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(54) Title:

WATER-SOLUBLE WORKING FLUID FOR FIXED-
ABRASIVE WIRE SAW

(57) Abstract:

PCT/JP2012/054115 ABSTRACT Provided is a water-soluble working fluid for a fixed-abrasive wire saw, the water-soluble working fluid comprising: (C) a carboxylic acid; (D) a compound which shows basicity on dissolution in water; and (E) water, wherein an electrical conductivity at 25°C of the water-soluble working fluid is 300 µS/cm or more and 3000 µS/cm or less; a pH at 25°C of the water-soluble working fluid is 5 or more and 10 or less; and a viscosity of a simulated fluid formed by adding 10 mass % of a silicon powder having an average particle size of 1.5 µm to the water-soluble working fluid and stirring a resultant mixture is less than 30 mPa.s at 25°C.

ABSTRACT

Provided is a water-soluble working fluid for a fixed-abrasive wire saw, the water-soluble working fluid comprising: (C) a carboxylic acid; (D) a compound which shows basicity on dissolution in water; and (E) water, wherein an electrical conductivity at 25°C of the water-soluble working fluid is 300 μ S/cm or more and 3000 μ S/cm or less; a pH at 25°C of the water-soluble working fluid is 5 or more and 10 or less; and a viscosity of a simulated fluid formed by adding 10 mass % of a silicon powder having an average particle size of 1.5 μ m to the water-soluble working fluid and stirring a resultant mixture is less than 30 mPa·s at 25°C.

DESCRIPTION

WATER-SOLUBLE WORKING FLUID FOR FIXED-ABRASIVE WIRE SAW

Technical Field

[0001] The present invention relates to a water-soluble working fluid for a fixed-abrasive wire saw.

Background Art

[0002] In recent years, the diameter of silicon wafers has been increased rapidly to reduce the manufacturing cost of semiconductor devices, and it has become important to improve the cutting technique of the silicon wafers. As an example of the cutting technique, there is a cutting method using a wire tool (wire saw), which causes less cutting loss and exhibits good processing efficiency. There are two types of cutting method, which are a loose abrasive method and a fixed abrasive method. As for the loose abrasive method, problems such as the followings have been pointed out: (1) since a slurry having abrasive grains dispersed in a fluid is used as a working fluid, a cut workpiece and the ambient environment are severely contaminated; (2) it is necessary to dispose of the slurry containing large amounts of cut debris that are produced during a cutting process; (3) it is difficult to separate the loose abrasive grains from the cut debris; and (4) a running speed of a wire is limited, causing limitation to improving the processing efficiency. In order to solve such problems, studies have been conducted on a fixed abrasive wire saw which has abrasive grains fixed onto a surface

of a wire such as a piano wire by electrodeposition, resin bond, or some other means. For example, Patent Document 1 discloses a fixed abrasive wire saw having abrasive grains of 30 to 60 μm fixed by resin bond. The fixed abrasive wire saw is moved at a speed of 1000 to 2500 m/min to cut a brittle material, thereby enabling highly efficient cut processing.

[0003] On the other hand, when cutting a brittle material by using a fixed abrasive wire saw, a working fluid is used together for the purpose of lubrication, cooling, and dispersion of cut debris. The working fluid to be used is preferably soluble in water. A non-aqueous working fluid containing almost no water has such drawbacks that: a non-aqueous volatile component (a solvent component) in the non-aqueous working fluid deteriorates working conditions; the working fluid becomes flammable; and a washing step, waste disposal, etc. after the processing become more complicated than in the case of using a water-soluble working fluid.

[0004] In addition, if silicon is to be cut, the following problem is likely to occur: since silicon is highly reactive, it reacts with water or an alkali during cut processing to produce hydrogen gas and cause fire. Therefore, it is preferable to use a working fluid which is inhibited from reacting with silicon. In this viewpoint, such a working fluid as described in Patent Document 2 is suggested.

Citation List

Patent Literature

[0005]

Patent Document 1: Japanese Patent Application Laid-Open (JP-A)
No. 2001-054850

Patent Document 2: JP-A No. 2003-082334

Summary of the Invention

Problems to be Solved by the Invention

[0006] A working fluid mainly composed of a glycol and water as described in Patent Document 2 is widely used when cutting a brittle material such as silicon, sapphire, glass, ceramics, and neodymium with a fixed abrasive wire. However, such a conventional working fluid sometimes hinders sufficiently stable cut processing. In particular, it is found that inclusion of cut debris into the working fluid causes various problems. When the working fluid is used in cutting a workpiece such as silicon, cut debris of the workpiece go into the working fluid, and in some cases the viscosity of the working fluid increases to the extent that affects the processing. In addition, when the cut debris are mixed in the working fluid, problems sometimes also occur that a pipe of equipment gets clogged with the cut debris, a hard deposit (hereinafter sometimes referred to as a "hard cake") is created in a groove of a main roller onto which a wire is to be hooked, causing skipping of the wire (causing the wire to come off the groove), and so on.

[0007] Accordingly, an object of the present invention is to provide a water-soluble working fluid for a fixed abrasive wire saw which can be inhibited from increasing its viscosity when mixed with cut debris and can prevent clogging of a pipe of equipment and generation of a hard cake that are caused by

the cut debris.

