particle A13 was obtained in the same manner as in Production Example 1, except that diethylaminoethanol was set to 2.1 parts and the amount of oleic acid was set to 0.71 parts. d50 was 85 nm.

[0099] [Measurement method for average particle diameter of silver particle]

Isopropyl alcohol was added to each silver particle and dispersed using an ultrasonic disperser to obtain a 0.5 mass% dispersion. Particle diameters of metal particles in the obtained dispersion were measured using Nanotrac UPA-EX150 (manufactured by Nikkiso Co., Ltd.), and an average particle diameter (d50) was obtained. Among the metal particles produced according to the above method, the metal particles A1 to A6 correspond to the silver particle (A) of the disclosure, and the metal particles A10 to A13 correspond to metal particles that are not the silver particle (A).

[0100] <Compound (B)>

The following materials were used as the compound (B).

B1: Bis(2-hydroxyethyl)aminotris(hydroxymethyl)methane (number of tertiary nitrogen atoms: 1, number of hydroxyl groups: 5, solid at 25°C)

B2: N,N,N',N'-Tetrakis(2-hydroxyethyl)ethylenediamine (number of tertiary nitrogen atoms: 2, number of hydroxyl groups: 4, liquid at 25°C)

B3: N,N,N',N",N"-Pentakis(2-hydroxypropyl)diethylenetriamine (number of tertiary nitrogen atoms: 3, number of hydroxyl groups: 5, liquid at 25°C)

B4: N,N,N',N'-Tetrakis(2-hydroxypropyl)ethylenediamine (number of tertiary nitrogen atoms: 2, number of hydroxyl groups: 4, liquid at 25°C)

B5: 1,3-Bis[tris(hydroxymethyl)methylamino]propane (number of secondary nitrogen

atoms: 2, number of hydroxyl groups: 6, solid at 25°C)

B6: N,N,N',N'-Tetrakis(2-hydroxyethyl)adipamide (number of tertiary nitrogen atoms: 2, number of hydroxyl groups: 4, solid at 25°C)

B11: Triethanolamine (number of tertiary nitrogen atoms: 1, number of hydroxyl groups: 3, liquid at 25°C)

B12: 2-Amino-2-hydroxymethyl-1,3-propanediol (number of hydroxyl groups: 3, solid at 25°C)

B13: 2-(2-Aminoethoxy)ethanol (number of hydroxyl groups: 1, liquid at 25°C)

B14: Xylitol (number of hydroxyl groups: 5, solid at 25°C)

B15: Imidazole (solid at 25°C)

[0101] <Dispersion medium (C)>

The following materials were used as the dispersion medium (C). The manufacturer name, supplementary information, and boiling point are indicated in parentheses.

Dispersion medium C1: Diethylene glycol monohexyl ether (glycol ether-based, boiling point 255°C)

Dispersion medium C2: Triethylene glycol monobutyl ether (glycol ether-based, boiling point 278°C)

Dispersion medium C3: Triethylene glycol monomethyl ether (glycol ether-based, boiling point 248°C)

Dispersion medium C4: 2-Ethyl-1,3-hexanediol (diol-based, boiling point 244°C)

Dispersion medium C5: Diethylene glycol monomethyl ether (glycol ether-based, boiling point 193°C)

Dispersion medium C6: 1-Decanol (boiling point 233°C)

Dispersion medium C7: “Terusolve TOE-100” manufactured by Nippon Terpene Chemicals, Inc. (boiling point 260°C)

[0102] <Compound (D)>

The following materials were used as the compound (D). Compounds D1 to D4 were all manufactured by Croda Japan K.K., and the carbon number, material name, and state of matter at 25°C are indicated in parentheses.

