



(11) **EP 2 415 853 A1**

(12) **EUROPEAN PATENT APPLICATION**
published in accordance with Art. 153(4) EPC

(43) Date of publication:
08.02.2012 Bulletin 2012/06

(21) Application number: **10758452.6**

(22) Date of filing: **19.03.2010**

(51) Int Cl.:
C10M 173/02 (2006.01) **B24B 37/00** (2012.01)
C10M 107/28 (2006.01) **C10M 107/34** (2006.01)
C10M 145/14 (2006.01) **C10M 145/26** (2006.01)
C10N 20/00 (2006.01) **C10N 20/04** (2006.01)
C10N 30/00 (2006.01) **C10N 40/32** (2006.01)

(86) International application number:
PCT/JP2010/054813

(87) International publication number:
WO 2010/113678 (07.10.2010 Gazette 2010/40)

(84) Designated Contracting States:
**AT BE BG CH CY CZ DE DK EE ES FI FR GB GR
HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL
PT RO SE SI SK SM TR**

(30) Priority: **31.03.2009 JP 2009086107**

(71) Applicant: **Idemitsu Kosan Co., Ltd.**
Chiyoda-ku
Tokyo 100-8321 (JP)

(72) Inventor: **KITAMURA, Tomohiko**
Ichihara-shi
Chiba 299-0107 (JP)

(74) Representative: **HOFFMANN EITLE**
Patent- und Rechtsanwälte
Arabellastraße 4
81925 München (DE)

(54) **WORKING FLUID FOR BRITTLE MATERIAL AND WORKING FLUID FOR HARD MATERIAL**

(57) Provided is a water-containing working fluid for a brittle material and a hard material, which includes (A) water, (B) a water soluble polymeric compound having an oxygen-containing group and a number average molecular weight of 6,000 to 3,000,000, and (C) a nonionic surfactant having a clouding point of 10 to 70°C in a 1% by mass aqueous solution. In a loose-abrasive grain method, the working fluid allows a slurry thereof to be

supplied into the working gap sufficiently and has excellent abrasive grain dispersion stability and excellent properties to wash and disperse cutting swarf. In a method using a fixed-abrasive grain wire saw, the working fluid rarely causes detachment of the fixed abrasive grains and has excellent properties to wash and disperse cutting swarf.

EP 2 415 853 A1

Description

[Technical Field]

[0001] The present invention relates to a working fluid for brittle materials and to a working fluid for hard materials and, more specifically, to a working fluid for brittle materials and hard materials which is suitably used for cutting brittle materials and hard materials with a wire saw using loose abrasive grains or with a fixed-abrasive grain wire saw.

[Background Art]

[0002] In the production of semiconductor products, it is important to work a silicon ingot, which is a brittle material, with high accuracy. Wire saw working is generally used to groove or cut the ingot from the viewpoint of working accuracy and productivity.

In grooving or cutting of a hard material which is difficult to cut, such as ceramics, quartz or sapphire, on the other hand, wire saw working using super hard abrasive grains such as diamond abrasive grains and cubic crystal boron nitride abrasive grains (cBN) is utilized.

[0003] As the wire saw working, there may be mentioned a method in which grooving, cutting or internal grinding is carried out while feeding loose abrasive grains to a sliding interface between the object to be worked and the wire, and a method in which a fixed abrasive grain wire saw having abrasive grains directly fixed to a surface of the wire is used.

In the former wire saw working using loose abrasive grains, a slurry in which loose abrasive grains are dispersed in a working fluid is used for cutting a brittle material or a hard material with the wire saw made of a piano wire or the like. Thus, the working fluid is required not only to have superior lubricating performance and cooling performance but also to have an ability to maintain loose abrasive grains in a dispersed state and to be easily washed away after working. In recent years, semiconductor products or the like tend to be getting larger in size and more integrated, and the properties of working fluid must be improved accordingly.

In the latter method using a fixed abrasive grain wire saw, there are generally used an electro-deposition wire saw prepared by fixing abrasive grains to a core wire such as a piano wire by electrolytic deposition or a resin-bond wire saw prepared by fixing abrasive grains using a binder such as a polymeric material. In such a method using a fixed abrasive grain wire saw, the working fluid is particularly required to have a low frictional coefficient in order to prevent detachment of the fixed abrasive grains.