Means for Solving the Problems

[0008] The inventors conducted an intensive study to solve the above problems, and found the followings:

(1) when the cut debris of a workpiece are mixed into a working fluid, the viscosity of the working fluid increases significantly because of the cut debris being fine powder (having a particle size of around 1 μm) or because the cut debris having a newly-formed surface actively act on each other to agglomerate containing the working fluid therearound and function as a viscosity improver;

(2) when the cut debris of a workpiece is mixed into a working fluid, since the specific gravity of the cut debris is larger than that of a composition constituting the working fluid, the cut debris having the larger specific gravity precipitates. At this time, if the cut debris precipitate uniformly in a dispersed state, the cut debris thus precipitated get solidified firmly and densely, and causes clogging of a pipe of equipment or generation of a hard cake; and

(3) the above problems (1) and (2) can be solved by properly adjusting the electrical conductivity of the working fluid to control the dispersion manner of the cut debris mixed in the working fluid.

[0009] The present invention has been made based on the above findings and has the following aspects.

A first aspect of the present invention is a water-soluble working fluid for a fixed-abrasive wire saw, the water-soluble

working fluid comprising: (C) a carboxylic acid; (D) a compound which shows basicity on dissolution in water; and (E) water, wherein an electrical conductivity at 25°C of the water-soluble working fluid is 300 μ S/cm or more and 3000 μ S/cm or less; a pH at 25°C of the water-soluble working fluid is 5 or more and 10 or less; and a viscosity of a simulated fluid formed by adding 10 mass % of a silicon powder having an average particle size of 1.5 μ m to the water-soluble working fluid and stirring a resultant mixture is less than 30 mPa·s at 25°C.

[0010] In the present invention, the "viscosity" refers to a viscosity measured by a Brookfield-type viscometer. The "average particle size" refers to an average particle size measured by using a laser diffraction/scattering type particle size distribution measuring device "LA910" manufactured by HORIBA.

[0011] The working fluid of the first aspect of the present invention preferably further comprises (A) at least one nonionic surfactant selected from copolymers of an alcohol, ethylene oxide, and propylene oxide, and polyoxyalkylene glycols.

[0012] The working fluid of the first aspect of the present invention preferably further comprises (B) a glycol.

[0013] A second aspect of the present invention is a water-soluble working fluid for a fixed-abrasive wire saw, the water-soluble working fluid comprising: (A) 0.1 mass % or more and 8 mass % or less of at least one nonionic surfactant selected from copolymers of an alcohol, ethylene oxide, and propylene oxide, and polyoxyalkylene glycols; (B) 0.1 mass % or more and 80 mass % or less of a glycol; (C) 0.01 mass % or more and 5 mass % or less

of a carboxylic acid; (D) 0.01 mass % or more and 7 mass % or less of a compound which shows basicity on dissolution in water; and (E) water, wherein an electrical conductivity at 25°C of the water-soluble working fluid is 300 μ S/cm or more and 3000 μ S/cm or less; a pH at 25°C of the water-soluble working fluid is 5 or more and 10 or less; and a viscosity of a simulated fluid formed by adding 10 mass % of a silicon powder having an average particle size of 1.5 μ m to the water-soluble working fluid and stirring a resultant mixture is less than 30 mPa·s at 25°C.

[0014] The working fluids of the first and the second aspects of the present invention preferably have a surface tension at 25°C of 20 mN/m or more and 50 mN/m or less.

[0015] The working fluids of the first and the second aspects of the present invention preferably further comprise a water-soluble polymer and/or a defoaming agent.

[0016] The working fluids of the first and the second aspects of the present invention preferably contain 10 mass % or more and 99.7 mass % or less of the (E) water, based on a total mass of the working fluid as 100 mass %.

Effects of the Invention

[0017] According to the present invention, it is possible to provide a water-soluble working fluid for a fixed abrasive wire saw which can be inhibited from increasing its viscosity when mixed with cut debris and can prevent clogging a pipe of equipment and generation of the hard cake that are caused by the cut debris.

Modes for Carrying Out the Invention

[0018] <Properties of the Working Fluid of the Present Invention>

(Electrical Conductivity)

The water-soluble working fluid for a fixed abrasive wire saw of the present invention has an electrical conductivity at 25 °C of 300 µS/cm or more and 3000 µS/cm or less. The upper limit of the electrical conductivity is preferably no more than 2000 µS/cm, and more preferably no more than 1000 µS/cm.

[0019] The electrical conductivity indicates how well electricity can be conducted, and is defined as the reciprocal of electrical resistivity. As the concentration of an ionic substance contained in the working fluid is higher, the electrical conductivity of the working fluid increases and better conducts electricity. Further, as the concentration of an ionic substance contained in the working fluid is higher, particulates (cut debris) having an electrical charge tend to agglomerate in the working fluid more easily. Namely, as the electrical conductivity of the working fluid increases, the apparent particle size of the cut debris mixed in the working fluid becomes larger and the cut debris precipitate more easily. At this time, since the cut debris precipitate in a sterically-bulky state, it is seen to be unlikely that a hard cake is generated. Further, the cut debris can precipitate faster when it is in the agglomerated state, and it can be easily separated from the working fluid by means of centrifugal separation or the like.

[0020] However, when the electrical conductivity is too high, it becomes difficult to disperse the cut debris in the working

fluid, thereby causing the viscosity of the working fluid to increase easily.

[0021] On the other hand, when the electrical conductivity is low (typically exemplified by pure water), the particulates (cut debris) having an electrical charge repel each other due to the electrical charge thereof and disperse. Therefore, even when the cut debris is mixed in, the viscosity of the working fluid is unlikely to increase.

