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EMULSION TYPE DRAWING COMPOUNDS CONTAINING CARBOXYALKYL CELLULOSE SALTS

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This invention relates to lubricants for metal working processes. More particularly it relates to lubricants of the emulsion type containing a solid filler and a water soluble salt of carboxyalkyl cellulose.

In metal working processes such as drawing, rolling, stamping and pressing, it is necessary to employ a lubricant to reduce the friction between the sliding metal surfaces to prevent sticking and scoring of the work and excessive wear on the die. In order to effectively separate the metal surfaces under the high pressures employed in such processes, it is usually necessary to employ lubricants containing inert fillers such as graphite, talc, bentonite, chalk, etc. These inert materials are usually applied to the surface of the work in the form of a suspension in a liquid carrier, such as water, a light mineral oil or an emulsion.

The emulsion-type lubricant containing an inert filler is the most generally satisfactory lubricant for cold metal forming processes, since by reason of its both oily and solid suspension ingredients it serves as a double-functioning lubricant, and in addition is easily removed after use by washing with water or with an alkali solution. For the sake of convenience and economy, lubricants of this type are ordinarily prepared by the manufacturer in the consistency of a soft grease concentrate having the solid filler dispersed therein and containing only a small amount of water sufficient to form a stable composition. This concentrate is then further diluted with water in the shop to the desired concentration of lubricant according to the specific requirements of the operation in which it is to be employed.

While a number of different compositions of the above type which have been employed heretofore in metal working operations have given satisfactory lubrication in relatively concentrated form, as for example in a dilution ratio of 1 to 1 or less, they are generally unsatisfactory at higher dilutions, chiefly because the emulsions thus produced have insufficient viscosity and adhesiveness to metal to form lubricating films of the necessary thickness upon the surface of the work. Another difficulty is that the emulsion breaks down either on standing or in use so that an oil film which cannot be removed by washing with water is deposited upon the surface of the work. Still another difficulty is that the particles of filler material tend to separate and sediment when the emulsion is permitted to stand in diluted form.

In accordance with my invention, an improved lubricating composition for the cold forming of metals comprises an aqueous emulsion of a mineral lubricating oil containing a solid filler and a small amount of a water soluble salt of a carboxyalkyl cellulose. My preferred composition is an aqueous emulsion of a mineral lubricating oil containing a fatty acid soap as an emulsifying agent and a solid filler comprising in major proportion calcium carbonate in finely divided form. The composition is preferably slightly alkaline with a small amount of free alkali.

Lubricating compounds of the above composition form smooth, adherent, thick films upon metal surfaces when employed in either concentrated or dilute form. They emulsify readily with water and form uniform and stable emulsions of good viscosity and other desirable lubricating characteristics over a wide range of dilution. Even in very dilute form, they provide good lubricating films having uniform distribution of both solid particles and oily component. Furthermore, their lubricating properties do not deteriorate upon standing by loss of emulsion or by settling out of the solid particles.

The water soluble salts of carboxyalkyl cellulose employed are chiefly alkali metal or alkaline earth metal salts of cellulose derivatives in which a portion of the hydroxy groups are etherified with carboxy-containing aliphatic groups. As examples of such compounds which may be mentioned are sodium carboxymethyl cellulose, ammonium carboxyethyl cellulose, calcium carboxypropyl cellulose and potassium carboxyhexyl cellulose. The preferred compound of this class is sodium carboxymethyl cellulose, which may be readily obtained in the known manner by dissolving cellulose fibers in sodium hydroxide solution to accomplish some degradation in the molecular chain length and to form sodium cellulose, which is then treated with monochloracetic acid. In order to obtain a water soluble product, the process is carried out in such a manner that at least about 10 per cent and preferably about 13 per cent of the hydroxy groups are etherified. A number of products of this character are available commercially, a particularly suitable product being the water soluble sodium carboxymethyl cellulose sold by the Drilling Specialties Co., Bartlesville, Oklahoma, under the trade name of "Driscose." This has been employed extensively as a drilling mud additive.

The carboxyalkyl cellulose salts may be present in extremely small amounts, such as 0.05 per cent or less, or in amounts above 1 per cent where the compositions are to be diluted with water in high dilution ratios such as 1:3 or higher. For most purposes they are employed in amounts between about 0.1 and 1 per cent, and preferably from about 0.2 to 0.5 per cent by weight based on the weight of the undiluted composition.