Further, in each of the above wire saw working methods, the working fluid is required to show excellent properties to wash away and disperse cutting swarf so that grinding swarf scarcely causes clogging of nozzles or working gaps to ensure stable working.

[0004] Known working fluids which have been hitherto used for such wire saw working is generally a working oil (working oil using a mineral oil or synthetic oil as a base oil). Since the working oil has an environmental problem and has a problem in safety, however, there is recently a demand for an aqueous working fluid, in particular, a working fluid having a high water content.

[0005] With regard to such a water-containing working fluid, Patent Document 1 discloses a non water-soluble dispersing medium composition which contains 1 to 20% by weight of water, and Patent Document 2 discloses an water-soluble, metal working fluid composition containing a specific amine compound. Although these compositions are water-containing working fluids, they use a base oil such as a mineral oil as a major ingredient. Thus, they do not completely solve the aforementioned environmental problem or problem in safety. Patent document 3 discloses a machining fluid containing an aqueous bentonite dispersion and a specific additive, and Patent Document 4 discloses an aqueous composition including siliceous acid colloid particles dispersed in a dispersing medium containing water, a hydrophilic polyhydric alcohol compound and a lipophilic polyhydric alcohol compound. Although these compositions do not use a base oil such as a mineral oil, the water content thereof is not high. Especially, the working examples of Patent Document 4 indicate that the performance is deteriorated when water is used in a large amount.

[Background Art]

[Patent Document]

[0006]

[Patent Document 1] Japanese Patent Application Publication No. H11-100590

[Patent Document 2] Japanese Patent Application Publication No. 2002-285186

[Patent Document 3] Japanese Patent Application Publication No. H08-60176

[Patent Document 4] Japanese Patent Application Publication No. H11-302681

[Summary of the Invention]

[Problems to be Solved by the Invention]

[0007] As described above, a water-containing working fluid is known. When the water content of the known working fluid is increased, however, abrasive grains used in a loose abrasive grain method are apt to sediment so that it becomes difficult to evenly supply the abrasive grains in the working gap. Although the abrasive grains might be expected not to easily sediment by an increase of the viscosity of the working fluid, it becomes difficult to feed a sufficient amount of the working fluid to the working gap especially when the viscosity is excessively high. Further, in the case of the method using a fixed abrasive grain wire saw, an increase of the water content of the known working fluid causes an increase of the coefficient of friction so that the fixed abrasive grains are apt to be detached.

In this circumstance, it is highly desired to provide a working fluid that has a high performance and can solve the environmental problem and problem of safety.

The present invention has been made under the above-described circumstances, and it is, therefore, an object of the present invention to provide a water-containing working fluid which, when used in a loose-abrasive grain method, allows a slurry thereof to be supplied into the working gap sufficiently and has excellent abrasive grain dispersion stability and excellent properties to wash and disperse cutting swarf, and to provide a water-containing working fluid which, when used in a method using a fixed-abrasive grain wire saw, rarely causes detachment of the fixed abrasive grains and has excellent properties to wash and disperse cutting swarf.

[Means for Solving the Problem]

[0008] The present inventors have made an earnest study and, as a result, found that the above problems can be solved by a working fluid containing specific additives.

That is, the present invention provides:

1. A working fluid for hard and brittle materials comprising the following components (A) to (C):

(A) water,

(B) a water soluble polymeric compound having an oxygen-containing group and a number average molecular weight of 6,000 to 3,000,000, and

(C) a nonionic surfactant having a clouding point of 10 to 70°C in a 1% by mass aqueous solution;

2. The working fluid for hard and brittle materials as recited in above 1, wherein the oxygen-containing group of the component (B) is a group selected from a carboxyl group, a hydroxyl group, an oxyethylene group and an oxypropylene group;

3. The working fluid for hard and brittle materials as recited in above 1 or 2, wherein the component (B) is a carboxylic acid-based polymeric compound and/or an alkylene glycol-based polymeric compound;

4. The working fluid for hard and brittle materials as recited in above 3, wherein the component (B) is a combination of a carboxylic acid-based polymeric compound and an alkylene glycol-based polymeric compound;

