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3,843,529

METAL WORKING LUBRICANT COMPOSITIONS
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U.S. Cl. 252—30

8 Claims

ABSTRACT OF THE DISCLOSURE

A water-dispersible metal working lubricant comprising effective amounts of:

- (a) molybdenum disulfide
- (b) graphite
- (c) a binder mixture of carboxymethylcellulose and amine-functional silane
- (d) bentonite

SPECIFICATION

The present invention relates to a water-based lubricant. In one aspect, the invention relates to metal surfaces having a lubricant coating thereon.

Lubrication of metals during the working and forming processes is often necessary. The metal working lubricant should lower the friction, improve surface finish and protect "new" surfaces against atmospheric corrosion. Solid lubricants, such as molybdenum disulfide or graphite, are used as metal working lubricants. The use of such lubricants is limited in certain areas of metal working wherein burnishing of the lubricant into the metal surface cannot be tolerated. For example, lubricant contaminated surfaces present difficulties during plating operations.

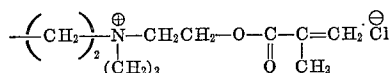
Thus, it is an object of the present invention to provide metal surfaces with an effective, but easily removed lubricant coating.

It is another object of the invention to provide novel aqueous lubricant compositions.

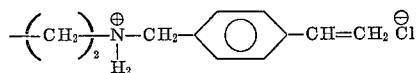
A further object of the invention is to provide metal articles with an adherent corrosion resistant coating.

These and other objects of the invention will be apparent to one skilled in the art upon reading the disclosure and claims.

The present invention provides an article comprising a solid metal surface, the surface having coating of a mixture comprising (a) a particulate solid lubricant consisting of a molybdenum disulfide-graphite mixture containing from about 50 to 80 percent by weight molybdenum disulfide and (b) a binder for the lubricant consisting essentially of 40 to 60 weight percent carboxymethylcellulose and 40 to 60 weight percent of an amine-functional silane of the formula $(\text{RO})_3\text{Si}(\text{CH}_2)_3\text{NHR}'$, hydrolyzates and water soluble partial condensates thereof in which R is an alkyl radical containing from 1 to 4 inclusive carbon atoms; and R' is selected from the group consisting of the hydrogen atom, the $(\text{CH}_2)_2\text{NH}_2$ radical, the



radical and the



radical; the weight ratio of lubricant (a) to binder (b) in the coating being in the range of from 1:1 to 3:1.

The invention also provides a lubricant comprising about 50 to 75 weight percent of the molybdenum disulfide-graphite mixture (a) and about 25 to 50 weight percent of the carboxymethylcellulose-silane binder (b)

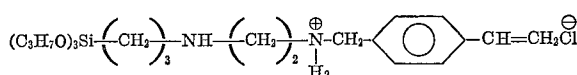
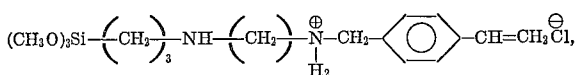
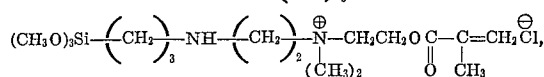
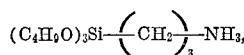
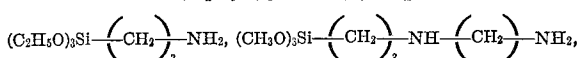
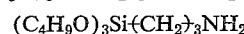
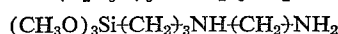
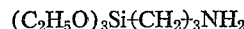
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described above; this being the composition of the coating on the metal article.

The use of the molybdenum disulfide-graphite mixture is considered essential in formulating the lubricants of the invention. When combined with the binder, the defined mixture gives superior performance as compared to formulations utilizing either graphite or molybdenum disulfide alone. The solid lubricant components are present in the lubricant composition as particles. While the particle size is not critical, for ease in forming coating dispersions, it is preferred that particles in the 1 to 100 micron diameter size range be utilized.

The silane-carboxymethylcellulose components provides a binder system for the lubricant particles in the form of a coating which is adherent to metal surfaces. These components are water-soluble, thus providing easy removal of the coating by washing. The binder system also minimizes burnishing of the metal by the solid lubricants during forming operations.

Silanes operable in the practice of the invention include



When formulated in aqueous lubricant dispersions, the alkoxy substituents hydrolyze to form $(\text{HO})_3\text{Si}\equiv$. It is possible for some of the hydroxyl groups to condense to form a low-molecular weight, water-soluble siloxane (SiOSi) polymer or partial condensate which retains alkoxy and/or hydroxyl-functionality and the defined amine-substituents. See U.S. Pat. 3,630,827 concerning these silanes.

