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LUBRICATING COMPOSITION

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This application is a continuation-in-part of co-pending application Ser. No. 387,984, filed Aug. 6, 1964 and now abandoned.

This invention relates to an improved lubricating composition and more particularly relates to an improved soap-type lubricant which is useful in lubricating chemically coated metal surfaces prior to deformation.

In the art of the cold forming of metals, e.g., metal deformation or metal drawing, it is customary to provide a chemical coating on the metal surface to be deformed, such as a phosphate coating, an oxide coating, an oxalate coating, a sulfide coating, or the like. Additionally, prior to the actual deformation of the metal surface, the chemical coating on the metal surface is frequently treated with a lubricant, such as a hot aqueous soap solution. In this manner, there is a reaction between the hot aqueous soap solution and the previously applied chemical coating, such as a phosphate coating, to form a metallic soap which is integral with the phosphate coating and thus with the underlying metal surface, which integral coating is substantially insoluble in water. The thus-formed coating is grayish to white in color, smooth and velvety to the touch and crushes to a continuous, unctuous film on the application of pressure to the metal surface. This continuous film has been found to be an excellent parting layer between the metal and the die during deforming operations.

Once the deforming operation has been completed, the resulting metal article is generally subjected to an annealing or finishing operation. Typical finishing operations include plating, the application of corrosion resistant or decorative coatings, buffing, polishing, and the like. For the most part, the success of these subsequent finishing operations depends to a great extent on having the surface of the formed metal article substantially clean. After the deforming operation appreciable quantities of the lubricant composition ordinarily remain on the metal surface. Frequently, this remaining lubricant material is in the form of such a tenacious film or layer that its removal from the metal surface prior to the annealing or finishing operation is effected only with great difficulty. It is, therefore, desirable that the nature of the lubricant be modified, if possible, so that the residual film or layer of lubricant can be more easily removed from the metal surface.

Additionally, in applying many soap-type lubricants to a chemical coating, and in particular those soap lubricant compositions containing appreciable quantities of inorganic pigment materials, such as borax, it is found that not only must the soap lubricant composition be maintained at a relatively high temperature, e.g., above about 180 degrees Fahrenheit, in order to maintain it in a sufficiently fluid condition for application to the metal surfaces, but frequently where roller coating applications are used, the rollers themselves must be heated to prevent solidification of the lubricant composition on the roll during application. Accordingly, it is desirable if the composition of the soap lubricant is such that it has sufficient fluidity at the lower temperatures, e.g., 150 degrees Fahrenheit or lower, that it can be applied without heated rollers.

It is, therefore, an object of the present invention to

provide a novel soap-type lubricant composition which is useful in metal deforming operations.

Another object of the present invention is to provide a novel soap-type lubricant composition as described above, which composition is more readily removed from the metal surface to which it has been applied after the surface has been subjected to a deforming operation.

A further object of the present invention is to provide an improved process for the deforming of metal surfaces, which process utilizes the improved soap-type lubricant composition of the present invention.

These and other objects will become apparent to those skilled in the art from the description of the invention which follows.

Pursuant to the above objects, the present invention includes a lubricant composition concentrate of the soap type, which composition comprises, in combination, an alkali metal pyrophosphate and the soap of a fatty acid. Such lubricant compositions have been founnd to be especially useful in metal deforming operations when applied over a chemical coating on the metal surfaces to be deformed, such as a phosphate coating or the like.

More specifically, the improved lubricant concentrate compositions of the present invention are comprised of a fatty acid soap in an amount within the range of about 15 to about 97 percent by weight of the composition and an alkali metal pyrophosphate in an amount within the range of about 3 to about 75 percent by weight of the composition. By the term "alkali metal pyrophosphate," as used in the specification and claims, it is intended to include the pyrophosphates of lithium, sodium, potassium, cesium, and rubidium. Of these, excellent results have been obtained when using tetrapotassium pyrophosphate (TKPP) and primary reference will be made hereinafter to this material. This is not, however, to be taken as a limitation of the present invention, but only as being exemplary thereof. Additionally, it is to be appreciated that in referring to the present composition containing a soap, it is intended to include both those compositions which contain the soap, per se, as well as those compositions which contain ingredients which react to form the soap in situ in the composition, such as those compositions containing a fatty acid, or a fat, or an oil and an alkali, such as an alkali metal hydroxide or an alkali metal carbonate. Typical of the fatty acid soaps used are those containing about 8 to about 22 carbon atoms, with those containing about 12 to about 18 carbon atoms being preferred.