5. The working fluid for hard and brittle materials as recited in any one of above 1 to 4, wherein the component (C) is a nonionic surfactant containing an oxypropylene group and having a number average molecular weight of 70 to 5,000;

6. The working fluid for hard and brittle materials as recited in any one of above 1 to 5, wherein the content of the component (A) is 50 to 99% by mass, the content of the component (B) is 0.01 to 30% by mass, and the content of the component (C) is 0.1 to 20% by mass, based on the total amount of the working fluid;

7. The working fluid for hard and brittle materials as recited in any one of above 1 to 6, wherein the working fluid has a pH of 2 to 10; and

8. The working fluid for hard and brittle materials as recited in above 7, wherein the working fluid contains an acid component and an alkaline component, and wherein the pH has been adjusted by controlling a blending ratio of the acid and alkaline components.

[Effect of the Invention]

[0009] According to the present invention, it is possible to provide a water-containing working fluid which, when used in wire saw working by a loose abrasive grain method, allows a slurry thereof to be supplied into the working gap sufficiently and has excellent abrasive grain dispersion stability and excellent properties to wash (disperse) cutting swarf, and to provide a water-containing working fluid which, when used in a method using a fixed-abrasive grain wire saw,

rarely causes detachment of the fixed abrasive grains and has excellent properties to wash (disperse) cutting swarf.

[Best Mode for Carrying out the Invention]

[0010] A working fluid for hard materials and brittle materials (hereinafter occasionally referred to as "hard and brittle materials") of the present invention is a working fluid including (A) water, (B) a specific water soluble polymeric compound, and (C) a specific nonionic surfactant.

[0011] Though water used as the component (A) of the working fluid for hard and brittle materials according to the present invention is not specifically limited, purified water, particularly deionized water, is preferably used. The amount of water is generally 50 to 99% by mass, preferably 60 to 95% by mass, based on the total amount of the working fluid. When the amount of the water is 50% by mass or more, the flammability of the fluid is lowered so that the safety is improved. Further, such an amount is preferred from the environmental standpoint. There is no reason for specifying the upper limit. The amount of water is generally 99% by mass or less in view of the amount of the other components.

[0012] The water soluble polymeric compound used as the component (B) of the working fluid for hard and brittle materials according to the present invention is one which contains an oxygen-containing group and has a number average molecular weight of 6,000 to 3,000,000. In the present invention, the incorporation of the component (B) can control the viscosity of the working fluid so that, when used in a loose abrasive grain method, it is possible to improve the dispersion stability of the abrasive grains in the slurry and the washing efficiency of cutting swarf. When used in a fixed abrasive grain wire saw method, it is possible to improve the adhesion of the working fluid to the wire, to improve the penetration thereof into the working gap, to increase the cutting speed and to prevent detachment of the abrasive grains. When the number average molecular weight is less than 6,000, it is not easy to attain the viscosity controlling effect. Too large a number average molecular weight in excess of 3,000,000 causes a difficulty in feeding a sufficient amount of the slurry to the working gap in the loose abrasive grain method, because the viscosity excessively increases. From the above point of view, the number average molecular weight is preferably 10,000 to 1,500,000.

As the oxygen-containing group, there may be mentioned, for example, a carboxyl group, a hydroxyl group, an oxyethylene group and an oxypropylene group. Here, the carboxyl group and hydroxyl group may be each in the form of an anion formed by deprotonation or neutralization.

The term "water soluble polymeric compound" is a polymeric compound that is soluble in water and generally refers to a polymeric compound having a solubility in water at 20°C of at least 0.1 g per 100 g of water. The term "slurry" as used herein is intended to refer to a mixture containing the working fluid and abrasive grains.

[0013] Among compounds of the component (B), carboxylic acid-based polymeric compounds and alkylene glycol-based polymeric compounds are preferred for reasons of not only their excellent viscosity controlling effect but also their excellent functions as a dispersing agent and as an agent for improving washing performance.