Certain of the silanes, such as the gamma-amino-propylsilanes are commercially available. The quaternary salt of the aminoacrylate-functional silane is known and can be prepared by reaction of gamma-chloropropyltri-alkoxysilane with 2-(dimethylamino)ethyl methacrylate in the presence of sulfur and methyl iodide. Reaction in a solvent at about 100° C. gives good yields of the salt. The hydrochloride salt is also known and is obtained by reacting beta - aminoethyl - gamma - aminopropyltrialkoxysilane with vinylbenzylchloride.

The lubricant coating can be applied to any of the solid metals processed by techniques such as extruding, drawing and cold forging. Exemplary of such metals are steel, aluminum, copper, brass, bronze, titanium, tungsten and Monel metal. In addition to providing surface lubrication during the metal forming operation, the described coating provides protection against atmospheric corrosion prior to the metal working.

The lubricant coating can be applied as a paste or solvated dispersion by conventional processes such as troweling, dipping, brushing or spraying. Any solvent for carboxymethylcellulose can be used in formulating the pastes or dispersions. The solvent should be volatile to

the extent that it evaporates at room temperature or slightly above to leave a uniform coating or dry film of lubricant. Suitable solvents include water, methanol, ethanol, isopropanol and the like. For reasons of economy, safety and ease of handling, water is the preferred solvent.

A dispersion especially suitable for the dip-coating of metal articles contains about 15 to 30 weight percent of the described molybdenum disulphide-graphite mixture, about 5 to 7.5 weight percent carboxymethylcellulose, about 5 to 7.5 weight percent of an amine-functional silane of the formula $(\text{CH}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{NH}(\text{CH}_2)_2\text{NH}_2$ and about 0.5 to 2 weight percent bentonite; the remainder (53 to 74.5 weight percent) being water. The bentonite acts as a stabilizer for the dispersion as well as reducing foaming during mixing of the dispersion. Conventional additives, such as dyes, bactericides, corrosion inhibitors and the like, can also be used in formulating the above-described lubricant composition.

Metal articles are coated by dipping or immersing in the aqueous dispersion and allowing the coating to dry at room temperature. A thin, uniform, void-free lubricant film is obtained in about 6 to 8 hours at room temperature. Of course, the coating can be cured at elevated temperatures in a shorter period of time. The coating also provides protection from corrosion upon prolonged exposure to atmospheric conditions. The dry coating is strongly adhered to the metal, yet after serving its lubricating function, the coating residue is easily removed by washing in water.

The following examples are illustrative, and not intended to be limiting, of the invention delineated in the claims.

EXAMPLE 1

A mixture of 25 grams of molybdenum disulphide powder and 9.2 grams of graphite was added to 105 grams of

rusted equally. This demonstrates that all of the coating was removed by washing.

The endurance life of the above-described lubricant coating in a sliding steel-on-steel application was determined by use of a Falex test machine run in accordance with ASTM D-2625. Endurance life was reported as the time required for failure to occur at 1000 lb. load and at 290 r.p.m. Test pins dip-coated with the described aqueous lubricant and allowed to dry had an endurance life ranging from 158 to 219 minutes.

For purposes of comparison, a lubricant composition containing 34 grams of graphite (instead of the graphite/molybdenum sulfide mixture) was formulated with the same amounts of the other components described above. Pins coated with this lubricant had a wear life of about five minutes.

In another test, an Alpha LFW-1 test machine (described in U.S. 3,028,746) was operated at 72 r.p.m. under a load of 630 lbs. using a standard steel ring (R_c60) and a standard steel block (R_c30). The ring was coated with the lubricant of the invention described above. After 500 0cycles, the LFW-1 wear scar was 1.0 mm. and failure did not occur until after 138,220 cycles.

These data demonstrate the excellent lubrication obtained by use of the composition of the invention.

EXAMPLE 2

Lubricant compositions containing various silanes were formulated. The compositions contained 16.5 weight percent molybdenum disulphide, 6.1 weight percent graphite, 1.3 weight percent bentonite, 69.5 weight percent of a 10 percent aqueous solution of carboxymethylcellulose and 6.6 weight percent of the silane. Falex pins were dipped in the aqueous lubricants and the coating was allowed to air dry. The endurance life (as determined by the Falex test) for each of the coatings containing different silanes is listed below:

Silane present in lubricant	Average endurance life (minutes)
$(\text{C}_2\text{H}_5\text{O})_3\text{SiCH}_2\text{CH}_2\text{CH}_2\text{NH}_2$	239
$(\text{CH}_3\text{O})_3\text{Si}-\left(\text{CH}_2\right)_3-\text{NH}-\left(\text{CH}_2\right)_2-\text{N}^+\left(\text{CH}_3\right)_2-\text{CH}_2\text{CH}_2\text{O}-\text{C}(=\text{O})-\text{C}(\text{CH}_3)=\text{CH}_2\text{Cl}^-$	323
$(\text{CH}_3\text{O})_3\text{Si}-\left(\text{CH}_2\right)_3-\text{NH}-\left(\text{CH}_2\right)_2-\text{N}^+\left(\text{CH}_3\right)_2-\text{CH}_2-\text{C}_6\text{H}_4-\text{CH}=\text{CH}_2\text{Cl}^-$	190

10 percent aqueous solution of carboxymethylcellulose. During mixing of these components, 10 grams of



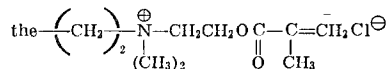
and 2 grams of bentonite were added. The homogeneous aqueous lubricant contained 22.6 weight percent lubricant particles, 6.9 weight percent carboxymethylcellulose, 6.6 of the described silane, 1.3 weight percent bentonite and 62.6 weight percent water.

