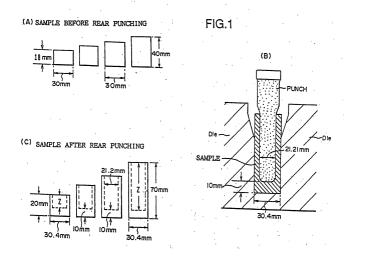
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### (54) AQUEOUS LUBRICANT FOR PLASTIC WORKING OF METALLIC MATERIAL AND METHOD OF LUBRICANT FILM PROCESSING

(57) An aqueous lubricant used for plastic working of metallic material which comprises (A) a water soluble inorganic salt, (B) lubricative agent selected from molybdenum disulfide and graphite and (C) wax, wherein the components are dissolved or dispersed in water, and wherein a concentration ratio (weight ratio) of (B) /(A) is in the range of  $1.0 \sim 5.0$  and (C) / (A) is  $0.1 \sim 1.0$ . And a method of lubricative film processing on a metallic material, in that the lubricative film formed by applying the

aqueous lubricant is in a dried weight of  $0.5 \sim 40 \text{g/m}^2$ . A water soluble inorganic salt (A) is preferably selected from the group consisting of a sulfate, a silicate, a borate, a molybdete and a tungstate. The wax (C) is preferably a natural wax or a synthetic wax which is dispersed in water and has a melting point of  $70 \sim 150^{\circ}$ C. The aqueous lubricant can be used for imparting excellent lubricity with ease to the surface of a metal having no chemical conversion layer formed thereon.



### Description

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#### FIELD OF THE INVENTION

<sup>5</sup> **[0001]** This invention relates to an aqueous lubricant used for plastic working of metallic material such as iron and steel, stainless steel, titanium, aluminum and others, wherein the surface of the metallic material has not been given any chemical conversion treatment. Also, it relates to a process of using the lubricant.

**[0002]** Being described in more detail, this invention relates to an aqueous lubricant used for producing a lubricative film suitable for plastic deforming work such as forging, wire drawing, tube drawing and others, on the surface of the metallic materials such as iron and steel, stainless steel, titanium, aluminum and others, wherein the surface of the metallic material has not been subjected to any chemical conversion treatment.

### BACKGROUND ART

<sup>15</sup> **[0003]** When cold plastic working are performed on the metallic material such as iron and steel, stainless steel and others, lubricative film are generally provided on the surface of the metallic material in order to prevent burning defects and galling defects which are arisen by metallic contact between the metallic material and tool.

**[0004]** Regarding the lubricative film being provided on the metal surface, there are lubricative film in which lubricative agent is made to adhere physically on the metal surface and other type of lubricative film in which chemical conversion lower are produced on the metal surface provided by the chemical conversion to the chemical co

- layer are produced on the metal surface previously by chemical conversion treatment of the metallic material and then lubricative agent are applied on the chemical conversion layer.
   [0005] The lubricative agent being adhered physically on the metal surface are used generally for cold working of slight amount of reduction since adhesive power of these are inferior than the adhesive power of the lubricative agent being applied on the chemical conversion layer.
- <sup>25</sup> **[0006]** In using the chemical conversion film, phosphate film or oxalate film are provided on the metal surface, which has a role as a carrier for the lubricative agent being applied on it. The lubricative film of this type are constructed by 2 layers, the carrier layer and the lubricative agent layer, and shows very excellent resistance against burning defect of the metallic material. And are used in a wide range of the cold working such as wire drawing, tube drawing, forging and others. And besides in the field of the cold working of heavy amount of reduction, it is widely used to provide a
- <sup>30</sup> phosphate film or oxalate film, and a lubricative agent are applied on that. [0007] The lubricative agent applied on the chemical conversion layer may be divided into two groups in terms of the usage. The first group includes a lubricative agent to be mechanically adhered onto the chemical conversion layer and the second group includes a lubricative agent which reacts with the chemical conversion layer.
- [0008] The first group of lubricative agent includes those being prepared by using mineral oil, vegetable oil or synthetic oil as base oil and containing an extreme pressure additive in the base oil, also includes other one being prepared by dissolving a solid lubricative agent, such as graphite and molybdenum disulfide, together with a binder component into the water. These are adhered and then dried.

**[0009]** These lubricative agent of the first group may have advantages of easy for handling the solution since they may be used simply by means of spray coating or dipping coating. However, as they have just a low lubricative properties, they tend to be used for a case where slight amount of deformation of the metallic material is required.