The emulsifying agent employed in these compositions is preferably an alkali metal fatty acid soap, obtained by saponifying a fat of animal, vegetable or synthetic origin with an alkali metal hydroxide or other compound. A particularly suitable soap is the sodium soap of wool fat. In addition to the fatty acid soap other emulsifying agents may be employed such as petroleum sulfonates.

The lubricating oil component may be any suitable medium viscosity fraction from a paraffinic, naphthenic or mixed base crude, obtained by simple distillation or by any of the usual refining methods such as solvent treating, acid treating, etc. It is preferably a distillate oil fraction from a naphthenic base crude having an SUS viscosity at 100° F. in the range from about 275 to about 325.

The solid filler is preferably calcium carbonate or calcium carbonate mixed with a minor amount of a different solid such as talc, oyster shell or clays of various types. The calcium carbonate may be obtained by grinding a natural material such as chalk to suitable particle size or it may be prepared chemically. A precipitated calcium carbonate is preferably used, having a particle size substantially below about 325 mesh but not more than about 40 per cent by weight of particles below 10 microns. A particularly suitable material of this character is sold commercially under the trade name of "Swansdown."

In addition to the components recited above, the composition may also contain minor amounts of other materials of the type commonly employed in lubricating compositions of this character such as antifoam agents, extreme pressure agents such as sulfur-containing compounds, materials or compounds to improve the appearance or odor of the composition, and so forth.

In concentrated form, the lubricating emulsions of this invention may comprise about 10 to 70 per cent by weight of solid filler comprising in major proportion calcium carbonate, about 5 to 20 per cent by weight of a fatty acid soap containing a small amount of free alkali, about 0.1 to 1 per cent or more by weight of a water soluble salt of carboxyalkyl cellulose, about 0.5 to 10 per cent of water and the remainder a mineral lubricating oil of medium viscosity.

Compositions of different filler concentrations may be made up for different uses. For example, a suitable composition for light drawing operations comprises about 15 to 20 per cent by weight of calcium carbonate, about 12 to 18 per cent by weight of a fatty acid soap obtained by saponifying wool grease with a small excess of alkali,

about 0.1 to 0.5 per cent by weight of sodium carboxymethyl cellulose, about 1 to 5 per cent of water and a naphthene base lubricating oil having an SUS viscosity at 100° F. of about 275 to 325. For more severe drawing operations the emulsion preferably contains about 32 to 38 per cent by weight of calcium carbonate, about 10 to 16 per cent by weight of the fatty acid soap and about 0.1 to 0.5 per cent by weight of sodium carboxymethyl cellulose. For deep drawing operations, the emulsion preferably contains about 52 to 58 per cent by weight of calcium carbonate, about 8 to 14 per cent by weight of the fatty acid soap and about 0.2 to 1 per cent by weight of sodium carboxymethyl cellulose.

The above compositions may be used directly in the form of a paste by spreading them over the surface of the metal to be pressed, rolled or otherwise shaped, or they may be diluted with water to form free-flowing emulsions. For example, they may be diluted with water in a dilution ratio of 1 to 1 or higher, such as in a dilution ratio 1 to 2 or even 1 to 3 in some cases.

The emulsion may be made up by conventional procedures. For example, a fat such as tallow, palm oil, degras or other suitable saponifiable material may be saponified with an alkaline solution such as sodium or potassium hydroxide, and the oil, filler, water and carboxyalkyl cellulose salt worked in gradually.

According to my preferred method for preparing these lubricating compounds, the saponification is carried out at 290–310° F. employing a small excess of alkali, amounting to from about 0.1 to about 0.5 per cent excess based on the weight of the finished product as calculated from the saponification number of the fat, and in the presence of a portion of the oil which forms the oil phase in the final product. Following the saponification, the mixture is maintained at this temperature until the soap is dehydrated and the remainder of the mineral oil added with stirring. The calcium carbonate is then added slowly with continued stirring at a temperature of 260–280° F. When the temperature has dropped below about 190–200° F., an odorant material, water and carboxyalkyl cellulose salt are added and the mix cooled to 105–110° F. with continued stirring until the compound is uniform and of a soft buttery consistency of about 300 penetration at 77° F. The product is drawn at this temperature and is not reheated in excess of 115° F.

The following example is given to illustrate the preparation of a typical compound of my invention and to demonstrate its superior properties as compared with a drawing compound of the usual type employed at the present time.

