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(S) Lapping emuision compound and method.

G) A composition of matter suitable for use as a lapping composition is provided which has improved lubricity, abrasive suspension and cleanability and which contains an amide of the formula $\rm R_1CON~(R_2OH)~(R_3OH)$ in an amount of 4% to 20% by weight and an oil in an amount of 65% to 96% by weight. The lapping composition may have a wide range of amounts of water mixed into it to form a water-in-oil emulsion in which an abrasive agent may be suspended. Because of the wide ratios available between the water and oil phase amounts, the emulsion can be prepared relatively oil rich for fine lapping operation and may later be diluted for rough lapping operation.

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Lapping Emulsion Compound And Method

Technical Field

This invention relates generally to a composition of matter suitable for use in lapping and a method of lapping and more particularly to such a composition and method when used in conjunction with an abrasive material.

Background Art

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Lapping or polishing processes 10 . conventionally utilize an abrasive grain carried in a liquid vehicle to provide lubrication and abrasion between two rubbing surfaces. In some systems the abrasive is kept suspended by constant agitation. In other systems, the abrasive is kept suspended in 15 the liquid by utilizing an appropriate thickening agent. During the lapping operation the two rubbing surfaces become quite hot and are cooled by evaporation of the carrier liquid. In some instances, the supports for the surfaces are 20 conductively cooled as by a liquid coolant to further cool the surfaces. In the systems which utilize a suspending agent, as the liquid is evaporated the suspending agent becomes more concentrated and can present a real problem in being difficult to clean off of the surfaces following the 25 lapping operation.

Since the liquid evaporates as it is used to cool the surfaces being rubbed together, it is desirable that it be relatively low in cost. Water is particularly advantageous because of its low cost and very high heat of vaporization, whereby it provides especially effective cooling. However, water has very poor lubricating properties. Oil

alone can be used, but it has a relatively low heat of vaporization, thus requiring large quantities of oil for adequate cooling, and is more expensive than water. To provide good lubricating properties, it is desirable to combine an organic or other liquid having good bulk and polar lubricity with water. It is particularly desirable to form a water-in-oil emulsion so as to have oil-like lubricity and to adequately suspend the abrasive. This can be accomplished by use of an appropriate emulsifying agent. The same emulsifying agent can also provide suspending of the abrasive. A discussion of such an emulsion can be found in U.S. Patent 4,059,929, issued November 29, 1977 to David C. Bishop.

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15 Higher amounts of heat are generated in the rough lapping operation than are generated in the later fine lapping operation. As a result, the lapping compounds utilized for rough lapping should generally have a much higher percentage of water in 20 them or the compound should be supplied at a higher flow rate. When water-in-oil emulsified type systems are utilized, the oil to water ratio must usually fall in a relatively small range of concentrations or the emulsion will not be 25 sufficiently stable or maintain a useful viscosity. As a result, it is not generally possible to first utilize a water-in-oil emulsion system for fine lapping and then to later dilute that system with water, and add larger abrasive to it, for the rough lapping operation. If this is attempted, the 30 solution will generally deemulsify to its separate liquids or change to an oil-in-water emulsion with reduced abrasive-supporting capacity.

Further, many of the chemicals presently used to provide lapping systems are relatively expensive. That is, the emulsifiers are generally expensive and the organic liquid is generally expensive. Thus, a system wherein the organic liquid can be diluted after a fine lapping operation with addition of water and then reused for rough lapping would be highly desirable. Yet further, if a relatively inexpensive emulsifying agent could be utilized, this too would be desirable.

According to one aspect of the present invention we provide a composition of matter suitable for use in lapping comprising: at least one amide of formula

 R_1 CON (R_2 OH) (R_3 OH)

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(wherein R₁ represents a optionally substituted
15 alkyl, alkenyl, aryl, alkynyl, alkaryl, aralkyl, alkenaryl,
aralkenyl or alkynaryl radical having at least eight
carbon atoms; and

R₂ and R₃, which may be the same or different, each represent an optionally substituted alkylene, alkenylene or arylene diradical having less than eight carbon atoms) said amide being present in an amount which falls within a range from 4 to 20%, by weight, of said composition; and

an oil, in an amount which falls within a range from 65 to 96%, by weight, of said composition.