Metal test panels (1" x 3") of aluminum and steel were dipped in the above dispersion and allowed to air-dry for about 4 hours. Uniform adherent, void-free coatings were obtained. Coated steel panels were placed in a Cyclic Environment Tester and subjected to wet conditions for 30 minutes, followed by dry conditions for 15 minutes for a total of three hours. The coated panels showed no evidence of rust. Uncoated steel panels were discolored and beginning to rust after three hours under the same conditions.

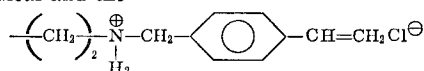
A portion of steel test panel was coated with the described dispersion and allowed to air dry for 24 hours. The coating was then removed by washing with water. The panel was exposed to atmospheric conditions (average humidity of 80 percent) for one month. The uncoated portion and coated/washed portion of the panel were

That which is claimed is:

1. A lubricant composition consisting essentially of
 - (a) about 50 to 75 percent by weight of a mixture of solid lubricant particles, the mixture containing from about 50 to 80 weight percent molybdenum disulphide, the remainder being graphite; and
 - (b) about 25 to 50 percent by weight of a binder consisting essentially of about 40 to 60 weight percent carboxymethylcellulose and about 40 to 60 weight percent of an amine-functional silane of the formula $(\text{RO})_3\text{Si}(\text{CH}_2)_3\text{NHR}'$, hydrolyzates and partial condensates thereof in which R is an alkyl radical containing from 1 to 4 inclusive carbon atoms; and R' is selected from the group consisting of the hydrogen atom, the $(\text{CH}_2)_2\text{NH}_2$ radical,



radical and the



radical.

2. The lubricant composition of claim 1 including a solvent for the binder (b).

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3. The lubricant composition of claim 2 wherein the solvent is water.

4. A lubricant composition in accordance with claim 3 consisting essentially of

- (a) about 15 to 30 weight percent of said mixture of solid lubricant particles;
- (b) about 5 to 7.5 weight percent carboxymethyl-cellulose;
- (c) about 5 to 7.5 weight percent of said amine-functional silane;
- (d) about 0.5 to 2 weight percent bentonite; and
- (e) about 53 to 74.5 weight percent water.

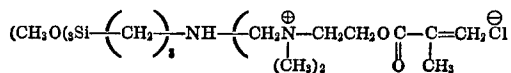
5. A lubricant composition in accordance with claim 4 wherein the silane is of the formula



6. A lubricant composition on in accordance with claim 4 wherein the silane is of the formula

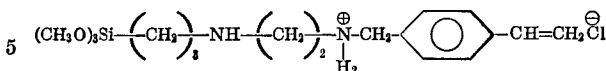


7. A lubricant composition in accordance with claim 4 wherein the silane is of the formula



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8. A lubricant composition in accordance with claim 4 wherein the silane is of the formula



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DANIEL E. WYMAN, Primary Examiner

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U.S. Cl. X.R.

72—42; 252—25, 28, 49.5, 389

UNITED STATES PATENT AND TRADEMARK OFFICE
CERTIFICATE OF CORRECTION

PATENT NO. : 3,843,529
DATED : October 22, 1974
INVENTOR(S) : ROBERT G. BERTRAND

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 5, line 23, that portion of the formula reading

$$\text{"(CH}_3\text{O)}_3\text{Si} \begin{array}{c} \oplus \\ \text{---} \end{array} \text{(CH}_2\text{)}_3\text{NH} \begin{array}{c} \oplus \\ \text{---} \end{array} \text{(CH}_2\text{)N-CH}_2\text{CH}_2\text{"}$$
 should read
$$\text{(CH}_3\text{)}_2$$

$$\text{-- (CH}_3\text{O)}_3\text{Si} \begin{array}{c} \oplus \\ \text{---} \end{array} \text{(CH}_2\text{)}_3\text{NH} \begin{array}{c} \oplus \\ \text{---} \end{array} \text{(CH}_2\text{)N-CH}_2\text{CH}_2 \text{--}$$

$$\text{(CH}_3\text{)}_2$$

Signed and Sealed this

twenty-seventh Day of January 1976

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks

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