

1

3,801,504
**NON-FLAMMABLE WATER BASED HOT FORGING
LUBRICATING COMPOSITIONS**
Aubrey L. Stone, Port Arthur, Tex., assignor to
Texaco Inc., New York, N.Y.
No Drawing. Filed Mar. 22, 1971, Ser. No. 126,892
Int. Cl. C10m 3/04, 3/14, 3/18
U.S. Cl. 252—23 9 Claims

ABSTRACT OF THE DISCLOSURE

Lubricant composition comprising:

- (a) water base,
- (b) clay thickener,
- (c) graphite,
- (d) borax,
- (e) sodium metasilicate,
- (f) non-clay thickener,
- (g) polyalkoxylated alkyphenols.

This invention concerns non-flammable water-based metal working lubricants. More particularly, this invention relates to the above lubricants containing alkali metal metasilicates and borax, and which have improved characteristics over compositions of the prior art.

BACKGROUND OF THE INVENTION

The metal industry is frequently used as a bellweather of industrial development and the affluence of a country. Upon first blush this seems rather anomalous since most metals have relatively little intrinsic value until they have been worked. That is, it is only after the metal has been forged, molded or otherwise shaped into useful forms that the value of the original, unworked metal appreciates manifold.

Forging is a process for increasing the density, toughness and strength of the metal by grain deformation. It can be performed while the metal is in the hot state or cold. Both processes have advantages and disadvantages. While cold forging upgrades metallurgical properties, hot forging requires the expenditure of less energy and permits greater deformation. Two primary factors which are especially important in hot forging operations are the rate of production and die life. The dies are very expensive and every precaution, including the choice of proper lubricants, is taken to extend die life. The use of lubricants is to minimize metal "pick-up" and wear. Improper lubrication can cause undesired metal slippage and can result in defective forgings.

However, the primary requirements of lubricants, particularly in hot forging operations, is to cool down the temperature of the workpiece and die as well as to wet the hot metal and thereby minimize frictional effects caused by the workpiece sliding over the die. Further requirements are to eliminate the hazards of fire and minimize air pollution. Inasmuch as the die temperatures during use can range from 300° F. to about 600° F., the use of conventional soap thickened mineral oil-based lubricants is both hazardous and ineffective since the oil base rapidly burns off leaving soap, which by itself is not a satisfactory lubricant. Water-based lubricants containing graphite, mica, molybdenum disulfide, while eliminating fire hazards, have not been entirely satisfactory in their wetting characteristics and have in many instances caused or accelerated corrosion.

In view of the problems encountered with both oil-based and water-based lubricants, a need exists to develop a water-based lubricant which, in addition to being non-flammable, has improved hot metal wetting characteristics and which minimizes or inhibits corrosion.

Therefore, it is an object of this invention to provide

2

water-based hot forging lubricant concentrates which have substantially improved wetting properties and which minimize or inhibit corrosion.

These objects, among many others, are achieved by the preparation of the improved non-flammable, hot forging composition where preparation is described below:¹

A portion of the water used in the finished hot forging concentrate, usually about one-quarter to two-thirds of the total water, is charged to an oil-free kettle or vessel of an appropriate size fitted with the usual heating, cooling and mixing means. All of the clay thickener is stirred into the kettle containing water until the clay is evenly dispersed. At the time all of the graphite used is added to the aqueous clay dispersion until a homogenous mixture is obtained. Ordinarily a previously and separately prepared aqueous gel comprising non-clay thickeners (such as sodium carboxymethylcellulose), borax and sodium metasilicate is added to the aqueous clay dispersion until a uniformly dispersed mixture is obtained. At this point, all of the surfactant is added with stirring followed by the addition of the remainder of the water which has been used as rinse of the separately prepared gel container. The black gelled mixture is stirred with circulation and put through a sieve of the desired pore size and stored for use.

The order of above blending procedure is critical to success in that varying the sequence of addition of the various components produces a lubricant possessing inferior stability.

(A) Clay thickeners.—The favored clay thickeners are selected from the group consisting of finely divided particles of the montmorillonite group of minerals. These are expanding lattice type minerals illustrated most commonly by bentonite and hectorite. The major difference between the former over the latter is that bentonite contains alumina as a major constituent while hectorite contains magnesia as a major component. More specifically, bentonite is a sodium aluminum silicate, while hectorite is a sodium magnesium-fluoro-litho-silicate. Usually the particle size of these clays is between 0.05 mm. and 0.01 mm. in diameter and they have an effective specific surface of the order of 200 to 400 square meters per gram.