Compound D1: Pripol 1009 (hydrogenated dimer acid with carbon number of 36; composed of two carboxy groups and a divalent hydrocarbon group having branched and cyclic structure; liquid)

Compound D2: Pripol 1040 (trimer acid with carbon number of 54; composed of three carboxy groups and a trivalent hydrocarbon group having branched and cyclic structure; liquid)

Compound D3: Pripol 2033 (dimer diol with carbon number of 36; composed of two hydroxyl groups and a divalent hydrocarbon group having branched and cyclic structure; liquid)

Compound D4: Priamine 1075 (dimer diamine with carbon number of 36; composed of two amino groups and a divalent hydrocarbon group having branched and cyclic structure; liquid)

[0103] <Compound (E)>

The following material was used as the compound (E).

Compound E1: Oleic acid

[0104] <Production of bonding paste>

[Example 1]

The metal particle A1 (90 parts), diethylene glycol monohexyl ether (10 parts), the compound B1 (0.2 parts), other component D1 (0.1 parts), and other component D2 (0.1 parts) were mixed using a planetary centrifugal mixer to prepare a bonding paste.

[0105] [Examples 2 to 43, Comparative Examples 1 to 9]

Bonding pastes were obtained in the same manner as in Example 1 except that the types of materials and the blending amounts (parts) were changed according to the compositions shown in Table 1A to Table 2B. In the tables, blank spaces indicate that the component was not included.

[0106] <Evaluation of bonding paste>

Using the obtained bonding pastes, bonding bodies were produced according to any of Production Methods 1 to 5 to be described below. The bonding strength and thermal cycle properties of the obtained bonding bodies were evaluated. The results are shown in Table 1A to Table 2B.

[0107] [Production Method 1]

Using the bonding pastes obtained in Examples 1 to 39 and Comparative Examples 1 to 9, bonding bodies were produced according to the following procedure.

The bonding pastes obtained in Examples 1 to 36 and Comparative Examples 1 to 9 were respectively printed once under printing conditions to be described below on a bonded part 1 (copper substrate (non-plated): 20 mm × 20 mm × 3 mm), then a bonded part 2 (gold-plated SiC element: 8 mm × 8 mm × 0.3 mm) was placed with the plated surface thereof facing the bonding paste surface, and non-pressurized bonding was performed under sintering conditions to be described below to obtain respective bonding bodies.

[Sintering conditions]

The placed laminate was put in a firing furnace under nitrogen atmosphere, was heated from 25°C to 80°C at 5°C/minute, and was preliminarily dried at 80°C for 90 minutes. Subsequently, the temperature was raised to 300°C at 8°C/minute, and after reaching 300°C, it was maintained at 300°C for 2 hours.

[0108] [Production Method 2]

Using the bonding paste obtained in Example 40, a bonding body was produced according to the following procedure.

The bonding paste obtained in Example 37 was printed once under printing conditions to be described below on a bonded part 1 (copper substrate (non-plated): 20 mm × 20 mm × 3 mm), then a bonded part 2 (gold-plated SiC element: 8 mm × 8 mm × 0.3 mm) was placed with the plated surface thereof facing the bonding paste surface, and non-pressurized bonding was performed under sintering conditions to be described below to obtain a bonding body.

[Sintering conditions]

The placed laminate was put in a firing furnace under nitrogen atmosphere, was heated from 25°C to 80°C at 5°C/minute, and was preliminarily dried at 80°C for 90 minutes. Subsequently, the temperature was raised to 300°C at 2°C/minute, and after reaching 300°C, it was maintained at 300°C for 2 hours.

[0109] [Production Method 3]

Using the bonding paste obtained in Example 41, a bonding body was produced according to the following procedure.

The bonding paste obtained in Example 38 was printed once under printing conditions to be described below on a bonded part 1 (copper substrate (non-plated): 20 mm × 20 mm × 3 mm), then a bonded part 2 (gold-plated SiC element: 8 mm × 8 mm × 0.3 mm) was placed with the plated surface thereof facing the bonding paste surface, and non-pressurized bonding was performed under sintering conditions to be described below to obtain a bonding body.