40 erties, they tend to be used for a case where slight amount of deformation of the metallic material is required. [0010] On the other hand, in the second group of the lubricative agent, a reactive soap such as sodium stearate is used for a cold working where high lubricative property is required. The reactive soap reacts with the chemical conversion layer and provides a layer of high lubricative property.

[0011] However, since the reactive soap cause a chemical reaction, composition control of the solution, temperature control for the chemical reaction and the renewal control of the deteriorated solution by discharging of the waste from the solution, etc, become very important during the process.

**[0012]** Recently, it is a big issue to reduce waste products from the industries for global environmental protection. And therefore, new lubricative agent and new lubricative process which do not discharge waste products have been highly desired. Also, some new processes which enable to simplify the complex control of the process and the solution in the above explained second group have been further desired.

**[0013]** In order to solve problems as described above, JP52-20967A, wherein a lubricant composition containing water soluble polymer or its aqueous emulsion as the base component, a solid lubricant and a film-forming agent has been disclosed. However, no composition which has the same degree of preferable effect as in the conventional process of using a chemical conversion layer has been obtained.

<sup>55</sup> **[0014]** In order to solve the problems as described above, another prior art of JP10-8085A has been disclosed. This prior art relates to an aqueous lubricant used for plastic working of metallic material in which (A) water soluble inorganic salt, (B) solid lubricative agent, (C) at least one oil selected from a group consisting of mineral oil, animal oil, vegetable oil and synthetic oil, (D) surface active agent and (E) water are well dispersed and emulsified homogeneously. However,

the lubricant according to this prior art is too unstable to use in an industry since it has to keep to emulsify the oil component, and is not showing a stable properties.

**[0015]** As another prior art, an invention of JP2000-63880A can be cited. This prior art is directed to a lubricant used for plastic working of metallic material comprising (A) synthetic resin, (B) water soluble inorganic salt and water, wherein

the ratio of (B)/(A) by weight in solid state is in a range from 0. 25/1 to 9/1 and the synthetic resin is kept dissolved or dispersed in the composition. However, this composition is also not stable to show a high lubricative properties in cold working of heavy amount of reduction, since its main component is the synthetic resin.

**[0016]** Therefore, it is an object of this invention to provide an aqueous lubricant used for plastic working of metallic material and a method of lubricative film processing, in which the metallic material has not been subjected to any chemical conversion treatment, and in which the problems existing in the conventional process may be solved and the problems in the global environmental protection may also be improved and is applicable to many sorts of metallic materials.

### DISCLOSURE OF THE INVENTION

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**[0017]** The inventors have investigated for solving the problems described above and have found that the excellent lubricative properties can be obtained by the aqueous solution containing water soluble inorganic salt, lubricative agent being selected from molybdenum disulphide and graphite, and wax at the specific ratio. Further, they have found out a method of lubricative film processing on the metal surface in saving the treating energy and in saving the treating space.

**[0018]** Namely, the present invention is an aqueous lubricant used for working the metallic material which contains (A) water soluble inorganic salt, (B) lubricative agent being selected from molybdenum disulphid and graphite, and (C) wax, and these components are dissolved or dispersed in water and weight ratio in solid state of (B)/(A) is in the range of  $1.0 \sim 5.0$  and a weight ratio in solid state of (C)/(A) is in the range of  $0.1 \sim 1.0$ .

[0019] It is preferable that (A) as above is one or more water soluble inorganic salt being selected from a group of sulfate, silicate, borate, molybdate and tungstate, and is preferable that wax as above is water dispersed natural wax or synthetic wax having melting point between 70~150°C.

**[0020]** Also, the present invention is a method of processing of the lubrication film of  $0.5 \sim 40$ g/m<sup>2</sup> as adherent weight on the surface of the metallic material wherein the aqueous lubricant of above is applied to the cleaned surface of the

<sup>30</sup> metallic material and is then dried. It is preferable that the surface of the metallic material is previously cleaned by one or more process selected from a group of shot blasting, sand blasting, alkaline degreasing and acid cleaning, and also preferable that the aqueous lubricant is applied on the surface of the metallic material after the metallic material is heated to 60~ 100°C.

#### 35 BRIEF DESCRIPTION OF DRAWINGS

### [0021]

Fig 1 : Illustrative drawing of rear punching test.