In addition to the tetrapotassium pyrophosphate and the soap, the compositions of the present invention may also contain various other adjuvants depending upon the particular characteristics of the lubricant compositions which are desired in each instance. For example, various so-called pigment materials, both meltable and unmeltable may be included in the lubricant composition. Additionally, organic resinous or polymeric binding materials may also be included in the composition, as well as rosin acid and/or various rosin soaps. Additionally, additives such as corrosion inhibitors, dyes, perfumes, and the like, may also be included in the lubricant composition as desired.

Exemplary of pigment materials which may be included in the lubricant composition of the present invention are antimony oxide, antimony sulfide, arsenous oxide, arsenous sulfide, barium pyrophosphate, bismuth sulfide, boric anhydride, calcium tetraborate, calcium carbonate, cadmium pyrophosphate, cobalt sulfide, chromium fluoride, copper sulfide, ferrous sulfide, ferrous phosphate, lead borate, lead chromate, lead molybdate, lead oxide, lead phosphate, lead metasilicate, lead sulfide, manganese pyrophosphate, manganese borate, mercury sulfide, mercury chloride, molybdic oxide, nickel sulfide, molybdenum

sulfide, sodium tetraborate (borax), vanadium pentoxide, zinc borate, zinc phosphate, clays, including diatomaceous earth, Fuller's earth, and the like. These and similar pigment materials may be added to the composition in addition to the alkali metal pyrophosphate or as a replacement for a portion of the alkali metal pyrophosphate in the composition. Where pigments are included in the lubricating composition, amounts within the range of about 0.1 percent to about 80 percent are typical, with amounts within the range of about 3 percent to about 75 percent being preferred.

Exemplary of the binders which may be used are acrylic ester polymers, e.g., polymethacrylates, alkyd resins such as the glycerol-phthalic anhydride type, cellulose acetate, cellulose nitrate, coumarone-indene polymers, styrene polymers, chlorinated diphenyl, chlorinated rubber, ethylene polysulfide polymers, chlorinated paraffins, neoprene rubber, vinyl chloride, vinyl acetate, melamine polymers, urea-aldehyde polymers, casein-formaldehyde polymers, phenol-formaldehyde polymers, starch, sodium naphthenate, amber, asphalt, bitumen, gilsonite, dextrin, gelatin, gum arabic, and the like. Where such binders are used in the present lubricating composition, they are typically present in amounts within the range of about 0.1 percent to about 25 percent, with amounts within the range of about 1.0 percent to about 15 percent being preferred.

The rosin acids which are suitable for inclusion in the present lubricating compositions may be in the form of commercial gum or wood rosin, tall oil, or may be extracted from tall oil residues. Accordingly, as used in the specification and claims, the term "rosin acid" is intended in its generally understood sense and may also include such specific acids as abietic acid, neo abietic acid, pimaric acid, levo-pimaric acid, dextro pimaric acid, isodextro pimaric acid, mixtures thereof, and the like, and the rosin soaps are soaps made from these acids. Where rosin soaps and/or rosin acids are included in the lubricating composition, they are typically present in amounts within the range of about 0.1 percent to about 30.0 percent, and preferably present in amounts within the range of about 0.5 percent to about 10.0 percent.

Corrosion inhibiting materials which may be included in the subject lubricating composition are exemplified by the alkali metal nitrates, the alkali metal nitrites, and the like. These and other inhibitors as are known to those in the art, when used, are typically present in amounts within the range of about 0.1 percent to about 5.0 percent and preferably in amounts within the range of about 0.1 percent to about 3.0 percent. Dyes, such as bismarck brown, and the like, and perfumes or other materials for imparting a pleasant odor to the composition, such as pine oil and the like, may also be incorporated in the lubricant composition in amounts sufficient to impart the desired color and odor to the composition.

In addition to the above adjuvants, the tetrapotassium pyrophosphate-soap lubricant concentrate composition of the present invention may also contain water, the amount depending upon the physical form which is desired for the composition. Typically, water in amounts up to about 80 percent by weight of the total composition may be used, with amounts within the range of about 15 to about 75 percent by weight of the total composition being preferred. As will be appreciated by those in the art, in determining the physical form or consistency of the lubricant concentrate composition which is desired, consideration will, of course, be given to the manner in which the composition is to be handled, packaged and transported. Accordingly, the amount of water included in the lubricant concentrate composition may vary widely and in some instances may even exceed 80 percent by weight, which has been indicated hereinabove as being typical.