[Sintering conditions]

The placed laminate was put in a firing furnace under nitrogen atmosphere, was heated from 25°C to 300°C at 8°C/minute, and after reaching 300°C, it was maintained at 300°C for 2 hours.

[0110] [Production Method 4]

Using the bonding paste obtained in Example 42, a bonding body was produced according to the following procedure.

The bonding paste obtained in Example 39 was printed once under printing conditions to be described below on a bonded part 1 (copper substrate (non-plated): 20 mm × 20 mm × 3 mm), then a bonded part 2 (gold-plated SiC element: 8 mm × 8 mm × 0.3 mm) was placed with the plated surface thereof facing the bonding paste surface, and non-pressurized bonding was performed under sintering conditions to be described below to obtain a bonding body.

[Sintering conditions]

The placed laminate was put in a firing furnace under nitrogen atmosphere, was heated from 25°C to 300°C at 2°C/minute, and after reaching 300°C, it was maintained at 300°C for 2 hours.

[0111] [Production Method 5]

Using the bonding paste obtained in Example 43, a bonding body was produced according to the following procedure.

The bonding paste obtained in Example 40 was printed once under printing conditions to be described below on a bonded part 1 (copper substrate (non-plated): 20 mm × 20 mm × 3 mm), then was put in a hot air oven and preliminarily dried at 180°C for 10 minutes. Subsequently, a bonded part 2 (gold-plated SiC element: 8 mm × 8 mm × 0.3 mm) was placed with the plated surface thereof facing the bonding paste surface after preliminary drying, and pressurized bonding was performed under sintering conditions to be described below to obtain a bonding body.

[Sintering conditions]

Under nitrogen atmosphere, while performing pressurization at a pressure of 30 MPa from above the bonded part 2, the temperature was raised from room temperature to 300°C at 20°C/minute, and after reaching 300°C, it was maintained at the same temperature for 5 minutes.

[0112] [Printing conditions (metal mask printing)]

Metal mask: opening 7.5 mm square, plate thickness 100 μm (manufactured by Seria Corporation)

Metal squeegee: 40 mm × 250 mm, thickness 1 mm (manufactured by Seria Corporation)

[0113] [Bonding strength]

For the obtained bonding body, the bonding body was fixed at the portion of the bonded part 1, a position 100 μm high from the interface between the bonded part 1 and the bonding layer toward the bonded part 2 was pushed at a speed of 500 μm/s, and a bonding strength (die shear

strength) at which the bonding was destroyed was obtained and evaluated based on the following evaluation criteria. Higher numerical values of the adhesion strength indicate better results, and 10 MPa or more is within the range of practical use. The measurement conditions are shown below.

[Measurement conditions]

Measuring apparatus: Universal bond tester (manufactured by Dage Japan Co., Ltd., 4000 Series)

Measurement height: 100 μm

Measurement speed: 500 μm/s

[0114] (Evaluation criteria)

S: 35 MPa or more

A+: 30 MPa or more and less than 35 MPa

A: 25 MPa or more and less than 30 MPa

A-: 20 MPa or more and less than 25 MPa

B: 15 MPa or more and less than 20 MPa

C: 10 MPa or more and less than 15 MPa

D: Less than 10 MPa

[0115] [Thermal cycle properties]

A cycle test described below was performed using the obtained bonding body, and a bonding strength (die shear strength) was measured and evaluated in the same manner as the [bonding strength] described above using the bonding body after the cycle test.

[Cycle test]

A process of holding the bonding body at -40°C for 30 minutes and then holding at 150°C for 30 minutes was taken as one cycle, and 500 cycles were performed.

[0116] [Table 1A]

Table 1A.