EXAMPLE

A charge comprising 950 pounds of degras having a saponification number of 120, 182 pounds of naphthene base lubricating oil having an SUS viscosity at 100° F. of 300, 400 pounds of water, and 182.5 pounds of a 49 per cent solution of caustic soda, corresponding to an excess of 0.1 per cent of alkali based on the weight of the finished product, was introduced into a steam heated kettle and heated with stirring for 2 to 3 hours at 290° F. to accomplish saponification and dehydration. The material was then maintained at this temperature for an additional 3 hours while 3704 pounds of the naphthene base lubricating oil was worked in slowly. The heating was then discontinued and 2700 pounds of "Swansdown" calcium carbonate added gradually with stirring. In order to improve the odor of the composition 25 pounds of pine tar was added also, when the temperature had dropped below 200° F. With continued stirring, 194 pounds of water was next added gradually during 1 hour while the temperature was maintained at 190° F., and finally 16 pounds of "Driscose" in admixture with 100 pounds of the calcium carbonate while the temperature was about 140° F. The stirring was continued for two hours after the "Driscose" was added, and the batch finally drawn at 105° F.

The product obtained by the procedure described above was a soft paste, light brown in color and of a smooth, non-grainy texture, having a penetration at 77° F. of 331. The test results given in the following table show the improved coverage and adhesion properties of this compound in comparison with those of an emulsion type lubricant of the usual composition. Lubricant No. 2 of the table is that described above. Lubricant No. 1 was prepared with the same lubricating oil base and with the same soap, but with a different type of filler and without the sodium carboxymethyl cellulose.

Table

Lubricant No.	1	2
Composition, percent by weight:		
Sodium soap of degras	11.60	12.96.
Lubricating oil	47.80	48.33.
Calcium carbonate		34.90.
Talc	38.90	
Pine tar	0.70	0.31.
Water	2.00	2.42.
Sodium carboxymethyl cellulose		0.20.
Tests:		
Emulsifiability	Good	Good.
Viscosity, 77° F., cps., 1:1 dilution—		
Freshly diluted	125	3,120.
After 24 hours		15,400.
Film weight, gm./sq. in., 1:1 dilution	0.04	0.45.
Sedimentation at 77° F., 1:2 dilution—		
After 24 hours	Visibile sedimentation.	None.
After 7 days		Do.
After 14 days		Do.

The viscosity values in centipoises given in the above table were obtained by means of the Brookfield Synchro Electric Viscometer upon samples which were prepared by mixing together 100 grams of the test material with 100 grams of distilled water at 77° F. until uniform.

The film weight values of the table were obtained in the following manner: A thin piece of polished steel plate, about 1.75 inches by 4 inches by 0.03 inch thick was weighed to the nearest 0.1 gram and immersed 2 inches into a beaker of the compound diluted one to one with distilled water and at a temperature of 77° F. After a few seconds it was removed, drained in a vertical position for one minute and reweighed. The weight of diluted compound retained on the plate was calculated from the increase in weight and the square inches of immersed surface.

The sedimentation test of the table was carried out by gradually diluting a 100 gram sample of the test compound with 200 grams of distilled water and permitting the diluted compound to stand in a stoppered graduate for 2 weeks at 77° F. At the end of the indicated periods the amount of sedimentation was observed.

The results given in the above table show the outstandingly superior adhesive and coverage properties of a typical compound of my invention over those of an emulsion type lubricant of conventional composition. These superior lubricating properties are strikingly demonstrated by the increase film weight obtained with my compound, amounting to over 10 times that obtained with the known compound. In addition, no sedimentation of filler occurred after 14 days with my compound in a 1:2 dilution with water, whereas in the case of the other compound sedimentation took place within 24 hours.

Obviously many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof, and therefore only such limitations should be imposed as are indicated in the appended claims.

I claim:

1. An improved lubricating composition for metal working which consists essentially of an aqueous emulsion of a naphthene base lubricating oil having an SUS viscosity at 100° F. of about 275 to 325, containing about 5 to 20 per cent by weight based on the weight of the composition of a soap obtained by saponifying wool grease with a small excess of alkali, about 10 to 70 per cent by weight based on the weight of the composition of finely divided calcium carbonate, about 0.1 to 1 per cent by weight based on the weight of the composition of sodium carboxymethyl cellulose, and about 0.5 to 10 per cent by weight based on the weight of the composition of water.

2. An improved lubricating composition for metal working which consists essentially of an aqueous emulsion of a naphthene base lubricating oil having an SUS viscosity at 100° F. of about 275 to 325, containing about 12 to 18 per cent by weight based on the weight of the composition of a soap obtained by saponifying wool grease with a small excess of alkali, about 15 to 20 per cent by weight based on the weight of the composition of calcium carbonate having a particle size below about 325 mesh, about 0.1 to 0.5 per cent by weight based on

the weight of the composition of sodium carboxymethyl cellulose, and about 1 to 5 per cent by weight based on the weight of the composition of water.