Even when water in an amount of 75 percent by weight of the total lubricant composition is used, the composition is still sufficiently concentrated as to be undesirable for many applications to metallic surfaces. Accordingly, in formulating a working composition for application to a metal surface, the concentrated lubricant composition will generally be further diluted with water. Typical working compositions may contain the lubricant concentrate in amounts within the range of about 10 to about 400 pounds per hundred gallons of solution, and preferably in an amount within the range of about 50 to about 200 pounds per hundred gallons of solution. The aqueous working lubricant compositions containing the lubricant concentrate in amounts within the range of about 10 to about 400 pounds per 100 gallons of solution will have a Babcock number within the range of about 0.4 to about 18.5 and a titration number within the range of about 3 to about 125. The Babcock number is obtained by the following procedure. A 100 milliliter Cassia flask is provided with a 50 milliliter sample of the working solution and to this sample 30 milliliters of 50 percent sulfuric acid is added and thoroughly admixed. The flask is then placed in a hot water bath having a temperature between 82 degrees centigrade and boiling. The level of the water should be high enough to cover most of the neck of the Cassia flask. After about 15–30 minutes, sufficient hot, boiled water is added to bring the oily layer into the graduated neck of the flask. When the oily layer has separated sharply, usually in about five minutes, the difference between the readings of the lower and upper edges of the oily column in the neck is the Babcock number. The titration number is obtained by the following procedure. Fifty milliliters of the hot lubricant solution at about 70 degrees centigrade, is put into a beaker with about 120 milliliters of hot water. This solution is then titrated with .88N H_2SO_4. The titration number is the number of milliliters of the H_2SO_4 needed to titrate the lubricant solution to the bromcresol green end point.

Such working solutions may be applied to the metal surfaces to be deformed in various ways, such as by immersion, flow coating, by spraying or by roller application. For such applications, the lubricant temperature may be widely varied, from about room temperature up to about 100 degrees centigrade. Generally, for application by immersion techniques, higher temperatures of the lubricant composition are desired, such as 60 to 100 degrees centigrade, while for roller applications, lower temperatures, e.g., room temperature to 60 degrees centigrade may be used. It has been found, however, that in the latter instance, i.e., the roller application of the lubricant composition at room temperature, it may be desirable to use the lubricant composition as a concentrate with no further dilution, applying the composition to the rolls with air pressure, a reciprocating piston pump or a centrifugal pump. It is believed that the details of the various techniques whereby the lubricant composition of the present invention may be applied are sufficiently familiar to those in the art that further description of the details of such methods are not necessary.

As has been noted hereinabove, the lubricant composition of the present invention is particularly adapted for application to chemically coated metal surfaces, prior to deformation of the surfaces. Suitable chemical coatings which may be applied to the surfaces prior to the application of the subject lubricant composition include phosphate coatings, oxide coatings, oxalate coatings, sulfide coatings, and the like.

In forming metal articles, in accordance with the process of the present invention, the subject lubricant composition is applied to a chemically coated metal surface, using application techniques as have been indicated hereinabove, to obtain the desired amount of the lubricant composition on the surface, the lubricant coating obtained is dried and thereafter the coated surface is subjected to drawing, cold forming, or other deforming operation. Generally, the application of the chemical coating to the metal surface, such as a phosphate coating, is preceded by a cleaning or pickling step and a rinse to remove the cleaning or pickling solution. Frequently, between the ap-

plication of the chemical coating and the lubricant coating, the chemically coated surface is also rinsed to remove any unreacted coating material. Although this latter rinse may be a water rinse, alkaline or neutralizing rinses are also frequently used. It is believed that the composition and nature of the various chemical coating materials, as well as the manner in which they may be applied to the metal surfaces and the specific details of the various metal deforming operations, such as cold forming and drawing, are all sufficiently well known to those in the art that a further and detailed description of these compositions and processes is not necessary.