The carboxylic acid-based polymeric compound is a polymeric compound obtained by polymerization of an unsaturated carboxylic acid containing a polymerizable group. Examples of the unsaturated carboxylic acid include acrylic acid, methacrylic acid, maleic anhydride, maleic acid, fumaric acid, crotonic acid and itaconic acid. Specific examples of the carboxylic acid-based polymeric compound include polyacrylic acid, polymethacrylic acid, polycrotonic acid, polyitaconic acid, polymaleic acid, polyfumaric acid, an acrylic acid-methacrylic acid copolymer, an acrylic acid-itaconic acid copolymer, an acrylic acid-maleic acid copolymer, an acrylic acid-acrylamide copolymer, an acrylic acid-acrylic ester copolymer, an acrylic acid-methacrylic ester copolymer, an acrylic acid-sulfonic acid type monomer copolymer and an acrylic acid-vinylpyrrolidone copolymer. These carboxylic acid-based polymeric compounds may be in the form of salts. Such salts may be, for example, sodium polyacrylate, potassium polyacrylate and ammonium polyacrylate.

The alkylene glycol-based polymeric compound is a polymer of alkylene glycol such as a polyalkylene glycol and derivative thereof. Specific examples of the alkylene glycol-based polymeric compound include polyethylene glycol, polypropylene glycol, a block copolymer of ethylene oxide and propylene oxide, and derivatives of these polymers (such as their ester derivatives and ether derivatives). Above all, a polymer containing oxyethylene groups is preferred.

[0014] In the present invention, the water soluble polymeric compound of the component (B) may be used singly or in combination of two or more thereof. In the case of the loose abrasive grain method, it is particularly preferable to use a carboxylic acid-based polymeric compound in combination with an alkylene glycol-based polymeric compound since dispersion stability of the abrasive grains and fluidity of sedimented abrasive grains are significantly improved.

[0015] In the working fluid for hard and brittle materials according to the present invention, the amount of the water soluble polymeric compound of the component (B) is generally 0.01 to 30% by mass, preferably 0.1 to 20% by mass, based on the total amount of the working fluid. When the using amount is 0.01 % by mass or more, sufficient viscosity controlling effect may be obtained. When the amount is 30% by mass or less, the viscosity is not excessively increased so that, in the case of the loose abrasive grain method, a sufficient amount of the slurry can be fed to the working gap and, in the case of the method using a fixed abrasive grain wire saw, a sufficient amount of the working fluid can be fed to the working gap.

[0016] The nonionic surfactant of the component (C) incorporated in the working fluid for hard and brittle materials

according to the present invention has a clouding point of 10 to 70°C, preferably 25 to 60°C, in a 1% by mass aqueous solution. In the present invention, the incorporation of the component (C) can improve the dispersion stability of the abrasive grains and the property to wash away cutting swarf in the case of a loose abrasive grain method. In the case of a fixed abrasive grain wire saw method, the component (C) can improve the washing efficiency of cutting swarf and reduce the coefficient of friction of the working fluid so that detachment of the abrasive grains is suppressed. When the clouding point is less than 10°C, the solubility becomes poor. Too high a clouding point in excess of 70°C causes an increase of foamability.

[0017] As preferred examples of the component (C), there may be mentioned a nonionic surfactant having an oxypropylene group (hereinafter occasionally referred to as PO-containing nonionic surfactant for brevity). Specific examples of the PO-containing nonionic surfactant include polypropylene glycol, an ester derivative of propylene glycol or polypropylene glycol, an ether derivative of propylene glycol or polypropylene glycol, and a block copolymer of propylene oxide and ethylene oxide (EO-PO block copolymer). Among them, EO-PO block copolymer is particularly preferred for reasons of dispersibility and low foamability.

The PO-containing nonionic surfactant is preferably a compound having a number average molecular weight of 70 to 5,000, more preferably 100 to 4,000. It is also preferred that the PO-containing nonionic surfactant contain oxypropylene groups in an amount of 40 to 90% by mass, more preferably 50 to 80% by mass, based on the whole molecule thereof.

[0018] In the present invention, the nonionic surfactant of the component (C) may be used singly or in combination of two or more thereof. The amount of the nonionic surfactant of the component (C) is generally 0.1 to 20% by mass, preferably 0.5 to 10% by mass, based on the total amount of the working fluid. When the using amount of 0.1 to 20% by mass, sufficient abrasive grain dispersing effect and washing and dispersing effect for cutting swarf may be obtained in the case of the loose abrasive grain method. In the case of the method using a fixed abrasive grain wire saw, it is possible to reduce the coefficient of friction of the working fluid so that detachment of the abrasive grains is suppressed and, further, to obtain effects such as improvement of the washing and dispersing efficiency for cutting swarf.