3. An improved lubricating composition for metal working which consists essentially of an aqueous emulsion of a naphthene base lubricating oil having an SUS viscosity at 100° F. of about 275 to 325, containing about 10 to 16 per cent by weight based on the weight of the composition of a soap obtained by saponifying wool grease with a small excess of alkali, about 32 to 38 per cent by weight based on the weight of the composition of calcium carbonate having a particle size below about 325 mesh, about 0.1 to 0.5 per cent by weight based on the weight of the composition of sodium carboxymethyl cellulose, and about 1 to 5 per cent by weight based on the weight of the composition of water.

4. An improved lubricating composition for metal working which consists essentially of an aqueous emulsion of a naphthene base lubricating oil having an SUS viscosity at 100° F. of about 275 to 325, containing about 8 to 14 per cent by weight based on the weight of the composition of a soap obtained by saponifying wool grease with a small excess of alkali, about 52 to 58 per cent by weight based on the weight of the composition of calcium carbonate having a particle size below about 325 mesh, about 0.2 to 1 per cent by weight based on the weight of the composition of sodium carboxymethyl cellulose, and about 1 to 5 per cent by weight based on the weight of the composition of water.

5. Claim 1 wherein the lubricating composition is diluted with water in a dilution ratio of from about 1:1 up to about 1:3.

6. An improved lubricating composition for cold metal working which consists essentially of an aqueous emulsion of a mineral lubricating oil, containing about 10 to 70 per cent by weight based on the weight of the composition of calcium carbonate in finely divided form, about 5 to 20 per cent by weight based on the weight of the composition of an alkali metal soap, about 0.1 to 1 per cent by weight based on the weight of the composition of a water soluble salt of carboxyalkyl cellulose wherein the alkyl group contains from 1 to 6 carbon atoms, and about 0.5 to 10 per cent by weight based on the weight of the composition of water, said composition being free from excess fatty acid and slightly alkaline with a small amount of free alkali metal hydroxide.

7. An improved lubricating composition for cold metal working which consists essentially of an aqueous emulsion of a mineral lubricating oil, containing about 10 to 70 per cent by weight based on the weight of the composition of calcium carbonate in finely divided form, about 5 to 20 per cent by weight based on the weight of the composition of an alkali metal soap, about 0.1 to 1 per cent by weight based on the weight of the composition of a water soluble salt of carboxymethyl cellulose, and about 0.5 to 10 per cent by weight based on the weight of the composition of water, said composition being free from excess fatty acid and containing about

0.05–0.5 per cent by weight of free alkali metal hydroxide.

8. A lubricating composition according to claim 7 in which the water soluble salt of carboxymethyl cellulose is sodium carboxymethyl cellulose.

9. A lubricating composition according to claim 7 in which the soap is an alkali metal soap obtained by saponifying wool grease.

10. A lubricating composition according to claim 7 in which the mineral lubricating oil is a naphthene base lubricating oil having an SUS viscosity at 100° F. of about 275 to 325.

11. An improved lubricating composition for metal working which consists essentially of an aqueous emulsion of a mineral lubricating oil, containing about 5 to 20 per cent by weight based on the weight of the composition of an alkali metal fatty acid soap, about 10 to 70 per cent by weight based on the weight of the composition of an inert finely divided filler, about 0.1 to 1 per cent by weight based on the weight of the composition of a water soluble salt of carboxyalkyl cellulose wherein the alkyl group contains from 1 to 6 carbon atoms, and about 0.5 to 10 per cent by weight based on the weight of the composition of water, said inert filler being selected from the group consisting of finely divided calcium carbonate, and a major proportion of finely divided calcium carbonate mixed with a minor amount of another inert filler selected from the group consisting of talc, oyster shell and clay.

12. Claim 11 wherein the lubricating composition is diluted with water in a dilution ratio of from about 1:1 up to about 1:3.

13. An improved lubricating composition for metal working which consists essentially of an aqueous emulsion of a mineral lubricating oil, containing about 5 to 20 per cent by weight based on the weight of the composition of an alkali metal fatty acid soap, about 10 to 70 per cent by weight based on the weight of the composition of finely divided calcium carbonate, about 0.1 to 1 per cent by weight based on the weight of the composition of a water soluble salt of carboxyalkyl cellulose wherein the alkyl group contains from 1 to 6 carbon atoms, and about 0.5 to 10 per cent by weight based on the weight of the composition of water.

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