			Tempera-ture *1	Example													
				1	2	3	4	5	6	7	8	9	10	11	12	13	14
Silver particle (A)	A1	d50=210nm	216	90	90	90	90	90	90	90	90	90	90	90	90	90	90
	A2	d50=120nm	193														
	A3	d50=155nm	200														
	A4	d50=185nm	210														
	A5	d50=290nm	235														
	A6	d50=390nm	240														
Compound (B)	B1	Number of hydroxyl groups: 5, number of tertiary nitrogen atoms: 1	245	0.2						0.1							
	B2	Number of hydroxyl groups: 4, number of tertiary nitrogen atoms: 2 (liquid at 25°C)	248		0.2					0.1	0.1						
	B3	Number of hydroxyl groups: 5, number of tertiary nitrogen atoms: 3 (liquid at 25°C)	263			0.2											
	B4	Number of hydroxyl groups: 4, number of tertiary nitrogen atoms: 2 (liquid at 25°C)	217				0.2				0.1	0.03	0.05	0.1	0.4	0.9	1.1
	B5	Number of hydroxyl groups: 6, number of secondary nitrogen atoms: 2	257					0.2									
	B6	Number of hydroxyl groups: 4, number of tertiary nitrogen atoms: 2 (amide)	294						0.2								
Dispersion medium (C)	C1	Diethylene glycol monohexyl ether (b.p.255°C)	114	10	10	10	10	10	10	10	10	10	10	10	10	10	10
	C2	Triethylene glycol monobutyl ether (b.p.278°C)	128														
	C3	Triethylene glycol monomethyl ether (b.p.248°C)	108														
	C4	2-Ethyl-1,3-hexanediol (b.p. 244°C)	110														
	C5	Diethylene glycol monomethyl ether (b.p.193°C)	91														
	C6	1-Decanol (b.p.233°C)	104														
	C7	Terusolve TOE-100 (b.p.260°C)	118														
Other component	D1	Dimer acid		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
	D2	Trimer acid		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
	D3	Dimer diol															
	D4	Dimer diamine															
	E1	Oleic acid															
Production method	1	Non-pressurized (with preliminary drying, temp. increase rate 8°C/min)		○	○	○	○	○	○	○	○	○	○	○	○	○	○
Sintering temperature T1°C of metal particle (A)				216	216	216	216	216	216	216	216	216	216	216	216	216	216
50% weight reduction temperature T2°C of compound (B)				245	248	263	217	257	294	247	232	217	217	217	217	217	217
50% weight reduction temperature T3°C of dispersion medium (C)				114	114	114	114	114	114	114	114	114	114	114	114	114	114
T1-T3				102	102	102	102	102	102	102	102	102	102	102	102	102	102
(T2+10)-T1				39	42	57	11	51	88	41	26	11	11	11	11	11	11
Content ratio% of metal particle with respect to mass of bonding paste				89.6	89.6	89.6	89.6	89.6	89.6	89.6	89.6	89.8	89.8	89.7	89.5	89.0	88.8
Content ratio% of compound (B) with respect to mass of metal particle				0.22	0.22	0.22	0.22	0.22	0.22	0.22	0.22	0.03	0.06	0.11	0.44	1.00	1.22
Evaluation result	Bonding strength			A+	S	A+	S	A-	B	A+	S	A-	A+	S	S	A+	A
	Thermal cycle properties			A	S	A+	S	A-	B	A	S	B	A+	A+	S	A-	B

*1: Sintering temperature or decomposition temperature (°C)

[0117] [Table 1B]

Table 1B.