It has been found that the formed metal articles produced in accordance with the procedure indicated above are more easily cleaned to effect removal of the residual lubricant and coating film from the surface prior to subsequent finishing operation, than are articles produced using a conventional soap type lubricant of the prior art over the chemical coating. Moreover, it has been found that the lubricant compositions of the present invention may be applied to the surfaces to be deformed at lower temperatures than have heretofore been possible with the prior art soap composition, particularly those containing pigments, such as borax. Additionally, in many instances, it has been found that the lubricant films produced from the compositions of the present invention have a greater hardness, thereby providing greater protection against scratches during mechanical handling of the pieces being processed. Similarly, it has been found that in many instances, the lubricant compositions of the present invention have increased corrosion resistance and do not tend to build up to an appreciable extent on the applicator rolls, when roller application techniques are used. In all instances, it has been found that the drawability of the metal surfaces which have been coated with the present lubricant composition is at least equal, and in many cases superior, to that obtained with the lubricants of the prior art.

In order that those skilled in the art may better understand the method of the present invention and the manner in which it may be practiced, the following specific examples are given. In these examples, unless otherwise indicated, the temperatures are given in degrees centigrade and parts are by weight.

EXAMPLE 1

A lubricant concentrate was made of 75 percent of a sodium tallow soap and 25 percent of tetrapotassium pyrophosphate (anhydrous). This lubricant material was then formulated into an aqueous working coating composition containing 0.75 pound of the solid composition per gallon of solution. 4 inch x 6 inch stainless steel panels were then immersed in the working solution for 1 minute, the solution being at a temperature of about 80 degrees centigrade. The panels were then removed from the lubricant composition and dried. After drying there was formed on the panels a substantially uniform coating which was gray to whitish in color.

EXAMPLE 2

In 740 grams of water there was dissolved 0.2 gram of bismarck brown dye. Thereafter, 84.5 grams of potassium hydroxide were added to the solution and dissolved therein and the solution was heated to about 95 degrees centigrade. Eighteen grams of sodium nitrite, 27 grams of borax (decahydrate) and 523 grams of tetrapotassium pyrophosphate (anhydrous) were then added to the heated water solution. Four hundred and fifty-four grams of stearic acid-palmitic acid mixture were melted and then heated to about 95 degrees centigrade. To this melt were added 4.5 grams of pine oil and the fatty acid-pine oil mixture was then added to the previously prepared water solution with stirring. The resulting mixture was held at about 95 degrees centigrade for about 20 minutes, the solution being stirred to effect thorough mixing and reaction. Thereafter, the solution was cooled, with stirring, to

room temperature. An aqueous working lubricant composition was then made up containing 1.5 pounds of the lubricant concentrate per gallon of solution. This material, at a temperature of about 75 degrees centigrade, was applied by means of a roller coater to automobile bumper blanks which had previously been coated with a conventional zinc phosphate coating. After application of the lubricant, the resulting film was dried and the thus-coated blanks were subjected to a standard cold forming operation. A total of about 1000 bumper blanks were treated in this manner and in each instance, a satisfactory bumper was obtained.

EXAMPLE 3

A lubricant concentrate was formulated by admixing 46.7 percent of a sodium tallow soap, 20 percent of borax (decahydrate) and 33.3 percent of anhydrous tetrapotassium pyrophosphate. An aqueous working lubricant composition containing one pound per gallon of the concentrate was formulated and this material, at room temperature, was roller coated onto an oxalate steel coated surface, a reciprocating piston pump being used to supply the lubricant composition to the coating roll. After drying of the thus-applied coating, there was obtained a substantially uniform lubricating coating on the metal surface which was suitable for protecting the metal during relatively severe drawing operations.

EXAMPLE 4

One gallon of an aqueous soap solution, containing 227 grams of sodium tallow soap was made up and held at a temperature of 80 degrees centigrade. 4 inch x 8 inch steel panels which had been previously coated with a conventional zinc phosphate coating were then dipped into this soap lubricant solution and held therein for one minute. The panels were removed from the solution and were oven dried for 15 minutes at about 110 degrees centigrade. Additions of tetrapotassium pyrophosphate were then made to the aqueous soap solution, and zinc phosphate coated steel panels were dipped into the soap composition after each addition of the tetrapotassium pyrophosphate and were then oven dried, following the procedure as indicated above. All of the panels were weighed prior to the application of the soap composition and then were reweighed after the soap composition was dried on the panels. The gain in weight was the amount of the soap lubricant composition which was deposited on the panels. After the second weighing, the panels were then cleaned by being held in overflowing hot water at a temperature of about 71 degrees centigrade for 30 seconds, removed from the hot water and quickly immersed and immediately removed from a cold water dip and then blown dry with air. At this point, the panels were reweighed and the loss in weight was taken to be the amount of coating removed by cleaning of the panels. To determine the percent of cleaning obtained with each of the panels, the weight of the coating removed by the cleaning procedure was divided by the weight of soap lubricating composition applied to the panels, all multiplied by one hundred. Obviously, where this value is greater than 100, it indicates that in addition to the soap coating composition, some of the previously applied phosphate chemical coating composition was also removed. Using the procedure as described hereinabove, the following results were obtained:

Percent TKPP in soap composition:	Percent of coating composition removed by cleaning
0	100
1	101
4	109
10	123
25	147
50	132
100	114

EXAMPLE 5

The procedure of Example 4 was repeated with the exception that the aqueous soap composition which was used to coat the panels and to which the tetrapotassium pyrophosphate was added was a one gallon solution containing 113.5 grams of borax and 113.5 grams of sodium tallow soap. Using the procedure of Example 4 with this composition, the following results were obtained:

Percent TKPP in soap composition:	Percent of coating composition removed by cleaning
0	94
10	102
25	132
50	126
100	111

EXAMPLE 6

One gallon of an aqueous soap composition was made up as in Example 4. Steel bar stock which had been previously coated with a conventional zinc phosphate coating was then coated with this composition and the coating dried on the stock, using the procedure as set forth in Example 4. As in Example 4, additional bar stock was treated in the same manner after additions of tetrapotassium pyrophosphate were made to the soap composition. The treated bar stock was then run through a lube test machine, using the lubricity die to simulate the conditions encountered in a severe drawing operation. Thereafter, the bar stock was cleaned in accordance with the procedure set forth in Example 4 and the amount of coating removed from the bar stock by the cleaning, in milligrams per square foot of surface treated, was determined. Using this procedure, the following results were obtained:

Percent TKPP in soap composition:	Amount of coating removed by cleaning in milligrams/square foot
0	52
33	292
66	100
100	347

As seen from the results obtained in Examples 4–6, it is apparent that when using compositions containing tetrapotassium pyrophosphate, the cleaning of the metal surfaces treated is facilitated, in terms of removing the soap lubricant, as well as, in many instances, removing the phosphate chemical coating previously applied. Moreover, it is seen that these improved cleaning results are obtained even with the composite coating of the lubricant and the phosphate coating as obtained in a deforming operation.

The procedure of the above examples was repeated using soap lubricant compositions containing tetrasodium pyrophosphate and rosin acid, applied over oxide chemical coatings and sulfide chemical coatings, and similar results were obtained.

While there have been described various embodiments of the invention, the compositions and methods described are not intended to be understood as limiting the scope of the invention, as it is realized that changes therewithin are possible, and it is further intended that each element recited in any of the following claims is to be understood as referring to all equivalent elements for accomplishing the same results in substantially the same or equivalent manner, it being intended to cover the invention broadly in whatever form its principle may be utilized.

What is claimed is:

1. A lubricant composition useful in lubricating chemically coated metal surfaces prior to deformation which comprises the admixture of from about 15 to about 97 percent by weight of an alkali metal soap of a fatty acid containing 8–22 carbon atoms and from about 3 to about 75 percent by weight of an alkali metal pyrophosphate.

2. The lubricant composition as claimed in claim **1** wherein there is also contained water in an amount up to about 80 percent by weight.

3. The lubricant composition as claimed in claim **2** wherein the alkali metal pyrophosphate is tetrapotassium pyrophosphate.

4. A method of lubricating chemically coated metal which comprises coating the chemically coated metal with the lubricant composition as claimed in claim **1** prior to deforming.

5. The method as claimed in claim **4** wherein the lubricant composition is an aqueous solution of a concentrate which concentrate is the admixture of from about 15 to about 97 percent by weight of an alkali metal soap of a fatty acid containing 8–22 carbon atoms, from about 3 to about 75 percent by weight of an alkali metal pyrophosphate and water in an amount up to about 80 percent by weight.

6. A chemically coated metal surface having deposited thereon a lubricating amount of the lubricant composition as claimed in claim **1**.

7. The coated metal surface as claimed in claim **6** wherein the chemical coating is a phosphate coating, the alkali metal pyrophosphate is tetrapotassium pyrophosphate and the fatty acid is a mixture of stearic acid and palmitic acid.

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