40 Fig 2 : Illustrative drawing of spike test.

### BEST MODES FOR CARRYING OUT THE INVENTION

[0022] Now, the present invention is explained further in detail. The water soluble inorganic salt (A) used in the aqueous lubricant of the invention is contained in order to give hardness and strength to the coating film. For this purpose, it is required to have a property to be uniformly dissolved in the aqueous solution and to form a strong lubricative film after drying.

**[0023]** As the inorganic salt giving such property, it is preferable to use at least one selected from a group consisting of sulfate, silicate, borate, molybdate and tungstate. As the examples for the inorganic salt described above, sodium

- <sup>50</sup> sulfate, potassium sulfate, potassium silicate, sodium borate (sodium tetraborate), potassium borate (potassium tetraborate), ammonium molybdate, sodium molybdate and sodium tungstate may be given. Any of these salts may be used either alone or in combination of 2 or more salts.
  [0024] In the present invention one or more than one of the lubricative agent (B) being selected from molybdenum
- disulphide and graphite are used in order to enhance the lubricative agent (B) being selected from molybdenum disulphide and graphite are used in order to enhance the lubricative properties. They are contained in a form of being dispersed, and the known surfactant may be used when necessary.
  - **[0025]** The (B)/(A), namely the weight ratio in solid state of the water soluble inorganic salt (A) and the lubricative agent (B), is preferable to be in a range of  $1.0 \sim 5.0$ . And is more preferable to be  $2.0 \sim 4.0$ . When the ratio is less than 1.0, sliding properties of the lubricative film are decreased and not preferable.

[0026] However, when the ratio exceeds 5.0, the aqueous lubricant become unstable and is not preferable.

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**[0027]** As wax(C), it is preferable to use a natural wax or a synthetic wax, though there is no specific limitation in its chemical structure and the type. The wax may melt by a heat generated during the plastic deformation of the metallic material thereby improve the lubricative property of the coating layer. For this reason, it is preferable to use those having a melting point in a range of  $70 \sim 150^{\circ}$ C and being stable in aqueous lubricant and those not to weaken the

strength of the lubricative film so as to perform the preferable lubrication from the early stage of the plastic working. **[0028]** The practical examples for the wax may include paraffin wax, micro crystalline wax, petrolatum wax, fisher tropsch wax, polyethylene wax, polypropylene wax, carnauba wax, montane wax and the like. These waxes are preferably combined with another component and contained in a form of water dispersion or water emulsion in the aqueous lubricant of the invention.

**[0029]** The (C)/(A), namely the weight ratio in solid state of water soluble inorganic salt (A) and the wax (C) is preferably in a range of  $0.1 \sim 1.0$ , and more preferably in a range of  $0.2 \sim 0.8$ . When the ratio is less than 0. 1, sliding property of the lubricative film may be insufficient, while the adhesive performance of the coating layer may be insufficient when the ratio is more than 1.0.

<sup>15</sup> **[0030]** It is still possible to add further another oil or another solid lubricative matter to the aqueous lubricant of this invention in cold working with heavy amount of deformation.

**[0031]** When a surface active agent is required for dispersing the lubricative matter and the wax in the aqueous lubricant, any surface active agent of nonionic, anionic, amphoteric and cationic type may be used. Although being not limited, the nonionic surface active agent may include polyoxyethylene alkyle ether, polyoxyalkylene(ethylene and/or

20 propylene) alkyl phenyle ether, polyoxyethylene alkyl ester comprising polyethylene glycol (or ethylene oxide) and higher fatty acid(C12~C18, for example), plyoxyethylene sorbitan alkyl ester comprising sorbitan, polyethylene glycol and higher fatty acid (C12~C18, for example).

**[0032]** Although being not limited, the anionic surface active agent may include fatty acid salts, sulfuric acid ester salt, sulfonate salt, phosphoric acid ester salt, and dithiophosphoric acid ester salt. Although being not limited, the amphoteric surface active agent may include carboxylates either in amino acid configuration or in betaine configuration, sulfuric acid ester salt, sulfonate salt, phosphoric acid ester salt.

**[0033]** Although being not limited, the cationic surface active agent may include amine salt of fatty acid, quaternary ammonium salt and the like.