[0019] The working fluid for hard and brittle materials of the present invention may contain known additives such as a rust inhibitor, an antifoaming agent, an antioxidant, a metal deactivator and a disinfectant/antiseptic agent in an amount not to impair the purpose of the present invention. Examples of the rust inhibitor include alkylbenzene sulfonates, dinonylnaphthalene sulfonates, alkenyl succinic acid esters and polyhydric alcohol esters. Examples of the antifoaming agent include silicone oils, fluorosilicone oils and fluoroalkyl ethers. Examples of the antioxidant include phenol-based antioxidants and amine-based antioxidants. Examples of the metal deactivator include imidazoline, pyrimidine derivatives, thiadiazole, benzotriazole and thiazole. Examples of the disinfectant/antiseptic agent include p-oxybenzoic acid esters (parabens), benzoic acid, salicylic acid, sorbic acid, dehydroacetic acid, p-toluenesulfonic acid, salts of these acids, and phenoxyethanol.

The amount of the additives may be selected as appropriate depending on the object of use, and the total amount of the additives is usually about 0.01 to 5% by mass based on the total amount of the working fluid.

[0020] The working fluid for hard and brittle materials of the present invention preferably has a pH of 2 to 10, more preferably 4 to 8. When the pH is 2 or more, good rust preventing effect is obtainable. Corrosion of silicon, etc. may be prevented when the pH is 10 or less. The pH may be suitably adjusted by controlling the blending ratio of acid and alkaline components added to the working fluid for hard and brittle materials. Some of the various additives mentioned above may serve as acid or alkaline components. Examples of the acid component include polyacrylic acid and isonanoic acid. Examples of the alkaline component include N-methyldiethanolamine and cyclohexylethanolamine.

[0021] A method for producing the working fluid for hard and brittle materials of the present invention is not specifically limited. For example, there may be mentioned a method in which a concentrated working fluid for hard and brittle materials having a low water content is prepared. The concentration may be suitably adjusted by diluting the fluid before use. Such an embodiment is preferred from the standpoint of easiness in transportation and sale as well as easiness in adjustment of the concentration at the time of use. As such a concentrated working fluid for hard and brittle materials, there may be mentioned for example a fluid in which the component (C) is present in an amount of 0.001 to 50 parts by mass (preferably 0.01 to 30 parts by mass, more preferably 0.1 to 15 parts by mass) per 1 part by mass of the component (B) and in which a total content x (% by mass) of the components (B) and (C) is $5 \leq x < 100$ (preferably $80 \leq x \leq 99$) based on the total amount of the working fluid.

[0022] The slurry containing the working fluid for hard and brittle materials of the present invention generally has a viscosity of 40 to 200 mPa·s, preferably 50 to 180 mPa·s, at 25°C. When the viscosity of the slurry is within the above range, it is possible to feed a sufficient amount of the slurry to the working gap so that excellent machinability is attained. The slurry containing the working fluid for hard and brittle materials of the present invention has a relatively low viscosity and is generally high in water content. In general, such a low viscosity in the loose abrasive grain method results in an increased tendency to sedimentation of the abrasive grains. In the case of the working fluid for hard and brittle materials of the present invention which contains components (B) and (C) in combination, on the other hand, excellent dispersion stability of the abrasive grains is ensured.

[0023] The working fluid for hard and brittle materials of the present invention is suitably used in wire saw working of

brittle or hard materials using a wire saw or multiwire saw. Examples of the brittle material include silicon, quartz and carbon. Examples of the hard material include ceramics, quartz and sapphire.

In a loose abrasive grain method in the wire saw working, the working fluid for hard and brittle materials of the present invention and loose abrasive grains are mixed to prepare a composition (slurry) for working hard and brittle materials.

As the loose abrasive grains, any abrasive grains can be used without any particular limitations, and examples include SiC (silicon carbide) abrasive grains, alumina abrasive grains, cBN abrasive grains and diamond abrasive grains. While the amount of the loose abrasive grains may be determined as appropriate depending on the object of use, the mass ratio of the working fluid for hard and brittle materials to the loose abrasive grains is usually 95:5 to 10:90, preferably 90:10 to 30:70.