			Tempera-ture *1	Example														
				15	16	17	18	19	20	21	22	23	24	25	26	27	28	29
Silver particle (A)	A1	d50=210nm	216	90	90	90	90	90	90	90	90							
	A2	d50=120nm	193									90						
	A3	d50=155nm	200										90					
	A4	d50=185nm	210											90				
	A5	d50=290nm	235												90	90		
	A6	d50=390nm	240														90	90
Compound (B)	B1	Number of hydroxyl groups: 5, number of tertiary nitrogen atoms: 1	245															
	B2	Number of hydroxyl groups: 4, number of tertiary nitrogen atoms: 2 (liquid at 25°C)	248	0.2						0.2					0.2		0.2	
	B3	Number of hydroxyl groups: 5, number of tertiary nitrogen atoms: 3 (liquid at 25°C)	263															
	B4	Number of hydroxyl groups: 4, number of tertiary nitrogen atoms: 2 (liquid at 25°C)	217		0.2	0.2	0.2	0.2	0.2		0.2	0.2	0.2	0.2		0.2		0.2
	B5	Number of hydroxyl groups: 6, number of secondary nitrogen atoms: 2	257															
	B6	Number of hydroxyl groups: 4, number of tertiary nitrogen atoms: 2 (amide)	294															
Dispersion medium (C)	C1	Diethylene glycol monohexyl ether (b.p.255°C)	114	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10
	C2	Triethylene glycol monobutyl ether (b.p.278°C)	128															
	C3	Triethylene glycol monomethyl ether (b.p.248°C)	108															
	C4	2-Ethyl-1,3-hexanediol (b.p. 244°C)	110															
	C5	Diethylene glycol monomethyl ether (b.p.193°C)	91															
	C6	1-Decanol (b.p.233°C)	104															
	C7	Terusolve TOE-100 (b.p.260°C)	118															
Other component	D1	Dimer acid				0.2						0.1	0.1	0.1	0.1	0.1	0.1	0.1
	D2	Trimer acid					0.2					0.1	0.1	0.1	0.1	0.1	0.1	0.1
	D3	Dimer diol						0.2										
	D4	Dimer diamine							0.2									
	E1	Oleic acid								0.5	0.5							
Production method	1	Non-pressurized (with preliminary drying, temp. increase rate 8°C/min)		○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
Sintering temperature T1°C of metal particle (A)				216	216	216	216	216	216	216	216	193	200	210	235	235	240	240
50% weight reduction temperature T2°C of compound (B)				248	217	217	217	217	217	248	217	217	217	217	248	217	248	217
50% weight reduction temperature T3°C of dispersion medium (C)				114	114	114	114	114	114	114	114	114	114	114	114	114	114	114
T1-T3				102	102	102	102	102	102	102	102	79	86	96	121	121	126	126
(T2+10)-T1				42	11	11	11	11	11	42	11	34	27	17	23	-8	18	-13
Content ratio% of metal particle with respect to mass of bonding paste				89.8	89.8	89.6	89.6	89.6	89.6	89.8	89.8	89.6	89.6	89.6	89.6	89.6	89.6	89.6
Content ratio% of compound (B) with respect to mass of metal particle				0.22	0.22	0.22	0.22	0.22	0.22	0.22	0.22	0.22	0.22	0.22	0.22	0.22	0.22	0.22
Evaluation result	Bonding strength			A-	A-	A	A+	A+	A+	S	S	B	A-	A+	A+	A-	A-	C
	Thermal cycle properties			A-	A-	A	A+	A-	A-	S	S	C	B	A+	A+	B	B	C

*1: Sintering temperature or decomposition temperature (°C)

[0118] [Table 2A]

Table 2A.