Hectorite having a specific area of the order of 300 to 350 square meters per gram and whose dimensions when dispersed in solution are approximately 1 millimicron by 700 millimicrons is the preferred clay thickener.

(B) Non-clay thickeners.—These optional thickeners are referred to as organic thickeners and include alkylcelluloses, polymethylvinyl ether-maleic anhydride, alkali metal alkylcelluloses, and mixtures of one or more of these organic thickeners can also be used.

A combination of hectorite with sodium carboxymethylcellulose is the preferred thickening system when used in the ratio of about 6 parts by weight of hectorite to about 1 part by weight of sodium carboxymethylcellulose. Hectorite is preferred as the clay thickener because of its ability to be utilized in smaller concentrations than the bentonites to obtain the same degree of gelation and also because it exhibits a greater stability in aqueous solution.

(C) Fillers.—Finely divided graphite having an average particle size of less than 50 microns is preferred. Preferably the particle size will have a particle size of less than 25 microns and will have a low ash content (less than 2.5% by weight) when fired. A mixture of graphite ranging from about 16 to 29 parts per weight per hundred parts by weight of the finished grease is the preferred concentration. A mixture of up to 5 parts by weight of finely particulated calcium carbonate when used with 16 to 29

¹ The components of the lubricants are given in terms of parts by weight per hundred parts by weight of the finished grease, in order to more easily calculate the relative proportions of the components present.

parts by weight of graphite appears to improve the uniformity of the finished grease composition.

(D) Surfactant.—The surfactants which have proven to be the most useful of the commercially available materials are those alkoxyated derivatives of alkylated phenols containing an average of from 2 to 30 moles of alkylene oxide in the molecule, and where the alkylating group or groups contain from 4 to 12 carbon atoms per molecule of phenol. The favored surfactant is nonylphenol ethoxylated with an average of from about 2 to 30 ethoxylate molecules. The preferred surfactant is nonylphenol containing an average of 9.5 moles of ethoxylate in the molecule.

(D) Dilution of concentrate.—The lubricants may be used as formulated or cut back by diluting with water. Generally, water dilutions ranging from about 1 part by weight up to about 5 parts by weight of water per part by weight of concentrate can be employed. However, inasmuch as the stability of the diluted concentrates decrease when the weight ratio of water to concentrate exceeds 1:1, about equi-weight ratios are preferred.

(E) Preferred compositions.—The components of the inventive lubricants are present in the following proportions:

(1) Water—from about 60 to 75 parts by weight.
(2) Thickener—from about 2.5 to about 5.5 percent hectorite clay, including optionally from 0.0 to 1.0 part by weight of sodium carboxymethylcellulose.

(3) Filler—from about 12 to 29 parts by weight of graphite, including optional calcium carbonate.

(4) Surfactant—from 0.0 to about 0.6 part by weight of a nonylphenol containing an average of 9.5 ethoxylate groups in the molecule.

(5) Borax—from about 0.75 to 2.0 parts by weight.

(6) Sodium metasilicate pentahydrate—from 1.5 to 6.5 parts by weight.

(F) Testing.—Three criteria are used to determine the suitability of a lubricant sample to perform under operational conditions. They are:

(1) Ability to wet oily metal.—A 1:10 ratio of a naphthenic oil in water emulsion is applied to a small piece of clean boiler plate and allowed to dry. A thin coating of the hot forging compound in diluted aqueous form is applied over the oily metal either by spraying or using a fiber paint roller. After standing for 5 minutes, the plates are rated visually for uniformity of coating. Ratings were either excellent, good, fair or poor.

(2) Brake on test.—A clean piece of boiler plate is coated on one side with a known amount of hot forging compound and allowed to dry. The plate is then placed in a furnace at 1600°–1800° F. for 6 minutes. After removal and cooling visual ratings as follows were used: (a) good—uniform residue, no peeling or cracking; (b) fair—cracking, no peeling; (c) poor—peeling and cracking of residue.