- **[0034]** Each of these surface active agent may be used either alone or in combination of two or more of them.
- <sup>30</sup> **[0035]** Aqueous lubricant of the present invention may further be applied as a lubricant for cold working (wire drawing, tube drawing, forging, etc) when the metallic materials of iron and steel, stainless steel, copper or copper alloy, aluminum or aluminum alloy, titanium or titanium alloy are already coated by known process of forming the phosphate layer (zinc phosphate, manganese phosphate, iron phosphate, tin phosphate, etc), oxalte layer (iron oxalate, etc), cryolite and calcium aluminate layer.
- <sup>35</sup> **[0036]** Shape of the metallic material is not especially limited, and not only bar and block but also shaped product being produced by hot forging (gear, shaft, etc) may be used.

**[0037]** According to a method of the lubricative film. processing in this invention, a purified surface of the metallic material is made to contact with the aforementioned aqueous lubricant and then dried, and produce the lubricative film of  $0.5 \sim 40 \text{g/m}^2$  on the surface of the metallic material. Thus the processing of the lubricative film of the invention is non-reactive type. The amount of the lubricative film produced on the surface of the metal may be adjusted according

- to the degree of deformation of the plastic cold working. And it is more preferable to be in a range of 2~20g/m<sup>2</sup>. **[0038]** When it is less than 0.5g/m<sup>2</sup>, the lubricity becomes insufficient. When more than 40g/m<sup>2</sup>, although special problems may not arise in lubricity, dregs may appear in the working and the cavity provided on the surface of the tool may be crammed by arisen dregs. The amount of the lubricative film may be calculated from the surface area of the
- <sup>45</sup> metallic material and the weight difference before and after the treatment.
   [0039] The weight concentration of the components are adjusted in order to control the amount of the lubricative film as above. In many cases, treatment solution may be obtained by diluting the concentrated aqueous lubricant by water. The type of the water used for this dilution is not limited, but deionized water or distilled water are preferable.
   [0040] In the processing of the lubricative film of the invention, surface cleaning of the metallic material is preferable
- to be carried out by one or more cleaning step being selected from shot blasting, sand blasting, alkali degreasing and acid cleaning. The main purpose of these cleaning is to remove a oxide scale being grown in the annealing or to remove a contamination of oil or others.

**[0041]** Recently, the reduction of the desposal of the waste water has been desired from the environmental point of view. In this invention, waste water may be possible to decrease to zero, for example, by shot blasting for cleaning the surface and by producing the lubricative film using the aqueous lubricant of the invention.

**[0042]** There are no specific limitation in the method of applying the aqueous lubricant of the invention to the surface of the metallic material. And dipping method, flow coat method, spray method and other method can be used. The application is sufficient when the surface is sufficiently covered by the aqueous lubricant, and there is no restriction in

applying time.

[0043] After the application, it is necessary that the aqueous lubricant is to be dried. Drying may be done by keeping it under the ordinary temperature, and it may also be preferable by keeping it at  $60 \sim 150^{\circ}$ C for  $10 \sim 60$  minutes. [0044] It is preferable that the aqueous lubricant is applied after heating the metallic material to  $60 \sim 100^{\circ}$ C, in order to increase the drying efficiency. Also, it is preferable to apply the aqueous lubricant after being heated to  $50 \sim 90^{\circ}$ C. [0045] Thus, drying efficiency may be much improved and the loss of heat energy may be much decreased.

**EXAMPLES** 

10 [0046] The advantageous effect of this invention will be explained more practically by showing embodiment examples and comparative examples.

(Sample for rear punching test)

15 [0047] Serious of steel rod samples of JIS S45C being spherodizing annealed, obtained in the market, having a diameter of 30mm and having a serious of heights in 18~40mm as shown in Fig (A) in which height of each rod are different in 2mm each other.

(Sample for spike test)

[0048] Steel rod samples of JIS S45C being spherodizing annealed, obtained in the market and having a diameter of 25mm and having a height of 30mm.

(Treating Process)

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#### [0049]

- Process A
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- ① Degreasing : using degreasing agent on the market (FINE CLEANER R 4360, by Nihon Parkerizing Co., Ltd), concentration :20g/L, temperature : 60°C, dipping time : 10 minutes.
- (2) Washing : by tap water, 60°C, dipping for 30 sec.
- (3) Lubricating treatment : contacting with lubricant, at 60°C, dipping for 10 sec.
- (4) Drying : 80°C, for 3min.

