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2,981,128

## PROCESS AND LUBRICANT COMPOSITION FOR ROLLING ALUMINUM

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No Drawing. Filed Mar. 7, 1957, Ser. No. 644,468

12 Claims. (Cl. 80—60)

This invention has to do with a lubricant useful for metal working operations. More specifically, the invention is directed to a soluble oil which serves as a lubricant and coolant in the rolling of non-ferrous metals, and particularly of aluminum.

This application is a continuation-in-part of my application Serial No. 578,601, filed April 17, 1956 and now abandoned.

The general method of rolling non-ferrous metals is well-known to those skilled in the art. Rolling involves passing a billet of the metal through the bit of two rolls a sufficient number of times to reduce the billet to a sheet of the desired thickness (i.e. gauge). The amount of reduction in each pass is dependent upon several factors including composition of the metal being rolled, the temperature of rolling, need for intermediate annealing and the like. During the rolling operation, however, it is necessary to cool and lubricate the working roll or rolls by means of a coolant-lubricant. The latter is usually applied to the working roll as a plurality of streams of liquid in sufficient total quantity to keep the working roll from softening, to keep the friction between the working roll and the metal being rolled to a minimum, and to prevent metallic "pick-up" on the rolls.

Typical rolling mills and the operation thereof are shown and described in "Metalworking Lubricants," E. L. H. Bastian, 1951, McGraw-Hill, New York.

Numerous lubricants have been proposed for use in metal working operations, but such lubricants have generally fallen short of the performance necessitated by current industrial practices. This is particularly so with regard to present day techniques for cold-rolling of non-ferrous metals such as aluminum. As the demand for aluminum has grown, industry has been forced to either speed up rolling mills now in operation or to invest tremendous sums of money for additional mills. The primary limitation upon the speed of present rolling mills appears to be the lubricant, since the latter must also serve as a coolant. It is obvious that considerable heat is generated when an aluminum strip about 1/4 inch thick is cold rolled to a thickness of a fraction of an inch. Because of the limitations of many prior lubricants, the speed of rolling mills has been relatively low. When the speed has been increased in an effort to increase production, excessive heat has developed and it has been necessary to stop the rolls and cease operations until the rolls have become cool enough for further use.

Soluble oil emulsions, developed in recent years, have proven fairly satisfactory in dissipating heat and in making possible higher rolling speeds in cold rolling operations. However, such materials have unsatisfactory features which limit their usefulness. They do not relieve

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the so-called "pick-up" problem. This phenomenon is the appearance of nodules or accretions, primarily consisting of aluminum fines, on the surface of the rolls. The accretions build up and form minute hills which, in turn, produce peak unit loads on the new portions of the metal being rolled and the deformation multiplies to such an extent that the mill must be shut-down and the rolls must be reground. In addition to considerable loss of time and its monetary value, there is an appreciable spoilage of semifinished metal.

Another shortcoming of soluble oil emulsions is their relatively low capacity to prevent metallic wear, which is caused by friction between the rolls and the metal being rolled. Thus, there has been a need for a new soluble oil possessing a high order of wear-reducing capacity.

Soluble oil emulsions have also been characterized, in general, by staining of metals such as aluminum. Stains found on roll aluminum have been of two types. One is a water stain, traceable to water in the emulsions, and the other an organic stain, traceable to the various organic components of the emulsions. These stains are such as to render the rolled aluminum unsaleable.

It is an object of this invention, therefore, to provide a soluble oil, and water-in-oil emulsions thereof, having excellent coolant properties, minimizing "pick-up" and stains, and possessing excellent wear-reducing capacity. A further object is to provide stable water-in-oil emulsions. Another object is to provide new emulsifiable oils.

Another object is to provide emulsions stable at elevated temperatures. Still another object is to provide emulsions which tolerate "tramp oil."

A further object is to provide soluble oils suitable for use with hard water such that stable emulsions are formed therewith and such that water hardness does not cause metal staining.

Still a further object is to provide a soluble oil and water-in-oil emulsions thereof which permit consecutive passes to be taken on the same metal strip without overheating of the strip or necessitating cooling between passes.

A related object is the provision of a soluble and water-in-oil emulsions thereof which make possible lower bearing temperatures on rolls.