(3) Spray test.—A vertically suspended piece of boiler plate (10" x 10" x 1/8") is heated before three triangularly arranged Fisher burners. The hot forging lubricants are applied by spraying (300 p.s.i.g.) at the nozzle (0.019 inch diameter), and gave a 90° spray pattern from a nozzle distance of 8–10 inches by one sweep requiring 3–4 seconds. The cooled pieces of plate are examined for uniform coverage.

To illustrate the detailed workings of the invention, two preparations of hot forging lubricants are submitted. Unless otherwise noted, all parts or percentages are by weight and all temperatures are ° F. rather than ° C.

EXAMPLE 1

Preparation of an illustrative hot forging lubricating composition

In this preparation the procedure as disclosed is used to give a composition as given below.

Component:	Parts by weight
Water	64.1
Hectorite clay	2.9
Nonylphenol ethoxylated with an average of 9.5 ethoxylate groups	0.4
Sodium metasilicate · 5H ₂ O	4.0
Sodium carboxymethylcellulose	0.6
Borax (Na ₂ B ₄ O ₇ · 10H ₂ O)	1.0
Graphite	27.0
	100.0

The non-flammable composition is a black, thixotropic semi-solid lubricant, designed to be diluted with water up to a 1:1 dilution. The 1:1 cutback is fluid enough to be pumped and sprayed, shows good hot metal properties and is non-corrosive to steel and forms a stable, non-settling product.

EXAMPLE 2

Preparation of a slightly different hot forging lubricant

Again the procedure as disclosed is employed to produce the product whose composition by weight is disclosed below.

Component:	Parts by weight
Water	71.5
Hectorite clay	2.9
Sodium metasilicate · 5H ₂ O	2.0
Sodium carboxymethyl-cellulose	0.6
Borax (Na ₂ B ₄ O ₇ · 10H ₂ O)	1.0
Graphite	22.0
	100.0

The non-flammable lubricant when diluted 1:1 with water like its counterpart of Example 1, is a black, thixotropic semi-solid which upon its 1:1 dilution can readily be pumped, and sprayed, gives uniform coverage to the hot metal and is a stable and non-settling product.

When a greater concentration of bentonite clay (4.0 parts by weight per 100 parts by weight of the finished grease) is substituted for hectorite at the expense of the water component (70.4 parts) using the recommended blending process, a satisfactory hot forging lubricant is prepared.

EXAMPLE 3

Preparation of another hot forging lubricant

Once more the preparative procedure is employed to produce the product whose composition by weight is disclosed below.

Component:	Parts by weight
Water	74.6
Hectorite clay	4.0
Nonylphenol ethoxylated with an average of 12 ethoxylate groups	0.4
Sodium metasilicate · 5H ₂ O	3.0
Sodium carboxymethyl-cellulose	1.0
Borax (Na ₂ B ₄ O ₇ · 10H ₂ O)	2.0
Graphite	13.0
Calcium carbonate	2.0
	100.0

When the above lubricant concentrate is cut back 1:1 with water, a satisfactory hot forging lubricant having the desired properties is produced.

However, when the components of Example 3 are added in a slightly different sequence, an unsatisfactory lubricant (not sufficiently stiff) is produced.

For example, using the sample proportions and components above, if the addition of surfactant to the water is made before the clay is dispersed into the water system, the grease lacks the necessary stiffness even if the re-

maining components of sodium metasilicate, sodium carboxymethylcellulose, borax and calcium carbonate are previously prepared and added to the water dispersion of surfactant and clay.

As the preceding specification and examples have indicated, the hot forging lubricants of this invention have several advantages over the known prior art. For example, in its diluted form (1:1), it provides good uniform coverage of the hot metal and can be applied by either spraying, dipping or by other dispersing methods of the art. It fulfills its primary function of cooling the workpiece and die, as well as setting the hot metal parts. In addition, the lubricant is non-flammable and minimizes air pollution.

In addition to the above advantages, the novel invention is relatively flexible in that numerous substitutions, changes and modifications can be made without departing from the inventive concept.

The true metes and bounds of this invention can best be determined by a perusal of the following claims read in the light of this specification.