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- Process B
  - (1) Shot blasting : Particle diameter : 0.5mm, treating for 5 min.
  - (2) Washing : by tap water, 90°C, dipping for 90sec.
  - ③ Lubricating treatment :contacting with lubricant at 70°C, dipping for 5 sec.
  - ④ Drying : room temperature (air blow), for 3 min.

(Rear Punching Test)·····Fig. 1

45 [0050] Series of steel rod samples in Fig. 1 (A) are cold worked by 200 ton crank press in Fig. 1 (B) to produce series of cup shaped products shown in Fig. 1 (C). In each punching, 10mm of bottom end was left, and the reduction of the sectional area was 50%. The defects on the inner surface of cup are inspected, and the maximum depth (Zmm) of cup for which no defects are observed are shown as punch depth (mm) in Table 1. In this test, die material is JIS SKDII, punch material is JIS HAP40, punch diameter is 21. 21mm, punching is 30 stroke/min.

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(Spike Test) ·····Fig. 2

[0051] Spike test has been carried out in the same way as show in JP5-7969A.

Die (1) has an inner surface of the funnel like shape. Rod sample (2) are set on the top of the die (1) as in Fig. 2 (A), 55 then being pressed and the bottom of the sample (2) are forced to move into the funnel hole of the die (1) as shown in Fig. 2 (B). By this process, spike having the shape corresponding to the funnel are produced. The height of the formed spike are shown as spike height in Table 1. The lubricating is excellent when the spike has a large spike height.

(Embodiment example 1)

**[0052]** Aqueous lubricant 1 as below (containing 1 wt % of nonionic surfactant for dispersion) was used and treated in Process A above.

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Aqueous lubricant 1

#### [0053]

 water soluble inorganic salt : sodium tetraborate, lubricative agent : molybdenum disulfide wax: polyethylene wax ratio (B/A) : 3.0 ratio (C/A) : 0.4
 amount of produced film, g/m<sup>2</sup> : 15

(Embodiment example 2)

[0054] Aqueous lubricant 2 as below (containing 1 wt % of nonionic surfactant for dispersion) was used in Process <sup>20</sup> B above.

Aqueous lubricant 2

### [0055]

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water soluble inorganic salt : sodium tetraborate lubricative agent : graphite wax : polyethylene wax ratio (B/A) : 2.0 ratio (C/A) : 0.8 amount of produced film,  $g/m^2$  : 15

(Embodiment example 3)

<sup>35</sup> **[0056]** Aqueous lubricant 3 as below (containing 1 wt % of nonionic surfactant for dispersion) was used in Process A above.

Aqueous lubricant 3

## 40 [0057]

water soluble inorganic salt : sodium silicate lubricative agent : graphite wax : polyethylene wax ratio (B/A) : 1.0 ratio (C/A) : 1.0 amount of produced film, g/m<sup>2</sup> : 15

(Embodiment example 4)

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**[0058]** Aqueous lubricant 4 as below (containing 1 wt % of nonionic surfactant for dispersion) was used in Process A above.

Aqueous lubricant 4

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[0059]

water soluble inorganic salt : sodium tungstate,

lubricative agent : molybdenum disulfide wax : paraffin wax ratio (B/A) : 4. 0 ratio (C/A) : 0.1 amount of produced film : g/m<sup>2</sup> : 15

(Embodiment example 5)

[0060] Aqueous lubricant 5 as below (containing 1 wt % of nonionic surfactant for dispersion) was used in Process <sup>10</sup> B above.

Aqueous lubricant 5

[0061]

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water soluble inorganic salt : potassium sulfate
lubricative agent : molybdenum disulfide
wax : paraffin wax
ratio (B/A) : 3.0
ratio (C/A) : 0.5
amount of produced film, g/m<sup>2</sup> : 15
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(Comparative example 1)

<sup>25</sup> **[0062]** Aqueous lubricant 6 as below (containing 1 wt % of nonionic surfactant for dispersion) was used in Process A above.

Aqueous lubricant 6

#### 30 [0063]

water soluble inorganic salt : potassium sulfate wax : paraffin wax ratio (C/A) : 0.1 amount of the produced film,  $g/m^2$  : 10

(Comparative example 2)

**[0064]** Aqueous lubricant 7 as below (containing 1 wt % of nonionic surfactant for dispersion) was used in treating process B above.