[Examples]

[0024] The following examples will describe the present invention in further detail. It should be noted, however, that the present invention is not limited to the examples in any way.

Examples 1 to 4 and Comparative Examples 1 to 6

[0025] Working fluids having the composition shown in Table 1 were prepared. The values shown in the table are in terms of parts by mass.

[0026] [Table 1]

Table 1

	Example				Comparative Example					
	1	2	3	4	1	2	3	4	5	6
Water	89.84	83.54	92.79	89.84	100.00	20.00	88.36	93.09	83.54	90.09
Water soluble polymeric compound 1	3.00	7.00	-	3.00	-	-	-	-	-	7.00
Water soluble polymeric compound 2	0.25	-	0.30	0.25	-	-	-	-	-	-
Water soluble polymeric compound 3	-	-	-	-	-	-	-	-	7.00	-
Nonionic surfactant 1	4.00	7.50	4.00	-	-	-	-	4.00	7.50	-
Nonionic surfactant 2	-	-	-	4.00	-	-	-	-	-	-
Propylene glycol	-	-	-	-	-	80.00	-	-	-	-
Isononanoic acid	0.07	0.04	0.07	0.07	-	-	0.28	0.07	0.04	0.07
Dodecandioic acid	1.11	1.05	1.11	1.11	-	-	4.44	1.11	1.05	1.11
N-methyldiethanolamine	1.03	0.52	1.03	1.03	-	-	4.12	1.03	0.52	1.03
Cyclohexyldiethanolamine	0.70	0.35	0.70	0.70	-	-	2.80	0.70	0.35	0.70
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Resorce saving efficiency (water content)	A	A	A	A	A	C	A	A	A	A
pH	6.7	7.5	6.3	6.7	7.0	7.0	8.2	8.2	7.7	8.2

[0027]

Water soluble polymeric compound 1: Polyalkylene glycol derivative (manufactured by NOF CORPORATION; trade name: UNILUBE 75DE-3800; number average molecular weight: 18,200; weight average molecular weight: 18,500)

Water soluble polymeric compound 2: Polyacrylic acid (manufactured by NIPPON SHOKUBAI CO., LTD.; trade name: AQUALIC AS58; number average molecular weight: 106,000; weight average molecular weight: 798,000)

Water soluble polymeric compound 3: Sodium polyacrylate (manufactured by NIPPON SHOKUBAI CO., LTD.; trade name: AQUALIC DL365; number average molecular weight: 2,500; weight average molecular weight: 5,400)

Nonionic surfactant 1: EO-PO block copolymer having clouding point of 30°C in 1% by mass aqueous solution and containing 79% by mass of oxypropylene groups based on the whole molecule (manufactured by Sanyo Chemical Industries, Ltd.; trade name: NEWPOL PE62; number average molecular weight: 2,500)

Nonionic surfactant 2: EO-PO block copolymer having clouding point of 56°C in 1% by mass aqueous solution and containing 59% by mass of oxypropylene groups based on the whole molecule (manufactured by Sanyo Chemical Industries, Ltd.; trade name: NEWPOL PE74; number average molecular weight: 3,400)

The number average molecular weight and weight average molecular weight herein are determined by GPC (gel permeation chromatography) using sodium polyacrylate as a molecular weight standard sample.

[0028] Evaluation ratings for resource saving efficiency (water content) in Table 1 are as follows:

- A: Water content is 80% by mass or more
- B: Water content is 40% by mass or more and less than 80% by mass
- C: Water content is less than 40% by mass

[Abrasive grain dispersion stability test]

[0029] An abrasive grain dispersion stability test was conducted on each of the obtained working fluids by the following method:

Abrasive grains (GC#1000 manufactured by FUJIMI INCORPORATED) and the working fluid were mixed in a mass ratio of 1:1 to prepare a slurry. The slurry (100 mL) immediately after the preparation was placed in a cylinder (inside diameter: 28 mm; inside volume: 120 mL) and allowed to quiescently stand at 30°C for 5 hours. Then the proportion of the separated liquid layer (the working fluid layer which the abrasive grains were separated) was calculated by the following equation:

$$\text{Proportion of separated liquid layer (\%)} = (\text{Volume of the separated liquid layer}) / (\text{Volume of the whole mixture}) \times 100$$

The results are shown in Table 2.