			Tempera-ture *1	Example													
				30	31	32	33	34	35	36	37	38	39	40	41	42	43
Silver particle (A)	A1	d50=210nm	216	90	90	90	90	90	90	79	83	94	96	90	90	90	90
	A2	d50=120nm	193														
	A3	d50=155nm	200														
	A4	d50=185nm	210														
	A5	d50=290nm	235														
	A6	d50=390nm	240														
Compound (B)	B1	Number of hydroxyl groups: 5, number of tertiary nitrogen atoms: 1	245														
	B2	Number of hydroxyl groups: 4, number of tertiary nitrogen atoms: 2 (liquid at 25°C)	248														
	B3	Number of hydroxyl groups: 5, number of tertiary nitrogen atoms: 3 (liquid at 25°C)	263														
	B4	Number of hydroxyl groups: 4, number of tertiary nitrogen atoms: 2 (liquid at 25°C)	217	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Dispersion medium (C)	C1	Diethylene glycol monohexyl ether (b.p.255°C)	114							21	17	6	4	10	10	10	10
	C2	Triethylene glycol monobutyl ether (b.p.278°C)	128	10													
	C3	Triethylene glycol monomethyl ether (b.p.248°C)	108		10												
	C4	2-Ethyl-1,3-hexanediol (b.p. 244°C)	110			10											
	C5	Diethylene glycol monomethyl ether (b.p.193°C)	91				10										
	C6	1-Decanol (b.p.233°C)	104					10									
	C7	Terusolve TOE-100 (b.p.260°C)	118						10								
Other component	D1	Dimer acid		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
	D2	Trimer acid		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Production method	1	Non-pressurized (with preliminary drying, temp. increase rate 8°C/min)		○	○	○	○	○	○	○	○	○	○				
	2	Non-pressurized (with preliminary drying, temp. increase rate 2°C/min)												○			
	3	Non-pressurized (without preliminary drying, temp. increase rate 8°C/min)													○		
	4	Non-pressurized (without preliminary drying, temp. increase rate 2°C/min)														○	
	5	Pressurized															○
Sintering temperature T1°C of metal particle (A)				216	216	216	216	216	216	216	216	216	216	216	216	216	216
50% weight reduction temperature T2°C of compound (B)				217	217	217	217	217	217	217	217	217	217	217	217	217	217
50% weight reduction temperature T3°C of dispersion medium (C)				128	108	110	91	104	118	114	114	114	114	114	114	114	114
T1-T3				88	108	106	125	112	98	102	102	102	102	102	102	102	102
(T2+10)-T1				11	11	11	11	11	11	11	11	11	11	11	11	11	11
Content ratio% of metal particle with respect to mass of bonding paste				89.6	89.6	89.6	89.6	89.6	89.6	78.7	82.7	93.6	95.6	89.6	89.6	89.6	89.6
Content ratio% of compound (B) with respect to mass of metal particle				0.22	0.22	0.22	0.22	0.22	0.22	0.25	0.24	0.21	0.21	0.22	0.22	0.22	0.22
Evaluation result	Bonding strength			S	A+	A-	C	C	A+	B	A+	A+	B	A	A-	C	S
	Thermal cycle properties			S	A-	B	C	C	A+	B	A+	A+	B	A	C	C	A+

*1: Sintering temperature or decomposition temperature (°C)

[0119] [Table 2B]

Table 2B.