[0022] However, when the electrical conductivity of the working fluid is too low and the cut debris are dispersed excessively in the working fluid, the cut debris precipitate uniformly in the dispersed state and be solidified firmly and densely. Due to the cut debris precipitated and solidified firmly and densely in this manner, problems occur easily such as clogging of a pipe of equipment and generation of a hard cake in a wire groove of a main roller resulting in skipping of the wire (causing the wire to come off the groove). In addition, when the cut debris are dispersed in the working fluid excessively, they precipitate slowly. Therefore, it is difficult to remove the cut debris in the working fluid by means of centrifugal separation or the like when actually trying to remove them for the purpose of waste disposal or recycling of the working fluid.

[0023] According to the working fluid of the present invention, by setting the electrical conductivity at 25°C of the working fluid in the range described above, it is possible to adequately disperse, agglomerate, and precipitate the cut debris in the working fluid. As a result, it is possible to inhibit increase in the viscosity of the working fluid, clogging of a

pipe of equipment, generation of a hard cake, and so on.

[0024] The electrical conductivity of the working fluid of the present invention can be adjusted by an amount of a substance ionized on dissolution in water added to the working fluid. For example, the electrical conductivity of the working fluid can be adjusted appropriately by adjusting the additive amount of the below described (C) component (a carboxylic acid) or the additive amount of the below described (D) component (a compound which shows basicity on dissolution in water), to the working fluid. The working fluid of the present invention may be diluted with water when used, but in this case as well, the electrical conductivity of the working fluid after dilution is preferably in the range given above.

[0025] (Viscosity)

A viscosity of the working fluid of the present invention is preferably 1 mPa·s or more and 25 mPa·s or less at 25°C. The upper limit of the viscosity is more preferably no more than 20 mPa·s. When the viscosity of the working fluid is too high, the amount of water in the working fluid is generally small and thus the ability of the working fluid to remove heat caused by processing using a fixed abrasive wire (hereinafter sometimes referred to as a "cooling ability") is weak. Therefore, such problems as degradation of processing precision and increase of load on a tool may arise. In addition, a viscosity of a fluid (simulated fluid) formed by dispersing a predetermined silicon powder in the working fluid of the present invention is preferably 1 mPa·s or more and less than 30 mPa·s at 25 °C, more preferably 1 mPa·s or more and 20 mPa·s or less at 25 °C, and still more

preferably 1 mPa·s or more and 15 mPa·s or less at 25 °C. The viscosity of the simulated fluid is based on measurement of a viscosity of a simulated fluid obtained by: adding 10 mass % of a silicon powder (particle size: 1.5 μm) to the working fluid of the present invention; mixing them by stirring; thereafter putting a stainless steel ball (2 mm in diameter) into a resultant mixture; stirring the mixture at 1000 rpm for 10 hours; and filtering off the stainless steel ball. If the viscosity of the working fluid described above is already too high, the viscosity of the simulated fluid containing the silicon powder will also be inevitably high. The viscosity of the working fluid and the simulated fluid can be measured by a Brookfield-type viscometer.

[0026] The viscosity of the working fluid of the present invention can be appropriately adjusted for example by the blending amount of the (A) component (a nonionic surfactant), the (B) component (a glycol), the (C) component (a carboxylic acid), the (D) component (a compound which shows basicity on dissolution in water), and the (E) component (water) that are described below. The viscosity of the working fluid of the present invention can also be adjusted by adding another additive (e.g. a viscosity modifier). The working fluid of the present invention may be diluted with water when used, but in this case as well the viscosity of the working fluid after dilution is preferably in the range given above.

[0027] (pH)

A pH of the working fluid of the present invention is 5 or more and 10 or less at 25°C. When the pH is less than 5, metal corrosivity of the working fluid becomes high, likely causing

corrosion of a metal component of equipment or a wire saw brought into contact with the working fluid. The metal corrosivity of the working fluid can be easily inhibited by setting the pH of the working fluid to be 5 or more at 25°C, though it depends on such factors as the amount of water contained in the working fluid. From this viewpoint, the pH of the working fluid of the present invention is preferably 6 or more at 25°C, and more preferably 7 or more at 25°C. On the other hand, if the pH of the working fluid is over 10, the reactivity of the working fluid with the cut debris (silicon) mixed in the working fluid will rise, thus likely causing generation of hydrogen by reaction of the working fluid with the cut debris, or likely causing significant increase in the viscosity of the working fluid during processing. By setting the pH of the working fluid at 10 or less at 25°C, it is possible to easily inhibit generation of hydrogen during processing and increase in the viscosity of the working fluid during processing.

[0028] The pH of the working fluid of the present invention can be appropriately adjusted by the blending amount of the (B) component (a glycol), the (C) component (a carboxylic acid), and the (D) component (a compound which shows basicity on dissolution in water) that are described below. The viscosity of the working fluid of the present invention can also be adjusted by adding another additive (e.g. a pH adjuster). The working fluid of the present invention may be diluted with water when used, but in this case as well the pH of the working fluid after dilution is preferably in the range described above.

[0029] (Surface Tension)

The inventors have found that the conventional working fluids have insufficient wettability on a wire used and insufficient permeability into a gap in a workpiece, and that the amount of the working fluids to reach a portion to be processed (i.e. a portion of the workpiece to be brought into contact with the wire) is insufficient, thus sometimes causing degradation of the cutting performance. When the surface tension of the working fluid is too high, the wettability and the permeability of the working fluid deteriorate, likely preventing the working fluid from reaching the portion to be processed. In an extreme case, the portion to be processed ends up being cut in a dry state. Therefore, heat is generated significantly in the portion to be processed, likely causing breaking of a wire due to an excessive load on a tool, or poor surface roughness of the workpiece due to degradation of the processing precision. In these viewpoints, the surface tension of the working fluid of the present invention is preferably 50 mN/m or less at 25°C, more preferably 45 mN/m or less at 25°C, and still more preferably 40 mN/m or less at 25°C. By setting the surface tension of the working fluid in the above range, it is possible to improve the wettability of the working fluid on the workpiece (silicon etc.) or the wire (nickel, resin, etc.) and to improve the permeability of the working fluid into the portion to be processed.