[Fluidity test for sedimented abrasive grains]

[0030] Slurry was prepared in the same manner as that in the abrasive grain dispersion stability test. The slurry (100 mL) was placed in a cylinder (inside diameter: 28 mm; inside volume: 120 mL) and allowed to quiescently stand at 30°C for 24 hours. Then the cylinder was inverted and, 1 hour after the inversion, the amount (mL) of the abrasive grains that flew from the sedimented layer was measured. The results are shown in Table 2.

[Viscosity of slurry]

[0031] Abrasive grains (GC#1500 manufactured by FUJIMI INCORPORATED) and the working fluid were mixed in a mass ratio of 1:1 to prepare a slurry. The slurry (100 mL) immediately after the preparation was measured for its viscosity at 25°C using a B-type rotary viscometer (model TVB-10 manufactured by TOKI SANGYO CO., LTD.). The results are shown in Table 2.

[Reciprocal dynamic friction test]

Test conditions:

[0032]

Tester: Model F-2100 manufactured by ORIENTEC Co., LTD.

Ball: 3/16 in. (SUJ2)

Tested plate: polycrystalline silicon

Sliding speed: 20 mm/s

Sliding length: 2 cm

Load: 1.96 N

A reciprocal dynamic friction test was carried out under the above test conditions to measure the frictional coefficient. The results are shown in Table 2.

[0033] [Table 2]

Table 2

	Example				Comparative Example					
	1	2	3	4	1	2	3	4	5	6
Abrasive grain dispersion stability test (%)	8.5	14.5	16.0	8.0	54.0	14.0	54.0	54.0	45.0	25.0
Fluidity test for sedimented abrasive grains (mL)	53	47	45	52	0	0	0	45	50	40
Viscosity of slurry (mPa·s)	84	110	140	82	-	110	-	-	-	200
Reciprocal dynamic friction test (frictional coefficient)	0.11	0.12	0.10	0.11	0.39	0.27	0.08	0.09	0.10	0.12

[0034] As is apparent from Table 2, the working fluid according to Examples has good abrasive grain dispersion stability and gives a slurry with low viscosity. Therefore, when used in wire saw working according to the loose abrasive grain method, high working accuracy and high working efficiency may be achieved. Additionally, when stirring of the slurry is kept stopped, the sedimented abrasive grains may be easily dispersed again.

Further, the working fluid according to Examples gives a frictional coefficient of as low as 0.10 to 0.12 when tested by the reciprocal dynamic friction test. Accordingly, when used in a method using a fixed abrasive grain wire saw, the working fluid can suppress detachment of the fixed abrasive grains and, also, can reduce cutting resistance.

[Industrial applicability]

[0035] The working fluid for hard and brittle materials according to the present invention, when used in a loose abrasive grain method, allows a slurry thereof to be supplied into the working gap sufficiently and has excellent abrasive grain dispersion stability and excellent properties to wash (disperse) cutting swarf. When used in a method using a fixed-abrasive grain wire saw, the working fluid rarely causes detachment of the fixed abrasive grains and has excellent properties to wash (disperse) cutting swarf. Additionally, the working fluid can solve the environmental problem and problem of safety.

Claims

1. A working fluid for hard and brittle materials comprising the following components (A) to (C):

(A) water,

(B) a water soluble polymeric compound having an oxygen-containing group and a number average molecular weight of 6,000 to 3,000,000, and

(C) a nonionic surfactant having a clouding point of 10 to 70°C in a 1% by mass aqueous solution.

