

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. In specific, it relates to a water-soluble working fluid for a fixed-abrasive wire saw, for use in cutting a silicon wafer with a fixed-abrasive wire saw.

Background Art

[0002] Nowadays, the importance of improving cutting techniques of a silicon wafer is increasing in order to reduce manufacturing costs thereof. Cutting by a wire saw in a loose-abrasive form is a mainly adopted method in processing a silicon wafer, but the loose-abrasive-type processing entails various problems as will be described below.

[0003] (1) Because a slurry made by dispersing abrasive grains in fluid is used as a working fluid, it is difficult to separate the loose abrasive grains and cut debris. (2) There is a limitation to the running speed of the wire, resulting in a limitation to improving the processing efficiency. (3) When the wire is made thin in order to reduce the kerf loss, it breaks during the processing, leading to decrease in the yield. (4) When the abrasive grains are refined in order to reduce the kerf loss, there will be an adverse result such as viscosity increase of the slurry, leading to increase in the kerf loss.

[0004] In order to solve these problems, a fixed-abrasive wire saw is being developed nowadays which has diamond abrasive fixed onto a surface of a piano wire or the like by electrodeposition, a resin bond, or some other means (Patent Document 1). When processing a brittle material by using the fixed-abrasive wire saw, a working fluid is used for the purposes of lubrication, cooling, and dispersion of cut debris generated. In consideration of the problem of flammability and some other factors as well, it is preferable to use a water-soluble working fluid. However, when a material to be cut is silicon, there will be high reactivity between the cut debris and the working fluid compared to general materials, resulting in reaction of the silicon debris (cut debris) with water or an alkali in the working fluid to generate hydrogen, and leading to likelihood of ignition. For these reasons, a working fluid which is inhibited from reacting with silicon is preferred. From such a viewpoint, there is suggested a working fluid described in Patent Document 2.

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] However, it is found that the cut debris has become

thin nowadays as the processing precision is improved; and therefore, the working fluid described in Patent Document 2 cannot exhibit enough stability. Particularly, it is found that the viscosity of the working fluid with the cut debris mixed therein increases, causing various problems. Further, when a working fluid contains a large amount of water and has a pH showing alkalinity, there occurs not only the problem of hydrogen generation but also the problem of viscosity increase or gelation.

[0007] The viscosity increase or gelation of the working fluid causes below problems.

(1) The amount of fluid brought by a wire varies, causing large nonuniformity in thickness of a cut material (deterioration of the product quality).

(2) Slipping of the wire occurs, causing the wire to break (decrease in the yield)

(3) It is difficult to perform washing in the subsequent step (it is difficult to remove the cut debris or the fluid between wafers).

(4) The life of the fluid becomes short, causing increase in the amount of fluid to be changed (increase in costs).

[0008] Accordingly, an object of the present invention is to provide a water-soluble working fluid for a fixed-abrasive wire saw which can inhibit: increase in its viscosity when mixed with cut debris; reaction between the working fluid and the cut debris, and thereby generation of hydrogen; and also viscosity increase or gelation of the working fluid.

Means for Solving the Problems

[0009] The inventors conducted an intensive study to solve the above problems, and obtained the following findings.

(1) The viscosity increase caused by Si cut debris mixed in occurs because the Si cut debris mixed in is in fine powder form and because dispersibility of the Si cut debris is poor.

(2) The above viscosity increase can be prevented by including a predetermined water-soluble polymer in the working fluid to increase dispersibility of the Si cut debris.

(3) When the Si cut debris is contained in the working fluid, the Si cut debris and the working fluid may react with each other, causing generation of hydrogen.

(4) When the working fluid contains a large amount of water, it may increase its viscosity or be gelated by having Si cut debris in it.

(5) The above problems (3) and (4) can be solved by including a predetermined water-soluble polymer in the working fluid.

[0010] The inventors examined the above problems intensively, and has completed the invention described below.

A first aspect of the present invention is a water-soluble working fluid for a fixed-abrasive wire saw (hereinafter sometimes referred to as a "working fluid of the present invention"), comprising: (A) at least one water-soluble polymer selected from polyvinylpyrrolidone and a copolymer including vinylpyrrolidone; and (B) water.