Other objects and advantages of the present invention will become apparent to those skilled in the art, from the following detailed description.

It has now been discovered that all of the aforesaid objects are realized by development of a new soluble oil comprising in certain proportions the following components:

|                                                                                                                                 | Percent by weight |
|---------------------------------------------------------------------------------------------------------------------------------|-------------------|
| (a) A mixture of alkyl esters of mixed, high molecular weight acids obtained by controlled oxidation of petroleum fractions     | 1 to 20           |
| (b) A fatty acid ester of a hexitol anhydride, said fatty acid having at least six carbon atoms                                 | 1-10              |
| (c) A polyoxyalkylene derivative of a fatty acid ester of a hexitol anhydride, said fatty acid having at least six carbon atoms | 1-10              |
| (d) An aluminum soap                                                                                                            | 0.5-5             |
| (e) A mineral oil having a viscosity of 1.4 centistokes to 65 centistokes at 100° F.                                            | 96.5              |

A particularly outstanding soluble oil of this invention is one having the following composition:

|                                                                                                                                                 | Percent<br>by weight |
|-------------------------------------------------------------------------------------------------------------------------------------------------|----------------------|
| (a) A mixture of methyl esters of mixed, high molecular weight acids obtained by controlled oxidation of petroleum fractions <sup>1</sup> ----- | 7.5                  |
| (b) Sorbitan monooleate -----                                                                                                                   | 2.0                  |
| (c) Polyoxyethylene sorbitan monooleate -----                                                                                                   | 2.0                  |
| (d) "Aluminum stearate" <sup>2</sup> -----                                                                                                      | 0.8                  |
| (e) Mineral oil having a viscosity of 5 centistokes at 100° F. -----                                                                            | 87.7                 |

<sup>1</sup> This material has the following chemical and physical properties:

|                                         |             |
|-----------------------------------------|-------------|
| Acid number -----                       | 5-20        |
| Saponification number -----             | 80-120      |
| Specific gravity at 70° C. -----        | 0.886-0.893 |
| Weight per gallon, lbs. -----           | 7.3-7.5     |
| Viscosity (centistokes) at 99° C. ----- | 12-16       |
| Melting point, ° C.—ASTM -----          | 33-43       |
| Flash, ° C. (O.C.) -----                | 149-171     |
| Fire, ° C. (O.C.) -----                 | 182-205     |
| Refractive index at 70° C. -----        | 1.438-1.440 |

<sup>2</sup> Aluminum di-soap of C<sub>12</sub>-C<sub>22</sub> acids, said acids being hydrogenated fish oil fatty acids; soap is sold as "Aluminum stearate."

The soluble oils of this invention can be diluted with water to the desired viscosity of the emulsion wanted. Generally, the emulsion will contain up to about 70 (seventy) percent by weight of water. Preferably, however, the emulsion will contain from about 25 to about 60 percent by weight of water. Expressing this in tabular form, as the soluble oil composition is expressed above:

|                                                                                                                                       | Percent<br>by weight |
|---------------------------------------------------------------------------------------------------------------------------------------|----------------------|
| (a) A mixture of alkyl esters of mixed, high molecular weight acids obtained by controlled oxidation of petroleum fractions -----     | 3.0-6.0              |
| (b) A fatty acid ester of a hexitol anhydride said fatty acid having at least six carbon atoms -----                                  | 0.8-1.6              |
| (c) A polyoxyalkylene derivative of a fatty acid ester of a hexitol anhydride, said fatty acid having at least six carbon atoms ----- | 0.8-1.6              |
| (d) An aluminum soap -----                                                                                                            | 0.3-0.6              |
| (d) A mineral oil having a viscosity of 1.4 centistokes to 65 centistokes at 100° F. -----                                            | 35-60                |
| (f) Water -----                                                                                                                       | 25-60                |