[0030] On the other hand, when the surface tension is too low, foaming of the working fluid is promoted. Therefore, a problem occurs that foam is introduced into the working fluid during the cutting process, resulting in degradation of the cooling ability of the working fluid. There is also a problem

that the foam generated from the working fluid overflows from a tank arranged for holding the working fluid. From these viewpoints, the surface tension of the working fluid of the present invention is preferably 20 mN/m or more at 25°C, and more preferably 30 mN/m or more at 25°C.

[0031] The surface tension of the working fluid of the present invention can be adequately adjusted for example by the blending amount of the (A) component or a defoaming agent described below. The working fluid of the present invention may be diluted with water when used, but in this case as well the surface tension of the working fluid after dilution is preferably in the range given above.

[0032] <Components Contained in the Working Fluid of the Present Invention>

The working fluid of the present invention may contain the components described below for example.

[0033] ((A) Component)

The working fluid of the present invention may contain at least one nonionic surfactant selected from copolymers of an alcohol, ethylene oxide, and propylene oxide, and polyoxyalkylene glycols. With the (A) component contained in the working fluid, the surface tension of the working fluid can be lowered, and the wettability and the permeability of the working fluid can be improved.

[0034] Although the alcohol mentioned above is not particularly limited, an alcohol having a carbon number of 8 to 12 can be employed for example.

[0035] Specific examples of the polyoxyalkylene glycols

mentioned above include: polyethylene glycol; polypropylene glycol; and a copolymer of polyoxyethylene and polyoxypropylene. A mass-average molecular weight of the polyoxyalkylene glycols (in terms of standard polystyrene by gel permeation chromatography) is preferably 10000 or less, more preferably 5000 or less, and still more preferably 3500 or less.

[0036] The working fluid of the present invention may be in the form of being diluted with water as necessary based on the working conditions of the cutting process. That is, the working fluid of the present invention may be made by preparing a concentrated composition formed of components in the working fluid of the present invention other than water and diluting the concentrated composition with water at a work site or the like. The working fluid of the present invention may be made in a highly concentrated form (that is, the working fluid of the present invention may be made using a small amount of water) to be used as it is or to be further diluted with water, depending on the working conditions of the cut processing. When actually using the working fluid of the present invention in cut processing, the lower limit of the content of the (A) component is preferably no less than 0.1 mass %, more preferably no less than 0.2 mass %, and still more preferably no less than 0.5 mass %, based on the total mass (100 mass%) of the working fluid of the present invention. The upper limit thereof is preferably no more than 0.8 mass %, more preferably no more than 0.7 mass %, and still more preferably no more than 0.6 mass %, based on the total mass (100 mass%) of the working fluid of the present invention. Herein, in the case of preparing the highly concentrated working fluid

of the present invention as described above, the upper limit of the content of the (A) component is preferably no more than 8 mass %, more preferably no more than 7 mass %, and still more preferably no more than 6 mass %, based on the total mass (100 mass%) of the working fluid of the present invention.

[0037] (B) Component)

The working fluid of the present invention may further contain a glycol as the (B) component. With a predetermined amount of the (B) component contained in the working fluid of the present invention, components other than the (B) component can be dissolved in the working fluid of the present invention stably and drying of the working fluid of the present invention can be inhibited.

[0038] Specific examples of the glycol as the (B) component are water-soluble glycols such as: ethylene glycol; propylene glycol; 1,4-Butanediol; hexamethylene glycol; neopentyl glycol; diethylene glycol; triethylene glycol; dipropylene glycol; tripropylene glycol; polyethylene glycol; polypropylene glycol; glycols of a copolymer of ethylene glycol and propylene glycol, a copolymer of ethylene oxide and propylene oxide, etc.; glycol monoalkyl ethers such as triethylene glycol monobutyl ether, triethylene glycol monomethyl ether, diethylene glycol monobutyl ether, and tripropylene glycol monomethyl ether; and a monoalkyl ether of a copolymer of ethylene oxide and propylene oxide. Among the above examples of the glycols, propylene glycol, dipropylene glycol, diethylene glycol, triethylene glycol, polyethylene glycol, and polypropylene glycol are preferred; and dipropylene glycol and diethylene glycol are especially preferred. They may

be used alone or in combination. A copolymer formed of two or more of the above may also be used. A mass-average molecular weight of the above glycols (in terms of standard polystyrene by gel permeation chromatography) is preferably 100 or more and 700 or less, and more preferably 100 or more and 200 or less.

[0039] When actually using the working fluid of the present invention in cut processing, the lower limit of the content of the (B) component is preferably no less than 0.1 mass %, more preferably no less than 0.5 mass %, and still more preferably no less than 2 mass %, based on the total mass (100 mass%) of the working fluid of the present invention. The upper limit thereof is preferably no more than 8 mass %, and more preferably no more than 7 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. Herein, in the case of preparing the highly concentrated working fluid of the present invention as described above, the upper limit of the content of the (B) component is preferably no more than 80 mass %, and more preferably no more than 70 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. By setting the content of the (B) component in the above range, it is possible to dissolve components other than the (B) component into the working fluid of the present invention stably and possible to inhibit drying of the working fluid of the present invention.