What is claimed is:

1. A non-flammable, water-based, thixotropic hot forging lubricant composition concentrate suitable for further dilution with water consisting essentially of the following admixture of components in the indicated proportions:

- (a) from about 60-75 parts by weight of water,
- (b) from about 2.5-5.5 parts by weight of clay thickener selected from the group consisting of bentonite and hectorite clays having a particle size of between 0.05 mm. and 0.01 mm. in diameter and an effective specific surface area in the order of 200 to 400 square meters per gram of clay,
- (c) from about 12-29 parts by weight graphite,
- (d) from about 0.75-2.0 parts by weight borax,
- (e) from about 1.5-6.5 parts by weight sodium metasilicate.

2. The lubricant of claim 1 wherein the composition includes up to 0.5 part by weight of surfactant selected from the group consisting of alkoxylated alkyl phenols containing an average of from 2 to 30 moles of alkylene oxide in the molecule, and where the alkylating group contains from 4 to 12 carbon atoms per molecule of phenol.

3. The lubricant of claim 2 wherein the surfactant is an ethoxylated nonylphenol.

4. The lubricant of claim 1 wherein up to 1.0 part by weight of sodium carboxymethylcellulose thickener is present in the hot forging lubricating composition.

5. A water-based, non-flammable hot forging lubricant concentrate consisting essentially of the following components in the indicated proportions:

Component:	Parts by weight
Water	64.1
Hectorite clay	2.9
Graphite	27.0
Nonylphenol ethoxylated with an average of 9.5 ethoxylate groups in the molecule	0.4
Sodium carboxymethylcellulose	0.6
Sodium metasilicate·5H ₂ O	4.0
Borax	1.0

6. A hot forging water-based, non-flammable lubricant concentrate consisting essentially of the following components in the indicated proportions:

Component:	Parts by weight
Water	71.5
Hectorite clay	2.9
Graphite	22.0
Sodium carboxymethylcellulose	0.6
Sodium metasilicate·5H ₂ O	2.0
Borax	1.0

7. A hot forging water-based, non-flammable lubricant concentrate consisting essentially of the following components in the indicated proportions:

Component:	Parts by weight
Water	75.0
Hectorite clay	4.0
Sodium metasilicate·5H ₂ O	3.0
Sodium carboxymethylcellulose	1.0
Borax	2.0
Graphite	13.0
Calcium carbonate	2.0

8. A process for preparing a homogenous non-flammable, water-based, thixotropic hot forging lubricant concentrate suitable for further dilution with water consisting essentially of the steps of admixing

- (a) from about one-quarter to two-thirds of the weight of water used in the finished water-based lubricant with from about 2.5 to 5.5 parts by weight of clay thickener selected from the group consisting of bentonite and hectorite clays having a particle size of between 0.05 mm. and 0.01 mm. and having an effective specific surface area in the order of 200 to 400 square meters per gram of clay, and from about 12 to 29 parts by weight of graphite until a homogenous admixture is formed, and

- (b) admixing a previously prepared homogenous admixture comprising from about 0.0 to 1.0 part by weight of sodium carboxymethylcellulose from about 1.5 to 6.5 parts by weight of sodium metasilicate pentahydrate and from about 0.75 to 2.0 parts by weight of borax with said clay-graphite-water mixture until a uniform dispersion is produced, and

- (c) admixing said aqueous dispersion with from 0.0 to 0.5 part by weight of an ethoxylated alkylphenol surfactant and the residual water to prepare the desired homogenous, non-flammable, water-based, hot forging lubricant.

9. The process of claim 8 wherein the clay thickener is hectorite, the ethoxylated alkylphenol surfactant is a nonylphenol containing an average of 9.5 ethoxylate groups per molecule of nonylphenol.

References Cited

UNITED STATES PATENTS

2,833,717	5/1958	Whitacre	252-33.4
2,735,814	2/1956	Hodson et al.	252-30
2,711,394	6/1955	Veatch et al.	252-30
2,588,625	3/1952	Ferner et al.	252-30
3,384,581	5/1968	Peace	252-30
2,258,202	10/1941	Burns	252-30
1,395,073	10/1921	Wiley	252-30
1,249,249	12/1917	Thorswik	252-30

FOREIGN PATENTS

583,905	9/1959	Canada	252-30
189,500	1/1966	U.S.S.R.	252-30

DANIEL E. WYMAN, Primary Examiner

I. VAUGHN, Assistant Examiner

U.S. Cl. X.R.

72-42; 252-30, 49.5