[0011] The working fluid of the present invention comprises a predetermined water-soluble polymer as (A) component. Thereby when it contains silicon powder, dispersibility of the silicon powder can be improved and viscosity increase of the working fluid

containing the silicon powder can be inhibited. Further, it is possible to inhibit the working fluid and the silicon powder from reacting with each other to generate hydrogen, and also possible to inhibit viscosity increase or gelation of the working fluid containing the silicon powder.

[0012] In the first aspect of the present invention, a weight-average molecular weight of the (A) component is preferably 2,000 to 1,000,000.

[0013] In the first aspect of the present invention, the content of the (A) component is preferably 0.02 mass% or more and 7 mass% or less, based on the total mass of the water-soluble working fluid for a fixed-abrasive wire saw as 100 mass%.

[0014] The water-soluble working fluid for a fixed-abrasive wire saw of the first aspect of the present invention preferably further comprises (C) an alkali salt of a polycarboxylic acid. The content of the (C) component is preferably 0.01 mass% or more and 10 mass% or less, based on the total mass of the water-soluble working fluid for a fixed-abrasive wire saw as 100 mass%. The working fluid of the present invention can be easily given dispersibility, washing property, and corrosion resistance by containing an alkali salt of a polycarboxylic acid in the working fluid as (C) component.

[0015] The water-soluble working fluid for a fixed-abrasive wire saw of the first aspect of the present invention preferably further comprises (D) one or more selected from the group consisting of glycols, glycol ethers, and polyoxyalkylene glycols. The content of the (D) component is preferably 0.1 mass% or more and 95 mass% or less, based on the total mass of the water-soluble

working fluid for a fixed-abrasive wire saw as 100 mass%.

[0016] The viscosity of the water-soluble working fluid for a fixed-abrasive wire saw is preferably 50 mPa·s or less at 25°C. If the viscosity of the working fluid itself is high, it becomes much higher when the working fluid contains silicon powder. Therefore, the viscosity of the working fluid itself is preferably not more than a predetermined value.

[0017] The viscosity of a working fluid (simulated fluid) formed by adding 10 mass% of silicon powder having an average particle size of 1.5 μm to the water-soluble working fluid for a fixed-abrasive wire saw of the first aspect of the present invention and stirring, is preferably 100 mPa·s or less at 25°C. If the viscosity of the simulated fluid containing predetermined silicon powder is high, the problems listed in the above section "Problems to be Solved by the Invention" are likely to occur.

Effects of the Invention

[0018] The water-soluble working fluid for a fixed-abrasive wire saw of the present invention comprises a predetermined water-soluble polymer. Therefore when the working fluid contains silicon powder, which is silicon cut debris, the silicon powder can be dispersed in the working fluid. Accordingly, viscosity increase of the working fluid containing silicon powder can be inhibited. Further, since the working fluid of the present invention comprises a predetermined water-soluble polymer, it is possible to inhibit the silicon powder and the working fluid from reacting with each other to generate hydrogen, and also possible to inhibit viscosity increase or gelation of the working

fluid containing the silicon powder.

Modes for Carrying Out the Invention

[0019] <Water-Soluble Working Fluid for a Fixed-Abrasive Wire Saw>

The water-soluble working fluid for a fixed-abrasive wire saw of the present invention comprises: (A) at least one water-soluble polymer selected from polyvinylpyrrolidone and a copolymer including vinylpyrrolidone; and (B) water.

[0020] ((A) Component)

The working fluid of the present invention comprises: at least one water-soluble polymer selected from polyvinylpyrrolidone and a copolymer including vinylpyrrolidone as (A) component. With the water-soluble polymer contained in the working fluid, it is possible to improve dispersibility of the silicon powder in the working fluid containing the silicon powder. As a result, viscosity increase of the working fluid containing silicon powder can be inhibited. It is also possible to inhibit reaction of the silicon powder and the working fluid to prevent generation of hydrogen in the working fluid containing silicon powder. Furthermore, viscosity increase or gelation of the working fluid containing silicon powder can be inhibited.