[0040] ((C) Component)

The working fluid of the present invention may also contain a carboxylic acid as the (C) component. Including a predetermined amount of the (C) component in the working fluid of the present invention enables, in combination with the (D)

component described below, such advantageous effects that: the pH and the electrical conductivity of the working fluid of the present invention can be adjusted; fluctuation of the pH of the working fluid of the present invention during cut processing can be reduced; metal corrosivity of the working fluid can be reduced; and components other than the (C) component can be dissolved into the working fluid of the present invention stably.

[0041] Examples of the carboxylic acid as the (C) component include citric acid, succinic acid, lactic acid, malic acid, adipic acid, oxalic acid, dodecanedioic acid, and acetic acid. Among them, citric acid and succinic acid are preferred, and citric acid is more preferred. They may be used alone or in combination.

[0042] When actually using the working fluid of the present invention in cut processing, the lower limit of the content of the (C) component is preferably no less than 0.01 mass %, and more preferably no less than 0.1 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. The upper limit thereof is preferably no more than 0.5 mass %, more preferably no more than 0.3 mass %, and still more preferably no more than 0.2 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. Herein, in the case of preparing the highly concentrated working fluid of the present invention as described above, the upper limit of the content of the (C) component is preferably no more than 5 mass %, more preferably no more than 3 mass %, and still more preferably no more than 2 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. By setting the

content of the (C) component in the above range, the above described advantageous effects enabled by inclusion of the (C) component can be realized. When the content of the (C) component is too small, the above advantageous effects may not be exhibited sufficiently. When the content of the (C) component is too large, the amount of the (D) component used to neutralize the (C) component increases, and therefore unintended increase in the electrical conductivity is caused and metal corrosion is caused easily.

[0043] (D) Compound Which Shows Basicity on Dissolution in Water

The working fluid of the present invention also contains, as the (D) component, a compound which shows basicity on dissolution in water (hereinafter sometimes referred to as a "basic compound"). Including a predetermined amount of the (D) component in the working fluid of the present invention enables, in combination with the (C) component described above, such advantageous effects that: the pH and the electrical conductivity of the working fluid of the present invention can be adjusted; fluctuation of the pH of the working fluid of the present invention during cut processing can be reduced; metal corrosivity of the working fluid can be reduced; and other components can be dissolved into the working fluid of the present invention stably.

[0044] Specific examples of the basic compound as the (D) component include: compounds containing alkali metal elements such as sodium hydroxide, potassium hydroxide, sodium carbonate, potassium carbonate, sodium hydrogen carbonate, and potassium hydrogen carbonate; and amines such as triethanolamine,

triisopropanolamine, ethylenediamine, N-(2-aminoethyl)-2-aminoethanol, and N-(β -aminoethyl)ethanol amine. They may be used alone or in combination.

[0045] When actually using the working fluid of the present invention in cut processing, the lower limit of the content of the (D) component is preferably no less than 0.01 mass %, and more preferably no less than 0.1 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. The upper limit thereof is preferably no more than 0.7 mass %, more preferably no more than 0.6 mass %, and still more preferably no more than 0.5 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. Herein, in the case of preparing the highly concentrated working fluid of the present invention as described above, the upper limit of the content of the (D) component is preferably no more than 7 mass %, more preferably no more than 6 mass %, and still more preferably no more than 5 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. By setting the content of the (D) component in the above range, the above described advantageous effects enabled by inclusion of the (D) component can be realized. When the content of the (D) component is too large, the amount of the (C) component used to neutralize the (D) component increases, thus causing not only unintended increase in the electrical conductivity but also deterioration of dispersion of the cut debris.

[0046] The working fluid of the present invention may contain a salt formed from the (C) component and the (D) component, instead of the (C) component and the (D) component described above.

The salt is preferably contained in the amount described above in terms of the (C) component and the (D) component. Specific examples of the salt include alkali metal salts of carboxylic acids and amine salts of carboxylic acids.

[0047] ((E) Component)

The working fluid of the present invention contains water as the (E) component. The kind of water is not particularly limited and may be distilled water, tap water, etc. In a case of using water with high electrical conductivity, for example water with high hardness, it is preferable to adjust the amount of ion contained. For example, a concentration of ion (hardness) in tap water varies among areas or countries.

[0048] When a cutting process is performed using a wire having fixed abrasive grains, a large amount of heat is generated during cut processing compared with the cutting process by the conventional loose abrasive method. Thus, a large load is imposed on the wire or a workpiece. Further, it is known that when the workpiece expands due to the heat generation, cutting precision is negatively affected. It is also known that diamond abrasive grains fixed onto the wire tend to wear away easily and that the life of the tool is negatively affected. Therefore, the working fluid used during the processing is required to have a cooling ability. In order to improve the cooling ability of the working fluid, it is necessary to increase the amount of water contained therein. However, when the workpiece is silicon, the water in the working fluid and the cut debris may react with each other to generate hydrogen gas. Since the amount of water in the conventional aqueous working fluid cannot be increased easily,

the problem of the cooling ability thereof has not yet been solved fundamentally. When hydrogen gas is generated by reaction of the cut debris mixed in the working fluid with the water in the working fluid, the working fluid takes in bubbles at a time of being supplied by a pump. As such, the amount of the working fluid supplied becomes unstable or the specific weight of liquid decreases due to the bubbles taken in, therefore leading to such problems that a wire machine which controls the working fluid by the specific weight of liquid detects the abnormality and stops the processing. There is also a risk that the hydrogen gas generated explodes due to static electricity.

[0049] According to the working fluid of the present invention, it is possible to inhibit reaction between the working fluid and the cut debris by including each of the components described above and also setting the pH at the predetermined value mentioned above. Therefore, the amount of water contained in the working fluid can be increased to improve the cooling ability of the working fluid. That is, according to the working fluid of the present invention, it is possible to attain both inhibition of the hydrogen generation and improvement of the cooling ability.