Aqueous lubricant 7

### [0065]

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water soluble inorganic salt : potassium sulfate lubricative agent : molybdenum disulfate ratio (B/A) : 0.5 amount of produced film, g/m<sup>2</sup> : 15

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(Comparative example 3)

[0066] Treatment was carried out in Process C as below.

55 · Process C

① Degreasing : using degreasing agent on the market (FINE CLEANER <sup>R</sup> 4360, by Nihon Parkerizing Co., Ltd), concentration : 20g/L, temperature : 60°C, dipping time : 10 min

② Washing : by tap water, room temperature, dipping for 30 sec.

③ Chemical conversion treatment : using chemical conversion agent containing zinc phosphate obtained in the market (PALBOND <sup>R</sup> 181X, by Nihon Parkerizing Co., Ltd), concentration : 90g/L, temperature : 80°C, dipping time : 10 min.

④ Washing : by tap water, room temperature, dipping for 30 sec,

(5) Soap treatment : Reactive soap lubricating matter on the market (PALUBE <sup>R</sup> 235, by Nihon Parkerizing Co., Ltd), concentration : 70g/L, temperature : 80°C, dipping for 5 min

6 Drying : 80°C, 3 min

### 10 (Comparative example 4)

**[0067]** Aqueous lubricant 8 as below was used in treating process A above.

Aqueous lubricant 8

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### [0068]

water soluble inorganic salt : borax : 10 %

- lubricative agent : calcium stearate : 10% oil constituent : palm oil ; 0.5 % surfactant : polyoxyethylene alkyl alcohol ; 1 %
- others : water amount of produced film,  $g/m^2$  : 10
- <sup>25</sup> (Comparative example 5)

**[0069]** Aqueous lubricant 9 as below (containing 1 wt % of nonionic surfactant for dispersion) was used in treating process A above

#### 30 Aqueous lubricant 9

### [0070]

water soluble inorganic salt : sodium tetraborate

35 synthetic resin : urethane resin

metallic salt of fatty acid : calcium stearate ratio in solid state (water soluble inorganic salt / synthetic resin) =2/2 ratio in solid state (calcium stearate / synthetic resin) =3/1 amount of produced film,  $g/m^2$  : 10

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**[0071]** Test results are shown in Table 1. It is clear from Table 1 that embodiment example  $1\sim5$  where aqueous lubricant for working of metallic material according to the present invention exhibit the excellent lubricity and simple and easy treating process.

**[0072]** Comparative example 1 where lubricative agent is not contained and comparative example 2 where wax is not contained are inferior in their lubricative properties.

[0073] In comparative example 3, where treatment was carried out in a conventional process of using the chemical conversion layer of phosphate and reactive soap, the lubricative property is as excellent as in the present invention. However, much waste matter may appear from the reaction of chemical conversion, and special complicated equipments become necessary in disposal of waste water and for controlling the aqueous lubricant, and the burden for keeping the environment become increase.

**[0074]** Also it is proved that the lubricity in spike test are inferior in comparative example 4 which is the same as those shown in JP10-8085A and in comparative example 5 which uses synthetic resin as main constituent and is the same as those shown in JP2000-63880A.

### 55 ADVANTAGE OF THE INVENTION

**[0075]** As it is clear from the description of above, it became possible to produce the film with the high lubricity in the simple and easy treatment by using the aqueous lubricant and the method of lubricative film processing of the