EP 2 415 853 A1

2. The working fluid for hard and brittle materials as recited in claim 1, wherein the oxygen-containing group of the component (B) is a group selected from a carboxyl group, a hydroxyl group, an oxyethylene group and an oxypropylene group.
- 5 3. The working fluid for hard and brittle materials as recited in claim 1 or 2, wherein the component (B) is a carboxylic acid-based polymeric compound and/or an alkylene glycol-based polymeric compound.
4. The working fluid for hard and brittle materials as recited in claim 3, wherein the component (B) is a combination of a carboxylic acid-based polymeric compound and an alkylene glycol-based polymeric compound.
- 10 5. The working fluid for hard and brittle materials as recited in any one of claims 1 to 4, wherein the component (C) is a nonionic surfactant containing an oxypropylene group and having a number average molecular weight of 70 to 5,000.
- 15 6. The working fluid for hard and brittle materials as recited in any one of claims 1 to 5, wherein the content of the component (A) is 50 to 99% by mass, the content of the component (B) is 0.01 to 30% by mass, and the content of the component (C) is 0.1 to 20% by mass, based on the total amount of the working fluid.
- 20 7. The working fluid for hard and brittle materials as recited in any one of claims 1 to 6, wherein the working fluid has a pH of 2 to 10.
8. The working fluid for hard and brittle materials as recited in claim 7, wherein the working fluid contains an acid component and an alkaline component, and wherein the pH has been adjusted by controlling a blending ratio of the acid and alkaline components.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2010/054813

A. CLASSIFICATION OF SUBJECT MATTER <i>C10M173/02</i> (2006.01)i, <i>B24B37/00</i> (2006.01)i, <i>C10M107/28</i> (2006.01)n, <i>C10M107/34</i> (2006.01)n, <i>C10M145/14</i> (2006.01)n, <i>C10M145/26</i> (2006.01)n, <i>C10N20/00</i> (2006.01)n, <i>C10N20/04</i> (2006.01)n, <i>C10N30/00</i> (2006.01)n, According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) <i>C10M173/02</i> , <i>B24B37/00</i> , <i>C10M107/28</i> , <i>C10M107/34</i> , <i>C10M145/14</i> , <i>C10M145/26</i> , <i>C10N20/00</i> , <i>C10N20/04</i> , <i>C10N30/00</i> , <i>C10N40/32</i>		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2010 Kokai Jitsuyo Shinan Koho 1971-2010 Toroku Jitsuyo Shinan Koho 1994-2010		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y A	JP 2003-82380 A (Dai-Ichi Kogyo Seiyaku Co., Ltd.), 19 March 2003 (19.03.2003), claims; paragraph [0030]; example 2 & CN 1405287 A & EP 1291408 A1 & KR 10-2003-0022066 A & US 6673754 B1	1-3, 5-8 4
Y A	WO 1999/051711 A1 (Kao Corp.), 14 October 1999 (14.10.1999), claims; page 5, line 24 to page 6, line 3; example 1 & DE 19983092 T1 & GB 2351294 A & JP 2000-44974 A & JP 11-323376 A & JP 11-286693 A & US 6383991 B1	1-3, 5-8 4
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.		
* Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family		
Date of the actual completion of the international search 07 June, 2010 (07.06.10)		Date of mailing of the international search report 15 June, 2010 (15.06.10)
Name and mailing address of the ISA/ Japanese Patent Office		Authorized officer
Facsimile No.		Telephone No.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2010/054813

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y A	JP 10-110183 A (Kyoisha Chemical Co., Ltd.), 28 April 1998 (28.04.1998), claims; paragraphs [0002], [0007], [0011] (Family: none)	1-3, 5-8 4
Y A	JP 2000-160185 A (Kyodo Yushi Co., Ltd. et al.), 13 June 2000 (13.06.2000), claims; paragraphs [0007], [0014] to [0018] (Family: none)	7-8 1-6
Y A	JP 10-324889 A (Neos Co., Ltd.), 08 December 1998 (08.12.1998), claims; paragraphs [0002], [0013], [0021]; examples 1 to 9 (Family: none)	1-3, 5, 7-8 4, 6
A	JP 2003-82335 A (Yushiro Chemical Industry Co., Ltd.), 19 March 2003 (19.03.2003), claims; test example 3 (Family: none)	1-8

Form PCT/ISA/210 (continuation of second sheet) (July 2009)

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2010/054813

Continuation of A. CLASSIFICATION OF SUBJECT MATTER
(International Patent Classification (IPC))

C10N40/32(2006.01)n

(According to International Patent Classification (IPC) or to both national
classification and IPC)

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- JP H11100590 B [0006]
- JP 2002285186 A [0006]
- JP H0860176 B [0006]
- JP H11302681 B [0006]