[0050] Increasing the amount of water in the working fluid is likely to easily cause corrosion of a metal component of equipment or a wire brought into contact with the working fluid. However, according to the working fluid of the present invention, the corrosion can be inhibited by including a predetermined amount of the (B) component and the (C) component, or adjusting the pH of the working fluid in the predetermined range given above. Therefore, the amount of water contained in the working fluid

can be increased to improve the cooling ability of the working fluid. That is, according to the working fluid of the present invention, it is possible to attain both inhibition of the corrosion and improvement of the cooling ability.

[0051] The lower limit of the content of the water is preferably no less than 10 mass %, more preferably no less than 25 mass %, and still more preferably no less than 50 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. By setting the content of the water to be 10 mass % or more, it is possible to easily ensure the cooling ability of the working fluid, prevent degradation of the processing precision, and reduce a load on a tool. On the other hand, the upper limit thereof is preferably no more than 99.7 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. When the amount of the water is 100 %, that is, in a case of pure water, it is unsuitable as a working fluid in view of the wettability and the permeability. However, it is seen that when other components specified in the present invention are contained even in a very small amount, at least some advantageous effects can be attained. In addition, the cooling ability of the working fluid is better as the amount of water contained in the working fluid is larger.

[0052] (Water-Soluble Polymer)

The working fluid of the present invention may contain a water-soluble polymer. Dispersibility of the cut debris in the working fluid of the present invention can be inhibited by setting the electrical conductivity of the working fluid at a predetermined value as described above. Including the

water-soluble polymer in the working fluid of the present invention makes it easier to control the dispersibility of the cut debris mixed in the working fluid.

[0053] The water-soluble polymer that can be used in the present invention is not particularly limited and may be selected from those used in the conventional working fluids. Examples thereof include polyvinylpyrrolidone and a copolymer having a structural unit derived from vinylpyrrolidone. The content of the water-soluble polymer may be adequately determined within the range that can realize the above advantageous effect enabled by inclusion of the water-soluble polymer, does not hinder the advantageous effects of other components, and does not cause negative influence on the working fluid.

[0054] (Defoaming Agent)

The working fluid of the present invention may contain a defoaming agent. By including the defoaming agent in the working fluid of the present invention, it is possible to reduce the foam generated in the working fluid.

[0055] A known defoaming agent may be used without particular limitations. However, it is preferable to employ a defoaming agent that can be dispersed in the working fluid stably.

[0056] When actually using the working fluid of the present invention in cut processing, the lower limit of the content of the defoaming agent is preferably no less than 0.01 mass %, more preferably no less than 0.02 mass %, and still more preferably no less than 0.03 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. The upper limit thereof is preferably no more than 0.06 mass %, more preferably

no more than 0.05 mass %, and still more preferably no more than 0.04 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. Herein, in the case of preparing the highly concentrated working fluid of the present invention as described above, the upper limit of the content of the defoaming agent is preferably no more than 0.6 mass %, more preferably no more than 0.5 mass %, and still more preferably no more than 0.4 mass %, based on the total mass (100 mass %) of the working fluid of the present invention. By setting the content of the defoaming agent in the above range, the above advantageous effect enabled by inclusion of the defoaming agent can be realized. If the content of the defoaming agent is too large, degradation of stability of the working fluid, such as separation of the working fluid, is likely to occur.

[0057] (Other Components)

The working fluid of the present invention may contain other components that are not described above. Various kinds of additives which do not cause negative influences on the workpiece, such as corrosion and discoloration of the workpiece and which do not affect stability of a system after mixing, may be added to the working fluid of the present invention within the range that does not affect the processibility. Examples of such additives include a viscosity adjuster, a pH adjuster, and an anti-oxidation agent. The viscosity adjuster, the pH adjuster, and the anti-oxidation agent are not particularly limited and known ones may be used. However, those that are soluble in water are preferred.

Examples

[0058] <Production of a Working Fluid>

Water-soluble working fluids for a fixed-abrasive wire saw of the present invention (Examples 1 to 4) and water-soluble working fluids for a fixed-abrasive wire saw not being the present invention (Comparative Examples 1 to 13) were made such that the working fluids had the respective compositions shown in Tables 1 and 2. The content of each component shown in Tables 1 and 2 is represented in mass %. Comparative Example 9 is a working fluid containing 10 mass % of a synthetic fluid produced by YUSHIRO CHEMICAL INDUSTRY CO., LTD. and 90 mass % of water. In addition, the "C10alcohol:EOPO" shown in Tables 1 and 2 refers to a copolymer of an alcohol having a carbon number of 10, ethylene oxide, and propylene oxide.

[0059] <Evaluations>

The working fluids produced were evaluated in terms of the evaluation items shown in Tables 1 and 2. The "appearance" shows the result of the appearance of each working fluid observed visually. The "pH" shows the measurement result of the pH at 25°C of each working fluid. The "clouding point" shows the measurement result of the clouding point of each working fluid. The "viscosity of working fluid" shows the viscosity at 25°C of each working fluid measured by using a Brookfield-type viscometer. The "surface tension" shows the result of the static surface tension at 25°C of each working fluid measured by using a digital tension meter RTM-101 manufactured by RIGO CO., LTD. and using the du Nouy ring method. The "electrical conductivity" shows the result of the electrical conductivity at 25°C of each working fluid.