[0021] As to the weight-average molecular weight of the water-soluble polymer as the (A) component determined by the gel permeation chromatography/multi-angle laser light scattering detection method, the lower limit thereof is preferably 2,000 or more, and more preferably 8,000 or more; and the upper limit thereof is preferably 1,000,000 or less, more preferably 700,000

or less, and further more preferably 500,000 or less. If the molecular weight is too small being outside the above range, the advantageous effect of adding the (A) component may not be exerted. On the other hand, if the molecular weight is too large, agglomeration or large viscosity increase in the working fluid is likely to occur.

[0022] Based on the total mass (100 mass%) of the water-soluble working fluid for a fixed-abrasive wire saw, the lower limit of the content of the (A) component is preferably 0.02 mass% or more, more preferably 0.05 mass% or more, and further more preferably 0.2 mass% or more; and the upper limit thereof is preferably 7 mass% or less, more preferably 5 mass% or less, and more preferably 3 mass% or less. If the content of the (A) component is too small being outside the above range, the advantageous effects of inhibiting viscosity increase and of preventing generation of hydrogen may be poorly exerted. On the other hand, if the content of the (A) component is too large, the viscosity of the working fluid is likely to increase.

[0023] The water-soluble polymer as the (A) component is polyvinylpyrrolidone or a copolymer including vinylpyrrolidone; and two or more kinds thereof may be used in mixture. As to the copolymer including vinylpyrrolidone, the proportion of the vinylpyrrolidone unit based on the entire copolymer is preferably 60 mol% or more. A monomer to be copolymerized with vinylpyrrolidone may be vinyl acetate.

[0024] ((C) Component)

The working fluid of the present invention may further comprise an alkali salt of a polycarboxylic acid as (C) component.

By adding the (C) component and adjusting the amount of addition thereof, it is possible to attain advantageous effects of giving the working fluid dispersibility, washing property, and corrosion resistance.

[0025] Examples of the polycarboxylic acid include adipic acid, oxalic acid, dodecanedioic acid, citric acid, and malic acid. Examples of the alkali include: hydroxides of alkali metal such as potassium hydroxide, sodium hydroxide; and amines such as triethanolamine, triisopropanolamine, ethylenediamine, and N-(2-aminoethyl)-2-aminoethanol.

[0026] Based on the total mass (100 mass%) of the working fluid of the present invention, the lower limit of the content of the (C) component is preferably 0.01 mass% or more, and more preferably 0.1 mass% or more; the upper limit thereof is preferably 10 mass% or less, more preferably 2 mass% or less, and further more preferably 1 mass% or less. If the content of the (C) component is too small being outside the above range, it is difficult to realize the advantageous effect of adding the (C) component. On the other hand, if the content of the (C) component is too large, there may occur viscosity increase or gelation of the working fluid, or precipitation from the original fluid.

[0027] ((D) Component)

The working fluid of the present invention may still further comprise, as (D) component, one or more selected from the group consisting of glycols, glycol ethers, and polyoxyalkylene glycols. Including the (D) component produces an advantageous effect of giving the working fluid lubricity and wettability.

[0028] Examples of the glycols include propylene glycol, diethylene glycol, ethylene glycol, and butylene glycol. Examples of the glycol ethers include alkyl ethers of the above glycols. Examples of the alkyl group include a methyl group, an ethyl group, and a butyl group. A part of the hydroxyl group of the glycols may be an alkyl ether, or all thereof may be an alkyl ether. Specific examples of the glycol ethers include diethylene glycol monoethyl ether, diethylene glycol diethyl ether, and propylene glycol monoethyl ether.

[0029] Examples of the polyoxyalkylene glycols include polyethyleneglycol, polypropylene glycol, and a copolymer of polyoxyethylene and polyoxypropylene; and the weight-average molecular weight thereof (in terms of polystyrene using the gel permeation chromatography) is preferably 10,000 or less, more preferably 5,000 or less, and further more preferably 400 or less.