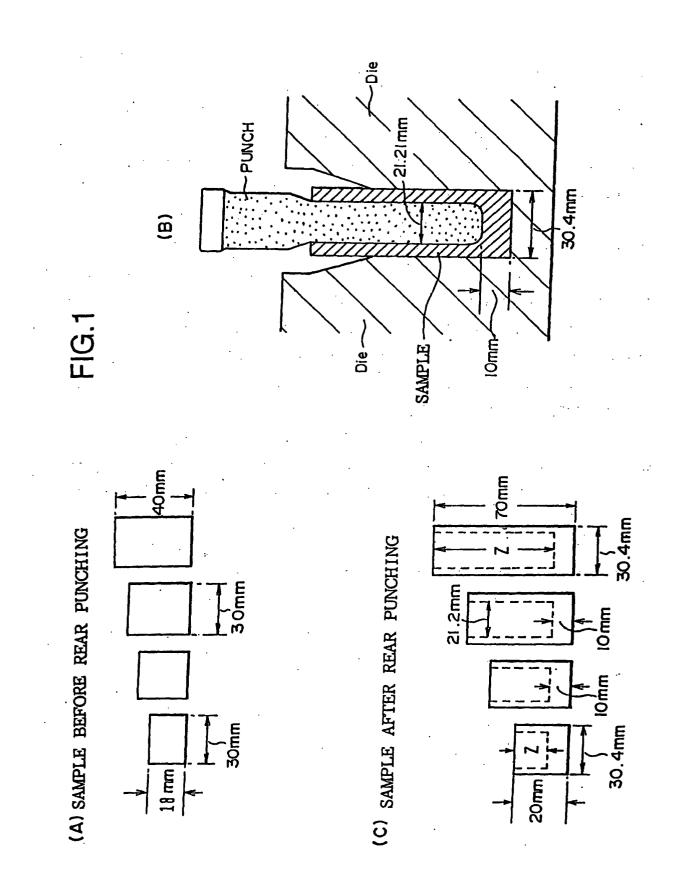
present invention. Also, the amount of arised waste matter was decreased and the preferable environmental protection became possible. Thus, this invention has a great industrial applicability.

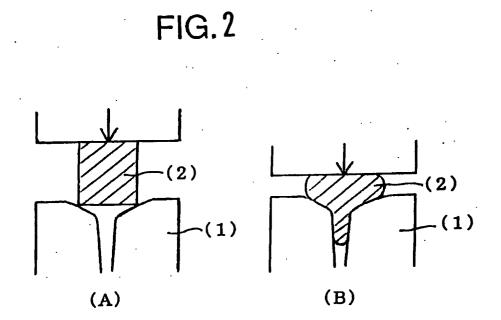
5		Number of step in treating process	treatment	punch depth (mm)	spike height (mm)
	embodiment example 1	4	application type	60	13.2
10	embodiment example 2	4	application type	60	13.2
	embodiment example 3	4	application type	60	13.2
15	embodiment example 4	4	application type	60	13.1
	embodiment example 5	4	application type	60	13.1
20	comparative example 1	4	application type	40	11.6
	comparative example 2	4	application type	40	11.7
25	comparative example 3	6	reactive type/ much waste matter	56	13.0
	comparative example 4	4	application type	56	12.5
30	comparative example 5	4	application type	56	12.6

Table 1

#### Claims

- <sup>35</sup> 1. Aqueous lubricant used for plastic working of metallic material which contains (A) water soluble inorganic salt, (B) one or more than one lubricative agent being selected from molybdenum disulfide and graphite, and (C) wax, and these components are dissolved or dispersed in water and weight ratio of (B) / (A) in solid state is in the range of 1.0~5.0 and weight ratio of (C)/(A) in solid state is in the range of 0.1~1.0.
- Aqueous lubricant used for plastic working of metallic material according to claim(1) wherein the water soluble inorganic acid is one or more than one water soluble inorganic salt being selected from a group of sulfate, silicate, borate, molybdete and tungstate.
- Aqueous lubricant used for plastic working of metallic material according to any of claim (1)~(2) wherein the wax is water dispersed natural wax or synthetic wax having melting point between 70~150°C.
  - 4. Method of lubricative film processing wherein aqueous lubricant according to any of claim (1)~(3) is applied to the cleaned surface of the metallic material and is dried, and produce the lubricative film of 0.5~40g/m<sup>2</sup> on the surface of the metallic material.
- 50
- 5. Method of lubricative film processing according to claim(4) wherein the cleaned surface is obtained by one or more than one cleaning step being selected from a group of shot blasting, sand blasting, alkali degreasing and acid cleaning.
- <sup>55</sup> **6.** Method of lubricative film processing according to any of claim (4) $\sim$ (5) wherein the aqueous lubricant according to any of claim (1) $\sim$ (3) is applied to the metallic material after the metallic material is heated to 60 $\sim$ 100 $^{\circ}$ C.