measured by using a conductivity meter ES-51 manufactured by HORIBA. The "viscosity of simulated fluid" shows the measurement result obtained by the following procedure. First, a simulated fluid was made by: adding 10 mass % of a silicon powder (average particle size: 1.5 μm) to each of the working fluids; mixing them by stirring; thereafter putting a stainless steel ball (2 mm in diameter) into each of the resultant mixtures; and stirring the mixtures at 1000 rpm for 10 hours. Next, the stainless steel ball was filtered off from each of the simulated fluids by using a metallic mesh (50 mesh), and thereafter the viscosity at 25°C of each of the simulated fluids was measured by using a Brookfield-type viscometer. The "amount of hydrogen generated" shows the measurement result of the amount of hydrogen generated within 30 minutes after heating 10 ml of each of the above simulated fluids up to 50°C. For evaluation on the "hard cake", each of the simulated fluids was let to stand in a glass container for a week, a supernatant liquid was removed, and thereafter hardness of a precipitation layer that remained at the bottom of the glass container was checked by using a medicine spoon. When the precipitation layer was so hard that the medicine spoon could not reach the bottom of the glass container, it was judged as poor. When the medicine spoon reached the bottom of the glass container, it was judged as good. The "precipitation performance" was evaluated visually from the color of the supernatant liquid after letting each of the simulated fluids stand in the glass container for a week. When the supernatant liquid was not clear due to the silicon powder suspended therein, it was judged as poor. When the supernatant liquid was clear,

it was judged as good. To evaluate the "corrosivity", the following procedure was taken: cast iron debris were put in a glass petri dish; the working fluid was poured thereinto until the cut debris were immersed in the working fluid completely; thereafter, the glass petri dish was let to stand for ten minutes with the lid put thereon; then, the working fluid was drained by inclining the petri dish with the lid put thereon; subsequently, the petri dish was put on a horizontal table and was let to stand for 24 hours in this state; and then generation of rust was evaluated through observation. In the evaluation of the corrosivity, "good" means that generation of rust was less than 10% of the cast iron debris.

[0060]

[Table 1]

| Source Materials and Evaluation Items | Example 1 | Example 2 | Example 3 | Example 4 | Comparative Example 1 | Comparative Example 2 | Comparative Example 3 | Comparative Example 4 | Comparative Example 5 |
|---------------------------------------|-----------------|-----------------|-----------------|-----------------|---------------------------|---------------------------|---------------------------|--------------------------|--------------------------|
| diethylene glycol | | | 1 | | 100 | 20 | | 20 | 20 |
| diisopropylene glycol | 5 | 20 | | | | | | 10 | |
| polypropylene glycol (Mw=1000) | | | | | | | | | 10 |
| citric acid | 0.3 | 0.4 | 0.1 | 0.1 | | | | | |
| decanedioic acid | | 0.1 | | | | | | | |
| potassium hydroxide | | 0.25 | | | | | | | |
| triethanolamine | | | 0.3 | 0.3 | | | | | |
| N-(β-aminoethyl)ethanol amine | 0.2 | 0.2 | | | | | | | |
| water | 91.78 | 70.63 | 98.38 | 99.38 | | 80 | 100 | 70 | 70 |
| polyoxyalkylene glycol (Mw=3000) | 2 | 8 | | | | | | | |
| C10alcohol:EOPO | 0.2 | 0.2 | 0.2 | 0.2 | | | | | |
| polyvinylpyrrolidone (Mw=70000) | 0.2 | 0.2 | | | | | | | |
| silicon-based defoaming agent | 0.02 | 0.02 | 0.02 | 0.02 | | | | | |
| Total | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| appearance | slightly yellow | slightly yellow | slightly yellow | slightly yellow | colorless and transparent | colorless and transparent | colorless and transparent | slightly white turbidity | slightly white turbidity |
| pH | 9.3 | 8.3 | 9.5 | 9.5 | 5.0 | 3.8 | 6.8 | 3.7 | 3.7 |
| clouding point [°C] | 36.0 | 23.0 | 30.0 | 30.0 | None | None | None | None | None |
| viscosity of working fluid [mPa·s] | 2 | 2 | 2 | 2 | 29 | 2 | 1 | 3 | 3 |
| surface tension [mN/m] | 27 | 27 | 25 | 25 | 47 | 50 | 72 | 48 | 48 |
| electrical conductivity [μ S/cm] | 998 | 2580 | 336 | 336 | <0.1 | 47 | 145 | 40 | 40 |
| amount of hydrogen generated [ml] | 4 | 6 | 6 | 6 | <0.1 | 25 | 32 | 20 | 20 |
| viscosity of simulated fluid [mPa·s] | 3 | 11 | 2 | 2 | 35 | 4 | 1 | 6 | 6 |
| hard cake | good | good | good | good | good | poor | poor | poor | poor |
| precipitation performance | good | good | good | good | good | poor | poor | poor | poor |
| corrosivity | good | good | good | good | good | good | good | good | good |

[0061]

[Table 2]