[0030] Based on the total mass (100 mass%) of the working fluid of the present invention, the lower limit of the content of the (D) component, is preferably 0.1 mass% or more, more preferably 0.5 mass% or more, further more preferably 1 mass% or more, still further more preferably 3 mass% or more, and in especial preferably 10 mass% or more; and the upper limit thereof is preferably 95 mass% or less, more preferably 90 mass% or less, and further more preferably 80 mass% or less. If the content of the (D) component is too small being outside the above range, the advantageous effect of adding the (D) component cannot be exerted. On the other hand, if the content of the (D) component is too large, the cooing property of the working fluid may degrade.

[0031] The working fluid of the present invention contains

the (A) component, and in some cases further contains the (C) component and the (D) component, the rest being (B) water. The water may be of any kind without particular limitations, and thus it may be distilled water, tap water, etc. A working fluid that contains a large amount of (B) water tends to increase its viscosity or be gelled when silicon powder is contained therein. By contrast, the working fluid of the present invention can inhibit viscosity increase or gelation of the working fluid when containing silicon powder, even with a composition in which a large amount of water is contained, for example a composition in which water accounts for 90 mass% or more based on the total mass of the working fluid.

[0032] <Characteristics of the Working Fluid>

The viscosity of the working fluid of the present invention is preferably 50 mPa·s or less, more preferably 25 mPa·s or less, and further more preferably 20 mPa·s or less, at 25°C. The viscosity of a working fluid (simulated fluid) formed by dispersing predetermined silicon powder in the working fluid of the present invention, is 100 mPa·s, preferably 55 mPa·s or less, more preferably 50 mPa·s or less, and further more preferably 45 mPa·s or less, at 25°C. The measurement of the viscosity is based on a simulated fluid obtained by: adding 10 mass% of silicon powder (particle size: 1.5 μm) to the working fluid of the present invention; stirring and mixing them; thereafter putting a stainless steel ball (2 mm in diameter) into the mixture; stirring the mixture at 1000 rpm for 10 hours; and removing the stainless steel ball by filtration. If the viscosity of the above described working fluid itself is too high, the viscosity of the simulated

fluid containing the silicon powder will also be inevitably high. Further, if the viscosity of the simulated fluid is too high, the problems described in the above section "Problems to be Solved by the Invention" are likely to occur. The viscosity of the working fluid and the simulated fluid can be measured by a Brookfield type viscometer.

[0033] The pH of the working fluid is preferably 5.0 or more and 9.0 or less. If the pH of the working fluid is too low, an iron material or a wire brought into contact with the working fluid is likely to corrode. On the other hand, if the pH of the working fluid is too high, it is likely that the working fluid and the silicon powder react with each other to generate hydrogen. The working fluid of the present invention may be used in the form of being diluted with water, but even in that case, the pH of the working fluid after diluted is preferably within the above range.

Examples

[0034] <Examples 1 to 13 and Comparative Examples 1 to 5>

The water-soluble working fluids for a fixed-abrasive wire saw of the present invention (Examples 1 to 13) and water-soluble working fluids for a fixed-abrasive wire saw not being the present invention (Comparative Examples 1 to 5) were prepared such that the compositions thereof are respectively as shown in Tables 1 to 3. The pH of each working fluid prepared was measured, and the viscosity thereof at 25°C was measured by a Brookfield type viscometer.

Additionally, simulated fluids were formed by: adding 10

mass% of silicon powder (average particle size: 1.5 μm) to each of the working fluids; stirring and mixing them; thereafter putting a stainless steel ball (2 mm in diameter) into the mixture; and stirring the mixture at 1000 rpm for 10 hours. After removing the stainless steel ball from each of the simulated fluids by filtration using a metal mesh (50 mesh), the viscosity (mPa·s, 25°C) of each simulated fluid was measured by a Brookfield type viscometer. Further, heating 10 ml of the simulated fluids up to 50°C, the amount of hydrogen generated within thirty minutes was measured.