	INTERNATIONAL SEARCH REPO	RT	International application No.			
			PCT/J	P01/03639		
	A. CLASSIFICATION OF SUBJECT MATTER Int.Cl <sup>7</sup> ClOM159/06, ClOM125/00, ClOM125/10, B21J3/00					
According to	o International Patent Classification (IPC) or to both na	ational classification a	nd IPC			
	S SEARCHED					
Minimum documentation searched (classification system followed by classification symbols) Int.Cl <sup>7</sup> ClOMl0l/00-ClOMl77/00, B21J 3/00						
Jits	Documentation searched other than minimum documentation to the extent that such documents are included in the fields searchedJitsuyo Shinan Koho1922-1996Toroku Jitsuyo Shinan Koho1994-2001Kokai Jitsuyo Shinan Koho1971-2001Jitsuyo Shinan Toroku Koho1996-2001					
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) WPI/L						
C. DOCU	MENTS CONSIDERED TO BE RELEVANT	<u></u>				
Category*	Citation of document, with indication, where ap		ant passages	Relevant to claim No.		
x	X WO 99/64544 A1 (HENKEL CORPORATION), 16 December, 1999 (16.12.99), Claims 2-4, 13-17, page 12, line 31 to page 13, line 1 & JP 2000-63880 A			1		
P	JP 2000-309793 A (Nippon Parker 07 November, 2000 (07.11.00), Claims, 1, 6 (Family: none)			1		
Further	r documents are listed in the continuation of Box C.	See patent fami	ily annex.			
"A" docume consider earlier of date "L" docume cited to special "O" docume means "P" docume than the Date of the a	categories of cited documents: ant defining the general state of the art which is not red to be of particular relevance document but published on or after the international filing set which may throw doubts on priority claim(s) or which is establish the publication date of another citation or other reason (as specified) ant referring to an oral disclosure, use, exhibition or other ent published prior to the international filing date but later e priority date claimed citual completion of the international search	priority date and understand the pu document of part considered novel step when the do "Y" document of part considered to inv combined with or combined with or combined with or combined mailing of th	published after the international filing date or i not in conflict with the application but cited to principle or theory underlying the invention ticular relevance; the claimed invention cannot be l or cannot be considered to involve an inventive boument is taken alone ticular relevance; the claimed invention cannot be volve an inventive step when the document is one or more other such documents, such ng obvious to a person skilled in the art per of the same patent family the international search report 2001 (21 07 01)			
19 July, 2001 (19.07.01) Name and mailing address of the ISA/ Japanese Patent Office		31 July 2001 (31.07.01) Authorized officer				
Facsimile No		Telephone No.				

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT	International application No.				
	PCT/JP01/03639				
Box I Observations where certain claims were found unsearchable (Continuation	of item 1 of first sheet)				
This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:					
Claims Nos.: because they relate to subject matter not required to be searched by this Authority, namely:					
2. Claims Nos.: because they relate to parts of the international application that do not comply extent that no meaningful international search can be carried out, specifically:	with the prescribed requirements to such an				
3. Claims Nos.: because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).					
Box II Observations where unity of invention is lacking (Continuation of item 2 of					
This International Searching Authority found multiple inventions in this international ap					
The common mater to claims 1 to 6 is "an aqueous lubricant for use in plastic working of a metallic material which comprises (A) a water soluble inorganic salt, (B) a specific lubricating agent and (C) a wax, in a specific solid concentration ratio, wherein the components are dissolved or dispersed in water". The above common matter is not novel and therefore is not a special technical feature in accordance with PCT Rule 13.2, since a partial search has revealed that the matter is disclosed in WO, 99/64544, A1 (HENKEL CORPORATION) 16. Dec. 1999 (16. 12. 99). Claims 1 to 6 do not appear to have another common matter which may be a special technical feature. Accordingly, claims 1 to 6 are not a group of inventions which are so linked as to form a single general inventive concept.					
1. As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.					
2. As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.					
3. As only some of the required additional search fees were timely paid by the ap only those claims for which fees were paid, specifically claims Nos.:	plicant, this international search report covers				
<ul> <li>4. X No required additional search fees were timely paid by the applicant. Conseque search report is restricted to the invention first mentioned in the claims; it is co Claim 1</li> </ul>					
Remark on Protest       Image: The additional search fees were accompanied by the appleted in the payment of additional search fees were accompanied by the appleted in the payment of additional search fees were accompanied by the appleted in the payment of additional search fees were accompanied by the appleted in the payment of additional search fees were accompanied by the appleted in the payment of additional search fees were accompanied by the appleted in the payment of additional search fees were accompanied by the appleted in the payment of additional search fees were accompanied by the appleted in the payment of additional search fees were accompanied by the appleted in the payment of additional search fees were accompanied by the appleted in the payment of additional search fees were accompanied by the appleted in the payment of additional search fees were accompanied the payment of ad					

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