According to a further aspect of the present invention we provide a water-in-oil emulsion wherein the oil phase of the said emulsion comprises a composition of matter according to the present invention, the said oil phase constituting from 5 to 75% by volume and the water phase constituting from 25 to 95% by volume of the said emulsion.

According to a yet further aspect of the present invention we provide a method of lapping a surface by rubbing the surface against a further surface whilst maintaining a lubricating abrasive film between the said surfaces wherein the said film comprises a water-in-oil emulsion according to the invention and a suspended

abrasive agent.

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The above described composition of matter provides both better polar and bulk lubricity than water systems during lapping operations, thereby allowing 5 reduction of cycle times for lapping. The resulting lapped parts are easily cleanable. Very reliable suspension of abrasives is obtained in the water-in-oil emulsion. Chemical costs are relatively low. The lapping medium can be diluted with additional water and reused 10 in rough lapping operations since the amide provides the capability for maintaining the water-in-oil emulsion and suspension of abrasive over a wide range of ratios of water to oil. The surface temperatures during lapping operations of the pieces being lapped are kept lower than 15 when conventional oil only lapping systems are utilized.

In accordance with one aspect of the present invention, a composition of matter has been developed which is useful in lapping. An essential ingredient of the composition of matter is an amide respresentable by the formula $R_1 \text{CON}\left(R_2\text{OH}\right)\left(R_3\text{OH}\right)$. In this formula the radical R_1 may itself be substituted by groups, for example halide or hydroxy groups, which do not deleteriously effect the properties of the amide which are desirable in the present invention. R_1 will generally have at least 8 carbon atoms, more preferably at least about 12 carbon atoms, and generally no more than about 40 carbon atoms

R₂ and R₃ are each organic diradicals having less than 8 carbon atoms, and preferably no more than about 4 carbon atoms. The diradicals may be saturated, unsaturated or aromatic. The diradicals may also include substituent groups such as halides, hydroxides, or the like, so long as these groups do not interfere with the overall usefulness of the amide as a lapping composition constituent. For example, R₂ and R₃ may be substituted or unsubstituted alkylene, alkenylene or arylene diradicals.

The amide will generally form from about 4% to about 20%, by weight, of the overall composition. The amount of the amide is generally selected to provide the desired emulsifying and suspending properties in the water-in-oil emulsion which is utilized as a lapping composition.

Also part of the composition of matter is an oil in an amount which falls within a range from about 65% to about 96%, by weight, of the composition. Any of a number of suitable oils may be utilized, although it is preferred that a hydrocarbon oil, such as a petroleum oil, be utilized. It is somewhat more preferred that a petroleum oil having a relatively high naphthene content be utilized. Such oils are generally referred to as naphthenic oils. Naphthenic oils tend to provide particularly good bulk lubrication. A particularly preferred naphthenic oil is one having a viscosity of about 40 to 45 Saybolt Seconds Universal at 37°C.

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A triglyceride ester (oil or fat) is preferably present as a part of the composition,

suitably in an amount which falls in a range from about 2% to about 10%, by weight thereof. The triglyceride ester, a polar oil or fat, tends to provide improved polar lubricity, i.e., improved lubricity under high 5 temperature and high pressure conditions. Any of a number of triglyceride esters can be utilized, for example, any of the naturally occuring vegetable or nut oils such as coconut oil, corn oil, cottonseed oil, safflower oil, babassu oil, castor oil, cocoa 10 butter, linseed oil, mustard oil, neem oil, nigerseed oil, oiticica oil, olive oil, palm oil, palmkernel oil, peanut oil, perilla oil, poppy-seed oil, rapeseed oil, sesame oil, soybean oil, sunflowerseed oil, tung oil, wheat-germ oil, or the like. Naturally occuring triglyceride esters obtained from 15 animals, such as lard, oil, butterfat, depot fat, neatis-foot oil, beef tallow or mutton tallow, are also quite suitable. Sulfurized lard is a particularly preferred triglyceride oil because it 20 provides particularly desirable lubricity under high temperatures and extreme high pressure conditions.