[0035] With regard to the weight-average molecular weight of polyvinylpyrrolidone (PVP), PVP K-15 had 9,700; PVP K-30 had 70,000; and PVP K-60 had 400,000. In addition, PVP/VA was a copolymer of vinylpyrrolidone and vinyl acetate; the weight-average molecular weight thereof was 32,000; and the proportion of vinylpyrrolidone in the copolymer was 70 mol%.

[0036]

[Table 1]

	Example 1	Example 2	Example 3	Example 4	Example 5	Example 6
PVP (M=2500)						
PVP (K-15)						
PVP (K-30)	0.3	0.3	0.3	1	3	5
PVP (K-60)						
PVP/VA						
propylene glycol				20	20	20
diethylene glycol						
polyoxyalkylene glycol (Mw=3000, ethylene oxide/propylene oxide=50%/50%)			5			
diethylene glycol monoethyl ether						
water	99.7	98.9	94	78.4	76.4	74.4
citric acid		0.3	0.3	0.3	0.3	0.3
dodecanedioic acid			0.1			
potassium hydroxide		0.2	0.1	0.3	0.3	0.3
triethanolamine		0.3				
N-(2-aminoethyl)-2- aminoethanol			0.2			
Total	100	100	100	100	100	100
viscosity of working fluid (mPa·s)	1.1	1.2	1.7	2.8	4.4	6.0
pH	7.2	7.1	8.8	6.2	6.2	6.3
viscosity of simulated fluid (mPa·s)	8	9	12	14	17	25
amount of hydrogen generated (ml)	4.8	4.7	4.1	2.7	2.1	1.8
note	10-fold dilution	10-fold dilution	10-fold dilution			

[0037]

[Table 2]

	Example 7	Example 8	Example 9	Example 10	Example 11	Example 12	Example 13
PVP (M=2500)	0.5						
PVP (K-15)		0.5					
PVP (K-30)			0.5			0.05	0.5
PVP (K-60)				0.5			
PVP/VA					0.5		
propylene glycol	70	70	70	70	70	70	
diethylene glycol							50
polyoxyalkylene glycol (Mw=3000, ethylene oxide/propylene oxide=50%/50%)							
diethylene glycol monoethyl ether							20
water	28.9	28.9	28.9	28.9	28.9	29.35	29
citric acid	0.4	0.4	0.4	0.4	0.4	0.4	0.3
dodecanedioic acid							
potassium hydroxide	0.2	0.2	0.2	0.2	0.2	0.2	0.2
triethanolamine							
N-(2-aminoethyl)-2- aminoethanol							
Total	100	100	100	100	100	100	100
viscosity of working fluid (mPa·s)	11.5	11.8	12.1	12.2	12.0	11.9	11.2
pH	6.0	6.0	5.9	6.0	6.0	5.9	6.1
viscosity of simulated fluid (mPa·s)	35	35	26	30	32	40	33
amount of hydrogen generated (ml)	2.1	1.6	1.3	0.9	1.5	2.5	1.6

[0038]

[Table 3]

	Comparative Example 1	Comparative Example 2	Comparative Example 3	Comparative Example 4	Comparative Example 5
PVP (M=2500)					
PVP (K-15)					
PVP (K-30)					
PVP (K-60)					
PVP/VA					
propylene glycol			20	70	
diethylene glycol					50
polyoxyalkylene glycol (Mw=3000, ethylene oxide/propylene oxide=50%/50%)		5			
diethylene glycol monoethyl ether					20
water	99.2	94.3	79.4	29.4	29.5
citric acid	0.3	0.3	0.3	0.4	0.3
dodecanedioic acid		0.1			
potassium hydroxide	0.2	0.1	0.3	0.2	0.2
triethanolamine	0.3				
N-(2-aminoethyl)-2- aminoethanol		0.2			
Total	100	100	100	100	100
viscosity of working fluid (mPa·s)	1.1	1.7	2.0	11.8	11.2
pH	7.9	8.8	6.2	6.0	6.1
viscosity of simulated fluid (mPa·s)	520	410	350	80	71
amount of hydrogen generated (ml)	18.5	16.7	15.1	3.4	3.3
note	10-fold dilution	10-fold dilution			

[0039] In the cases of the working fluids of Comparative Examples, there was viscosity increase in all of the simulated fluids to over 55 mPa·s. By contrast, as for the working fluids of the present invention (Examples 1 to 13), viscosity changes

were small in the simulated fluids, and none of these simulated fluids had a viscosity of over 45 mPa·s.