An emulsion of excellent character made from the aforementioned, outstanding base soluble oil is:

|                                                                      | Percent<br>by weight |
|----------------------------------------------------------------------|----------------------|
| (a) A mixture of methyl esters identified above in note (1) -----    | 3.0                  |
| (b) Sorbitan monooleate -----                                        | 0.8                  |
| (c) Polyoxyethylene sorbitan monooleate -----                        | 0.8                  |
| (d) Aluminum stearate, note (2) above -----                          | 0.3                  |
| (e) Mineral oil having a viscosity of 5 centistokes at 100° F. ----- | 35.1                 |
| (f) Water -----                                                      | 60.0                 |

As indicated above, one component of the compositions contemplated herein is a mixture of alkyl esters of mixed, high molecular weight acids obtained by controlled oxidation of petroleum fractions. Particularly effective, in combination with the other components described herein, is the mixture of methyl esters described above in note (1). While not equivalent to the latter, other such mixed esters can be used herein. Typical of such mixed esters are the following Alox products, numbered 100, 152, 301 and 475. Such mixtures are also referred to as petroleum oxidates.

A second component of the new compositions is a fatty acid ester of a hexitol anhydride wherein the fatty acid contains at least six carbon atoms. Esters of this character are well known in the art, being shown, for example, in U.S. Patent No. 2,322,822. Illustrative of such esters are sorbitan monolaurate, sorbitan mono-

palmitate, sorbitan monostearate, sorbitan tristearate, sorbitan monooleate, sorbitan trioleate, mannitan monolaurate and the like. Preferred herein, however, because of its excellent cooperative action with the other components of the new compositions, is sorbitan monooleate.

A third component, one closely related to the above-described second component, is a polyoxyethylene derivative of a fatty acid ester of a hexitol anhydride wherein the fatty acid has at least six carbon atoms. These derivatives are also well known in the art. They can be produced by reacting an olefin epoxide such as ethylene oxide with an ester of the character described above as the second component. Representative polyoxyalkylene derivatives which can be used herein are polyoxyethylene sorbitans such as the monolaurate, monopalmitate, monostearate, tristearate and trioleate. Particularly effective in combination with the other components of this invention, and therefore a preferred material, is polyoxyethylene sorbitan monooleate.

The fourth component of the new composition is an aluminum soap. While a great number of such compounds can be used within the scope of this invention, especially effective are aluminum stearates and particularly aluminum di stearate of the character identified in note (2) above.

The mineral oils or hydrocarbon vehicles suitable herein are those having viscosities ranging from 1.4 centistokes to 65 centistokes at 100° F. It is to be noted that this range is inclusive of kerosene fractions as well as lubricating oils. Preferred are oils of paraffinic character. Outstanding are oils having viscosities of about 5 centistokes (42 seconds, Saybolt) at 100° F.

The soluble oils and emulsions thereof coming within the scope of this invention, can also contain minor quantities of other ingredients which do not lessen the effectiveness of the components described above. For example, a bactericide or fungicide can be added in accordance with local mill practices, particularly in areas such as the southern United States. Typical of such materials are: phenols such as orthophenylphenol and sodium mercapto benzothiazol. These are usually used in concentrations of about 0.1% to 1.0% in soluble oils, and about 0.01% to 1.0% in emulsions.

The soluble oils of the invention can be prepared by adding the various components to each other in any order, and preferably at temperatures of the order of 180 to 210° F. The emulsions can be prepared by adding water slowly to the soluble oil in any suitable homogenizing equipment, such as a Manton-Gaulin Homogenizer, a Stratco Contactor, or in a mechanically agitated blending kettle, while agitating the soluble oil.

The soluble oils of this invention have been subjected to a severe wear test, namely, the Avery extreme pressure test. Test conditions and the equipment used therein are described by H. W. Browdson in Institute of Metals Journal (1930). The maximum value for this test equipment is a load of 70 pounds. This test simulates actual operating conditions, measuring load carrying capacity without "pick-up" of aluminum particles from the aluminum sheet or roll. The tests were run with aluminum test plates; an aluminum extensively rolled in the industry.

Given below in Table I are the results in the Avery test of soluble oil emulsions, including the aforementioned excellent soluble oil emulsion of this invention.

Table I

| Soluble oil emulsion:           | Avery value, pounds |
|---------------------------------|---------------------|
| Conventional oil-in-water ----- | 35                  |
| New soluble oil -----           | 70+                 |

It is obvious from the results presented in Table I that the new compositions have exceptional resistance to metallic "pick-up."