			Tempera-ture *1	Comparative Example								
				1	2	3	4	5	6	7	8	9
Silver particle (A)	A1	d50=210nm	216					90	90	90	90	90
	A2	d50=120nm	193									
	A3	d50=155nm	200									
	A4	d50=185nm	210									
	A5	d50=290nm	235									
	A6	d50=390nm	240									
Other silver particle	A10	d50=600nm	250	90								
	A11	d50=1100nm	285		90							
	A12	d50=20nm	180			90						
	A13	d50=85nm	185				90					
Compound (B)	B1	Number of hydroxyl groups: 5, number of tertiary nitrogen atoms: 1	245									
	B2	Number of hydroxyl groups: 4, number of tertiary nitrogen atoms: 2 (liquid at 25°C)	248	0.2		0.2						
	B3	Number of hydroxyl groups: 5, number of tertiary nitrogen atoms: 3 (liquid at 25°C)	263									
	B4	Number of hydroxyl groups: 4, number of tertiary nitrogen atoms: 2 (liquid at 25°C)	217		0.2		0.2					
Other compound	B11	Number of hydroxyl groups: 3, number of tertiary nitrogen atoms: 1	187					0.2				
	B12	Number of hydroxyl groups: 3, number of primary nitrogen atoms: 1	201						0.2			
	B13	Number of hydroxyl groups: 1, number of primary nitrogen atoms: 1	101							0.2		
	B14	Number of hydroxyl groups: 5, without nitrogen atom	244								0.2	
	B15	Without hydroxyl group, nitrogen atom conjugated system	132									0.2
Dispersion medium (C)	C1	Diethylene glycol monohexyl ether (b.p.255°C)	114	10	10			10	10	10		
	C2	Triethylene glycol monobutyl ether (b.p.278°C)	128			10	10				10	10
Other component	D1	Dimer acid		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
	D2	Trimer acid		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Production method	1	Non-pressurized (with preliminary drying, temp. increase rate 8°C/min)		○	○	○	○	○	○	○	○	○
Sintering temperature T1°C of metal particle (A)				250	285	180	185	216	216	216	216	216
50% weight reduction temperature T2°C of compound (B)				248	217	248	217	187	201	101	244	132
50% weight reduction temperature T3°C of dispersion medium (C)				114	114	128	128	114	114	114	128	128
T1-T3				136	171	52	57	102	102	102	88	88
(T2+10)-T1				8	-58	78	42	-19	-5	-105	38	-74
Content ratio% of metal particle with respect to mass of bonding paste				89.6	89.6	89.6	89.6	89.6	89.6	89.6	89.6	89.6
Content ratio% of compound (B) with respect to mass of metal particle				0.22	0.22	0.22	0.22	0.00	0.00	0.00	0.00	0.00
Evaluation result	Bonding strength			D	D	D	C	C	D	D	D	D
	Thermal cycle properties			D	D	D	D	D	D	D	D	D

*1: Sintering temperature or decomposition temperature (°C)

[0120] As shown in the results of Table 1A to Table 2B, it has been confirmed that, upon using the bonding paste of the disclosure combining the specific silver particle (A) and the specific compound (B), the bonding strength is very high, and even after performing the thermal cycle test, a decrease in bonding strength is suppressed.

In particular, with the bonding paste satisfying $T3 < T1 < (T2 + 10)$, even in the case of configurations in which stable bonding is extremely difficult, such as a non-plated substrate and a large-area SiC element, the bonding strength was very high, and even after performing the thermal cycle test, a decrease in bonding strength was suppressed (Example 26 and Example 27, and Example 28 and Example 29).

In addition, with the bonding paste containing the compound having a number of tertiary nitrogen atoms of 2 to 3, even in the case of configurations in which stable bonding is extremely difficult, such as a non-plated substrate and a large-area SiC element, the bonding strength was very high, and even after performing the thermal cycle test, a decrease in bonding strength was suppressed (Example 1 and Example 2).

In addition, with the bonding paste containing the dispersion medium having a boiling point of 250°C or higher, even in the case of configurations in which stable bonding is extremely difficult, such as a non-plated substrate and a large-area SiC element, the bonding strength was very high, and even after performing the thermal cycle test, a decrease in bonding strength was suppressed (Examples 4, 30, and 35 and Examples 31 to 34).

In addition, with the bonding paste containing the compound (D) with a carbon number of 20 to 80 having, in a quantity of two or more, at least one functional group selected from the group consisting of a hydroxyl group, a carboxy group, and an amino group, even in the case of configurations in which stable bonding is extremely difficult, such as a non-plated substrate and a large-area SiC element, the bonding strength was very high, and even after performing the thermal cycle test, a decrease in bonding strength was suppressed (Example 2 and Example 15, and

Example 4 and Example 16).

In addition, with the bonding paste containing the compound (E) with a carbon number of 14 to 20 having one carboxy group, even in cases of configurations in which stable bonding is extremely difficult, such as a non-plated substrate and a large-area SiC element, the bonding strength was very high, and even after performing the thermal cycle test, a decrease in bonding strength was suppressed (Example 15 and Example 21, and Example 16 and Example 22).