| Source Materials and Evaluation Items | Comparative Example 6 | Comparative Example 7 | Comparative Example 8 | Comparative Example 9 | Comparative Example 10 | Comparative Example 11 | Comparative Example 12 | Comparative Example 13 |
|--|---------------------------|---------------------------|------------------------------|---------------------------------|---------------------------------|---------------------------------|------------------------|------------------------|
| diethylene glycol | 20 | 20 | 20 | 20 | 20 | 20 | 20 | 20 |
| dipropylene glycol | 10 | 10 | 10 | | | | | |
| polypropylene glycol (Mw=1000) | | | | | | | | |
| citric acid | 0.3 | 0.3 | 0.33 | | | | 0.4 | 0.5 |
| dodecanedioic acid | | | | | 0.05 | 0.1 | 0.1 | 0.1 |
| potassium hydroxide | 0.08 | 0.22 | 0.29 | | | | 0.25 | 0.5 |
| triethanolamine | | | | | 0.22 | 0.44 | | |
| N-(β -aminoethyl)ethanol amine | | | | | | | 0.2 | |
| water | 69.62 | 69.48 | 69.38 | | | | 79.46 | 78.48 |
| polyoxyalkylene glycol (Mw=3000) | | | | | | | 79.73 | |
| Cl alcohol:EOPO | | | | | | | | 8 |
| polyvinylpyrrolidone (Mw=70000) | | | | | | | 0.2 | 0.2 |
| silicon-based defoaming agent | | | | | | | 0.02 | 0.02 |
| Total | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| appearance | colorless and transparent | colorless and transparent | light yellow and transparent | slightly yellow | slightly yellow |
| pH | 4.2 | 6.6 | 11.0 | 9.0 | 8.2 | 8.1 | 8.3 | 8.8 |
| clouding point [°C] | None | None | None | 23.0 | None | None | 23.0 | 33.0 |
| viscosity of working fluid [mPa·s] | 3 | 3 | 3 | 2 | 2 | 2 | 2 | 2 |
| surface tension [mN/m] | 48 | 48 | 48 | 34 | 42 | 42 | 27 | 27 |
| electrical conductivity [μS/cm] | 531 | 1381 | 1793 | 2380 | 153 | 297 | 2570 | 3380 |
| amount of hydrogen generated [ml] | 2 | 12 | 50< | 7 | 6 | 5 | 7 | 6 |
| viscosity of simulated fluid [mPa·s, 25°C] | 9 | 30 | 100< | 36 | 6 | 6 | 63 | 10 |
| hard cake | good | good | good | good | poor | poor | good | good |
| precipitation performance | good | good | good | good | poor | poor | good | good |
| corrosivity | poor | good | good | good | good | good | good | poor |

[0062] As shown in Table 1, Examples 1 to 4 exhibited favorable results in each of the evaluations on the hard cake, precipitation performance, and corrosivity. Additionally, in Examples 1 to 4, the amount of hydrogen generated was small and the viscosity of the simulated fluids was low. On the other hand, as shown in Tables 1 and 2, Comparative Examples 1 to 13 had poor results in any one of the evaluations on the hard cake, the precipitation performance and the corrosivity, had a high viscosity of the simulated fluids was high, or had a large amount of hydrogen generated.

[0063] The present invention has been described above as to the embodiment which is supposed to be practical as well as preferable at present. However, it should be understood that the present invention is not limited to the embodiment disclosed in the specification of the present application and can be appropriately modified within the range that does not depart from the gist or spirit of the invention, which can be read from the appended claims and the overall specification, and that a water-soluble working fluid for a fixed abrasive wire saw with such modifications are also encompassed within the technical range of the present invention.

Industrial Applicability

[0064] The water-soluble working fluid for a fixed-abrasive wire saw of the present invention can be used when cutting a silicon wafer by using a fixed-abrasive wire saw.

CLAIMS

1. A water-soluble working fluid for a fixed-abrasive wire saw, the water-soluble working fluid comprising:

(C) a carboxylic acid;

(D) a compound which shows basicity on dissolution in water;

and

(E) water,

wherein an electrical conductivity at 25°C of the water-soluble working fluid is 300 µS/cm or more and 3000 µS/cm or less;

a pH at 25°C of the water-soluble working fluid is 5 or more and 10 or less; and

a viscosity of a simulated fluid formed by adding 10 mass % of a silicon powder having an average particle size of 1.5 µm to the water-soluble working fluid and stirring a resultant mixture is less than 30 mPa·s at 25°C.

2. The water-soluble working fluid for a fixed-abrasive wire saw according to claim 1, further comprising (A) at least one nonionic surfactant selected from copolymers of an alcohol, ethylene oxide, and propylene oxide, and polyoxyalkylene glycols.

3. The water-soluble working fluid for a fixed-abrasive wire saw according to claim 1 or 2, further comprising (B) a glycol.

4. A water-soluble working fluid for a fixed-abrasive wire saw, the water-soluble working fluid comprising:

(A) 0.1 mass % or more and 8 mass % or less of at least one nonionic surfactant selected from copolymers of an alcohol, ethylene oxide, and propylene oxide, and polyoxyalkylene glycols;

(B) 0.1 mass % or more and 80 mass % or less of a glycol;

(C) 0.01 mass % or more and 5 mass % or less of a carboxylic acid;

(D) 0.01 mass % or more and 7 mass % or less of a compound which shows basicity on dissolution in water; and

(E) water,

wherein an electrical conductivity at 25°C of the water-soluble working fluid is 300 µS/cm or more and 3000 µS/cm or less;

a pH at 25°C of the water-soluble working fluid is 5 or more and 10 or less; and

a viscosity of a simulated fluid formed by adding 10 mass % of a silicon powder having an average particle size of 1.5 µm to the water-soluble working fluid and stirring a resultant mixture is less than 30 mPa·s at 25°C.

5. The water-soluble working fluid for a fixed-abrasive wire saw according to any one of claims 1 to 4, having a surface tension at 25°C of 20 mN/m or more and 50 mN/m or less.

6. The water-soluble working fluid for a fixed-abrasive wire saw according to any one of claims 1 to 5, further comprising a water-soluble polymer and/or a defoaming agent.

7. The water-soluble working fluid for a fixed-abrasive wire

saw according to any one of claims 1 to 6, containing 10 mass % or more and 99.7 mass % or less of the (E) water, based on a total mass of the water-soluble working fluid for a fixed-abrasive wire saw as 100 mass %.