[0040] The working fluids of Examples 1 to 3 and Comparative Examples 1 and 2 were those obtained by diluting their original fluids by 10 times. Therefore, a composition of the original fluids was such that the amount of each compound therein was 10 times the amount in their diluted ones.

[0041] (Group I)

Group I consisted of Examples 1 to 6 and Comparative Examples 1 to 3, being examples of working fluids containing large amount of water. The working fluids of Comparative Examples which had large amount of water (Comparative Examples 1 to 3) all showed viscosity increase and were gelated. However, the working fluids of the present invention added with PVP (Examples 1 to 6) were not gelated and showed little viscosity increase.

In addition, the amount of hydrogen generated in Examples 1 to 6 did not exceed 5 ml, whereas the amount of hydrogen generated in Comparative Examples 1 to 3 was more than 15 ml, which was a significant amount.

[0042] (Group II)

Group II consisted of Examples 7 to 12 and Comparative Example 4, wherein the kind and the amount of (C) component and of (D) component were the same and the (A) component was different. From this, the advantageous effect of inhibiting viscosity increase by each kind of polyvinylpyrrolidone (PVP) and a vinylpyrrolidone/vinyl acetate copolymer (PVP/VA) was shown.

In addition, comparing the amount of hydrogen generated in Examples 7 to 12 with that in Comparative Example 4, the amount

of hydrogen generated was small in the working fluids of the present invention containing polyvinylpyrrolidone etc.

[0043] (Group III)

Group III consisted of Example 13 and Comparative Example 5, containing (D) component different from that in Groups I and II. From this, the advantageous effect of inhibiting viscosity increase by polyvinylpyrrolidone (PVP) was shown regardless of the kind of (D) component.

In addition, comparing Example 13 and Comparative Example 5, the amount of hydrogen generated was small in Example 13 which included polyvinylpyrrolidone.

From above, it is shown that the working fluid of the present invention comprising at least one water-soluble polymer selected from polyvinylpyrrolidone and a copolymer including vinylpyrrolidone has advantageous effects of inhibiting viscosity increase and hydrogen generation in the simulated fluid having silicon powder dispersed therein.

[0044] 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 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

[0045] The water-soluble working fluid for a fixed-abrasive wire saw of the present invention can be especially suitably used in cutting a silicon wafer with using a fixed-abrasive wire saw.

CLAIMS

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

(A) at least one water-soluble polymer selected from polyvinylpyrrolidone and a copolymer including vinylpyrrolidone;

(B) water;

(C) an alkali metal salt of a polycarboxylic acid or an amine salt of a polycarboxylic acid; and

(D) one or more selected from the group consisting of glycols, glycol ethers, and polyoxyalkylene glycols, wherein the water-soluble working fluid does not comprise loose grain,

Wherein a content of said (A) component is 0.02 mass% or more and 7 mass% or less, based on the total mass of the water-soluble working fluid for a fixed-abrasive wire saw as 100 mass%,

Wherein a content of said (C) component is 0.01 mass% or more and 10 mass% or less, based on the total mass of the water-soluble working fluid for a fixed-abrasive wire saw as 100 mass%, and

Wherein a content of said (D) component is 0.1 mass% or more and 95 mass% or less, based on the total mass of the water-soluble working fluid for a fixed-abrasive wire saw as 100 mass%.

2. The water-soluble working fluid for a fixed-abrasive wire saw according to claim 1, wherein a weight-average molecular weight of said (A) component is 2,000 to 1,000,000.

3. The water-soluble working fluid for a fixed-abrasive wire saw according to claim 1 or 2, wherein a viscosity of the working fluid is 50 mPa·s or less at 25°C.

4. The water-soluble working fluid for a fixed-abrasive wire saw according to any one of claims 1 to 3, wherein the water-soluble working fluid is used in cutting a silicon wafer.