In addition, in the bonding body bonded using the non-pressurized bonding method including the preliminary drying process (2a) described above, even in the case of configurations in which stable bonding is extremely difficult, such as a non-plated substrate and a large-area SiC element, the bonding strength was very high, and even after performing the thermal cycle test, a decrease in bonding strength was suppressed (Example 4 and Example 41, and Example 40 and Example 42).

In contrast, in the case of using the bonding pastes of Comparative Examples, the bonding strength was significantly low, and the bonding strength after performing the thermal cycle test was also significantly low.

[0121] This application claims priority based on Japanese Patent Application No. 2022-183808 filed on November 17, 2022, the entire disclosure of which is incorporated herein by reference.

CLAIMS

[Claim 1] A bonding paste comprising:

a silver particle (A) having an average particle diameter of 100 nm to 500 nm;

a compound (B) having at least one nitrogen atom selected from the group consisting of a secondary nitrogen atom and a tertiary nitrogen atom and having a number of hydroxyl groups of 4 or more; and

a dispersion medium (C).

[Claim 2] The bonding paste according to claim 1, wherein in a case where a sintering temperature of the silver particle (A) is T1°C, a 50% weight reduction temperature of the compound (B) is T2°C, and a 50% weight reduction temperature of the dispersion medium (C) is T3°C, the bonding paste satisfies $T3 < T1 < (T2 + 10)$.

[Claim 3] The bonding paste according to claim 2, wherein the T2 is 200 to 300°C.

[Claim 4] The bonding paste according to any one of claims 1 to 3, wherein the compound (B) has a tertiary nitrogen atom.

[Claim 5] The bonding paste according to claim 4, wherein the number of tertiary nitrogen atoms in the compound (B) is 2 to 3.

[Claim 6] The bonding paste according to any one of claims 1 to 3, wherein the number of hydroxyl groups in the compound (B) is 4 to 6.

[Claim 7] The bonding paste according to any one of claims 1 to 3, wherein the compound (B) is liquid at 25°C.

[Claim 8] The bonding paste according to any one of claims 1 to 3, wherein a content ratio of the compound (B) is 0.05 to 1.0 mass% based on a mass of the silver particle (A).

[Claim 9] The bonding paste according to any one of claims 1 to 3, wherein the silver particle (A) has an average particle diameter of 150 nm to 400 nm.

[Claim 10] The bonding paste according to any one of claims 1 to 3, wherein the dispersion medium (C) has a boiling point of 250°C or higher.

[Claim 11] The bonding paste according to any one of claims 1 to 3, further comprising a compound (D) with a carbon number of 20 to 80 having, in a quantity of two or more, at least one functional group selected from the group consisting of a hydroxyl group, a carboxy group, and an amino group.

[Claim 12] The bonding paste according to any one of claims 1 to 3, further comprising a compound (E) with a carbon number of 14 to 20 having one carboxy group.

[Claim 13] The bonding paste according to any one of claims 1 to 3, wherein a content ratio of the silver particle (A) is 80 to 95 mass% based on a mass of the bonding paste.

[Claim 14] A bonding body obtained by bonding a first bonded part and a second bonded part with the bonding paste according to any one of claims 1 to 3.

[Claim 15] The bonding body according to claim 14, wherein the first bonded part is a non-plated substrate.

[Claim 16] The bonding body according to claim 14, wherein the second bonded part is SiC.

[Claim 17] A method for producing a bonding body, which is a method for producing a bonding body obtained by bonding a first bonded part and a second bonded part with the bonding paste according to any one of claims 1 to 3, the method for producing a bonding body comprising

applying the bonding paste to the first bonded part;

placing the second bonded part on an applied part of the first bonded part to which the bonding paste has been applied;

heating a placed laminate for preliminary drying; and

sintering the placed laminate under a non-pressurized environment.