While the amide discussed above is a surfactant, and provides desired surfactant and emulsifying properties as well as thickening properties, it is preferred that an additional surfactant be present, generally in an amount which falls in the range from about 1% to about 10%, by weight, of the composition. The additional surfactant has some use as a coemulsifying agent along with the amide, but its main use is to assure that the emulsifying agent, the oil, and the triglyceride ester, if present, can be easily washed off of pieces which are being lapped. Any of a

number of conventional surfactants can be utilized, although it is preferred that a nonionic surfactant be utilized. A particularly useful nonionic surfactant is an octylphenoxypolyethoxy ethanol having an hydrophylic lipophylic balance of about 10.4 and sold under the tradename "Triton X'45" by Rohm and Haas Company, Philadelphia, Pennsylvania.

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The amide can be formed by reacting an appropriate mono-hydroxylamine or preferably di-hydroxylamine, or mixture of such hydroxylamines, with an appropriate acid, generally a fatty acid. For example, diethanol amine has been reacted with oleic acid under reaction conditions which assure that the amide is formed in preference to the ester; that is, under conditions wherein the hydrogen which is attached to the nitrogen reacted with the acid group of the oleic acid, rather than with the hydrogen of one of the hydroxide groups. This has been accomplished by running the reaction at a temperature which is sufficiently high, generally a temperature of at least about 180°C. Water is produced by this reaction and the reaction must be run for a sufficient time to drive off at least most of the water and to allow the reaction to go sufficiently towards completion. It is not necessary to separate the amide from the reaction product obtained as just set out above. Instead, it can simply be combined with the hydrocarbon oil, and in appropriate cases also with the triglyceride ester and the surfactant. Alternatively, pure amide can be conventionally separated from the reaction mixture, or produced in any other manner, and later used to form a lapping oil concentrate.

A lapping oil concentrate includes the amide and the oil and generally also includes both the triglyceride ester and the additional surfactant. A particularly advantageous composition which has been prepared includes 7 to 9% of the amide formed by reaction of diethanol amine with oleic acid, 3 to 4% of Triton X-45, about 2.5% of sulfurized lard and the remainder of a naphthenic oil. These ingredients are thoroughly mixed to form a homogeneous solution.

When it is desired to prepare an emulsion for lapping purposes, a lapping oil concentrate as just described is mixed by slowly adding sufficient water to provide the desired emulsion. Vigorous mixing is normally necessary to create the emulsion.

A conventional abrasive agent, for example, aluminum oxide, silicon carbide, boron carbide, diamond, garnet, corundum, emery, silica, tripoli, or any of various metallic oxides or mixtures thereof, may be utilized. The grain particle size of the abrasive agent may vary but will generally fall in the range from about 0.25 micron to about 200 microns. The particle size chosen will depend upon the particular use of the lapping composition.

An emulsion can be prepared utilizing the lapping oil concentrate and a relatively small amount of water, for example 1 part of lapping oil concentrate to 1 part of water, and a relatively fine abrasive agent can be suspended therein. This composition can then be used for fine lapping operations. Thereafter, additional water and

additional (and coarser) abrasive can be added to prepare a composition which is useful for rough lapping operations. Further it is possible to have an oil phase which comprises the just discussed 5 lapping oil concentrate in an amount of 5% to 75%, by volume, and a water phase in an amount of 25% to 95% of the emulsion, by volume. Such a waterin-oil emulsion will remain stable over the above set out very wide range of water and oil con-10 centrations, due to the presence of the aforedescribed amide. When the oil concentration is relatively high and the water concentration is relatively low, the emulsion flows more or less like syrup. When the water concentration is relatively high and the 15 oil concentration is relatively low, the consistency of the emulsion is such that it sets up and resembles mayonnaise in consistency and appearance.

The preparation of the amide is preferably. carried out with non-stoichiometric proportions of 20 the amine and acid. In particular, the amount of the amine utilized is generally above that which would be required to stoichiometrically react with the acid. Generally, about a 30 to 60% excess of amine is utilized over that necessary for stoichiometric reaction with the acid. For example, 25 if the amine is diethanol amine and the acid is oleic acid, about 5 volumetric units of the amine would be reacted with about 9 volumetric units of the acid, whereas the stoichiometric mixture would 30 be 3 volumetric units of the amine to 9 volumetric units of the acid.

The amount of the abrasive agent suspended can vary over a wide range but will usually fall in a range from about 10% to about 50%, by weight, of the overall lapping composition, that is of the total weight of the water-in-oil emulsion plus the abrasive agent.