In addition to the Avery test results shown above, water and organic stain tests were made on the compositions identified in Table I. These tests were conducted in the following manner. The water stain test involved pouring ten cubic centimeters of emulsion into a coil of aluminum metal and holding the latter in an oven at 225° F. for 2 hours. Organic stain was determined by spreading 0.1 cubic centimeter of emulsion on an aluminum plate and maintaining the latter in a muffle at 650° F. for one-half hour. Results of the stain tests are provided in Table II following.

Table II

| Soluble Oil Emulsion           | Water Stain at 225° F. | Organic Stain at 650° F. |
|--------------------------------|------------------------|--------------------------|
| Conventional oil-in-water..... | Excessive.....         | Excessive.               |
| New soluble oil.....           | Trace to none.....     | Slight.                  |

Further emulsion stability tests were determined at various temperatures. These tests were made by preparing the new soluble oil emulsion, identified in Tables I and II above, and allowing a small bottle containing the same to stand at a constant temperature until a separation of oil or water was noted. Results of such tests are given in Table III below:

Table III

| Temperature, ° F. | Number of Days | Observations            |
|-------------------|----------------|-------------------------|
| -10 (frozen)..... | 2              | No separation.          |
| 70.....           | 10             | Very slight separation. |
| 70.....           | 30             | Slight oil.             |
| 140.....          | 10             | Do.                     |
| 220.....          | 1/24           | No separation.          |

It should be noted that there was no separation of water in any of the tests shown in Table III.

Viscosities of typical water emulsions of the new soluble oil identified in Tables I and II above, have been determined. At the same time, the stability of the emulsions have been observed. Such data is set out below in Table IV:

Table IV

| Emulsion, Percent by Weight of H <sub>2</sub> O | Viscosity S.S.U. at 100° F. | Stability     |
|-------------------------------------------------|-----------------------------|---------------|
| 0.....                                          | 54                          | O.K.          |
| 20.....                                         | 70                          | Satisfactory. |
| 40.....                                         | 110                         | Good.         |
| 60.....                                         | 300                         | Maximum.      |

The new soluble oils are also advantageous in that they have excellent tolerance to hard waters. For example, emulsions of excellent character are made with water containing as much as 1000 parts per million of salts.

Following laboratory evaluation of the compositions of this invention, an evaluation was made in an aluminum mill in a cold-rolling operation. With a 25 percent water emulsion of the new soluble oil shown in Tables I and II, applied to the rolls and the aluminum strip, the mill was operated continuously for a period of 5 days with no necessity for shut-down, since the rolls were cooled satisfactorily by the emulsion and the rolls and aluminum were lubricated satisfactorily. The mill was operated at twice the speed used with prior art lubricants. Furthermore, only a very slight water staining resulted with the new oil; this was substantially less than with the prior art oils. Also, the new emulsion permitted heavier reduction of aluminum than prior lubricants. For example, a substantial reduction in thickness of an aluminum strip was effected with at least one less pass of the strip through the mill as compared with the performance of prior art lubricants. Thus, the compositions of this invention have

made possible substantially greater rolling production with existing equipment by virtue of greater roll speeds, greater reductions, and multiple passes without necessity for cooling of rolls between passes.

In another evaluation on an industrial basis, 40 to 50 percent water emulsions of the following novel soluble oil were used in an aluminum mill in a hot-rolling operation. The emulsions were sprayed on the work rolls and back-up rolls of the mill as an aluminum billet passed through the rolls. The strips varied in width ranging up to about 12 feet. Satisfactory cooling of the rolls and aluminum was effected, and satisfactory lubrication of the rolls and aluminum was obtained, thus making possible continuous operation of the mill. The emulsions provide outstanding protection against metallic pick-up, even when they contain as much as about 50 percent of water. Corresponding results can be obtained when the soluble oil itself is used without water. The soluble oil used in the hot-rolling operation differed from soluble oil used in the cold-rolling operation, in that it contained 7.6 percent of mixed esters instead of 7.5 percent and 87.6 percent of oil instead of 87.7 percent.