The amide above described generally the reaction product of a fatty acid having 12 to 20 carbon atoms with diethanolamine. While oleic acid is particularly preferred, this is really only one example 10 of a large number of acids which may be reacted with diethanol amine, or with a similar amine, to form the amide useful in the present invention. It would be voluminous if not impossible to list all of the acids which may be utilized to form amides useful with the 15 -composition of the present invention, but a brief and incomplete list would include: lauric acid, myristic acid, caproic acid, caprylic acid, decenoic acid, palmitic acid, stearic acid, arachidic acid, margaric acid, erucic acid, gadoleic acid, ricinoleic acid, 20 eleostearic acid, oleic acid, linoleic acid, palmitoleic acid and linolenic acid. Basically, the acids which are most useful are those which are naturally occurring as parts of triglyceride esters, such as the various naturally occuring oils and 25 It is preferred that the acid utilized be a liquid, at least at 180°C, so that reaction with diethanol amine can proceed most efficiently.

Industrial Applicability

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The composition, emulsion and method previously described are useful in lapping to provide smooth surfaces, generally on metallic

parts, but also on mineral specimens. In particular, this lapping composition is useful to provide smooth surfaces for seals.

A lapping composition as set out herein
provides good polar and bulk lubricity and resulting
relatively short cycle times for lapping, is easily
cleaned off of pieces which have been lapped, very
reliably suspends abrasive agents, provides
relatively low surface temperatures during lapping
operations and utilizes relatively low cost
chemicals.

Further, the emulsion can be, in essence, reused, since it can first be used for fine lapping, and then diluted with more water and through the addition of more and larger abrasive particles, for use in rough lapping operations. This latter property flows from the fact that the emulsion is stable and thick over wide ranges of relative oil and water contents.

CLAIMS

1. A composition of matter suitable for use in lapping comprising:

at least one amide of formula: $R_1 CON(R_2 OH) (R_2 OH)$ (wherein

R₁ represents an optionally substituted alkyl, alkenyl, aryl, alkynyl, alkaryl, aralkyl, alkenaryl, aralkenyl or alkynaryl radical having at least 8 carbon atoms; and

 R_2 and R_3 , which may be the same or different, each represent an optionally substituted alkylene, alkenylene or arylene diradical having less than 8 carbon atoms) said amide being present in an amount which falls within a range from 4% to 20%, by weight, of said composition; and

an oil, in an amount which falls within a range from 65% to 96%, by weight, of said composition.

- 2. A composition as claimed in claim 1, further
 including
- a triglyceride ester in an amount which falls within a range from 2% to 10%, by weight, of said composition.
- 3. A composition as claimed in either of claims 1 and 2 further including
- a surfactant in an amount which falls in a range from 1% to 10%, by weight, of said composition.
- 4. A composition as claimed in any one of claims 1 to 3 wherein in said amide substituent group R_1 represents a substantially straight chain alkyl or alkenyl group having at least 12 carbon atoms and substituent groups R_2 and R_3 , which may be the same or different, each represent an alkylene or alkenylene diradical having no more that 4 cabron atoms, and wherein said oil is a hydrocarbon oil.
- 5. A composition as claimed in any one of claims 1 to 4 wherein said amide is of formula

 $\text{CH}_3 \text{ (CH}_2)_7 \text{CH=CH (CH}_2)_7 \text{CON (CH}_2 \text{CH}_2 \text{OH)}_2$.

- 6. A composition as claimed in any one of claims 1 to 5 wherein said oil has a high naphthene content.
- 7. A composition as claimed in any one of claims 1 to 6 wherein said amide is the reaction product with diethanolamine of a fatty acid which occurs in nature as part of a triglyceride ester
- 8. A water-in-oil emulsion wherein the oil phase of the said emulsion comprises a composition of matter as claimed in any one of claims 1 to 7, the said oil phase constituting from 5 to 75% by volume and the water phase constituting from 25 to 95% by volume of the said emulsion.
- 9. An emulsion as claimed in claim 8, further including a suspended abrasive agent.
- 10. A method of lapping a surface by rubbing the surface against another surface by whilst maintaining a lubricating abrasive film between the said surfaces, wherein the said film comprises a water-in-oil emulsion as claimed in claim 8 and a suspended abrasive agent.