Although the foregoing details have been directed to the use of the new compositions in rolling aluminum, they can also be used advantageously in otherwise changing the shape or size of aluminum. For example, they can also be used in broaching, cutting, drawing, extruding, forging, machining and pressing, and sawing. Furthermore, they can be used similarly with other non-ferrous metals such as copper, brass, and magnesium.

I claim:

1. A soluble oil, non-staining to aluminum, consisting essentially of: (a) from about 1 to about 20 percent by weight of a mixture of alkyl esters of mixed, high molecular weight acids obtained by controlled oxidation of liquid petroleum fractions; (b) from about 1 to about 10 percent by weight of a fatty acid ester of a hexitol anhydride, said fatty acid having at least six carbon atoms and said fatty acid ester ranging from a mono to a triester; (c) from about 1 to about 10 percent by weight of a polyoxyalkylene derivative of a fatty acid ester of a hexitol anhydride, said fatty acid having at least six carbon atoms and said fatty acid ester ranging from a mono to a triester; (d) from about 0.5 to about 5 percent by weight of an aluminum stearate; and (e) from about 96.5 to about 55 percent by weight of a hydrocarbon fraction having a viscosity of 1.4 centistokes to 65 centistokes at 100° F.

2. A soluble oil defined by claim 1 wherein (b) is sorbitan monooleate.

3. A soluble oil defined by claim 1 wherein (c) is polyoxyethylene sorbitan monooleate.

4. A soluble oil defined by claim 1 wherein (d) is aluminum distearate.

5. A soluble oil, non-staining to aluminum, consisting essentially of: (a) about 7.5 percent by weight of a mixture of methyl esters of mixed, high molecular weight acids obtained by controlled oxidation of liquid petroleum fractions; (b) about 2 percent by weight of sorbitan monooleate; (c) about 2 percent by weight of polyoxyethylene sorbitan monooleate; (d) about 0.8 percent by weight of aluminum distearate; and about 87.7 percent by weight of a mineral oil having a viscosity of about 5 centistokes at 100° F.

6. A water-in-oil emulsion, non-staining to aluminum, consisting essentially of: (a) from about 3 to about 6 percent by weight of a mixture of alkyl esters of mixed, high molecular weight acids obtained by controlled oxidation of liquid petroleum fractions; (b) from about 0.8 to about 1.6 percent by weight of a fatty acid ester of a hexitol anhydride, said fatty acid having at least six carbon atoms and said fatty acid ester ranging from a mono to a triester; (c) from about 0.8 to about 1.6 percent by weight of a polyoxyalkylene derivative of a

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fatty acid ester of a hexitol anhydride, said fatty acid having at least six carbon atoms and said fatty acid ester ranging from a mono to a triester; (d) from about 0.3 to about 0.6 percent by weight of an aluminum stearate; (e) from about 35 to about 60 percent by weight of a mineral oil having a viscosity of 1.4 centistokes to 65 centistokes at 100° F.; and (f) from about 25 to about 60 percent by weight of water.

7. An emulsion defined by claim 6 wherein (b) is sorbitan monooleate.

8. An emulsion defined by claim 6 wherein (c) is polyoxyethylene sorbitan monooleate.

9. An emulsion defined by claim 6 wherein (d) is aluminum distearate.

10. A water-in-oil emulsion, non-staining to aluminum consisting essentially of: (a) about 3 percent by weight of a mixture of methyl esters of mixed, high molecular weight acids obtained by controlled oxidation of liquid petroleum fractions; (b) about 0.8 percent by weight of sorbitan monooleate; (c) about 0.8 percent by weight of polyoxyethylene sorbitan monooleate; (d) about 0.3 percent by weight of aluminum distearate; (e) about 35.1 percent by weight of a mineral oil having a viscosity of about 5 centistokes at 100° F.; and (f) about 60 percent by weight of water.

11. In the rolling of aluminum wherein a strip of

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the metal is passed through the bit of a plurality of rolls to reduce the strip to a metal sheet of desired lesser thickness, and wherein a coolant-lubricant is applied to the working roll, the improvement which comprises: applying to the working roll an emulsion defined by claim 6.

12. In the rolling defined by claim 11, the improvement which comprises: applying to the working roll an emulsion defined by claim 10.

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