Patented Dec. 15, 1953

2,662,836

UNITED STATES PATENT OFFICE

2,662,836

PREPARING METAL SURFACES FOR DRAWING OPERATIONS

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No Drawing. Application January 6, 1950, Serial No. 137,282

15 Claims. (Cl. 117—167)

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This invention relates to the class of lubricants known as "drawing compositions" which are used to lubricate the surfaces of metals while they are being drawn or deformed under pressure through or between dies. More particularly, the invention relates to drawing compositions for metals having protective surface coatings and to methods of preparing such metals for drawing so that scratching, tearing, or fracturing of the metals is minimized.

In the commercial deformation of metal, it is customary to dispose a suitable lubricating composition between the metal and the deforming instrument, such as a die or cutting tool. When metal is deformed by a drawing operation, it is subjected to a stretching and thinning out action that is accompanied by a considerable flowing of the metal in various directions. This movement of metal takes place under high pressure, and it is to control this flow and prevent metal-to-metal contact between the work and the die that drawing compositions are employed.

For many years, most drawing compositions have been composed essentially of oils, fats, fatty acids, or soaps, or various mixtures thereof. These materials have been used both without dilution and in the form of water emulsions. Pigments have frequently been included in these compositions as inexpensive materials in the nature of fillers to increase the body or viscosity of the compounds and prevent their being squeezed out from between the work and the die, and also to compensate for the excessive solubilities of some emulsified oils. Soaps alone, or water emulsions thereof, have been quite satisfactory where the metals being drawn are highly ductile or the amount of deformation required is not great, so that only relatively low pressures are encountered. When drawing less ductile metals, such as steel, and particularly when "deep drawing" is practiced, however, the pressures involved are very great, and the lubricating qualities of soaps are generally insufficient even when used in high concentration. Oily materials are customarily substituted in whole or in part for soaps in operations involving very high pressures.

Other considerations that govern the suitability of particular types of drawing compositions for given conditions of use, are ease of application and removal of the compositions, their drying characteristics and sensitivity to varying atmospheric conditions, compatibility with mineral oil films commonly found on sheet steel, and other practical considerations of greater or lesser importance in different situations.

With the introduction to the metal drawing industry of steels having specially treated surfaces for facilitating drawing of the metal and for protecting it against corrosion by various agents, such as atmospheric moisture and industrial

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fumes, a new problem in producing suitable drawing compositions was presented. The special steel surfaces that have been developed are numerous and include surfaces containing complex compounds of phosphates, oxalates, sulfides, fluorides, chromates, etc., including combinations of these compounds with each other and with various modifying ingredients. A characteristic of these surfaces is that they are formed by chemical reaction of various reagents with, and consequent chemical modification of, the outer skin or surface of the steel. This involves the formation on the steel of an integral skin or layer of a complex crystalline, ferrous compound that is relatively porous and has capillary properties not detectable in the untreated steels.

With the advent of such surface treated steels, it was found that drawing compositions formerly considered superior for deep drawing operations commonly failed to show the expected superiority when applied to the surface treated steels; and a number of drawing compositions considered quite inferior when applied to ordinary steels were found to be better than was expected when applied to the surface treated steels. Apparently the chemical and/or physical characteristics of the outer crystalline layers of the surface treated steels are such that the crystals are completely wetted with difficulty. This may also have an appreciable effect upon the penetration of drawing compositions into the capillary crevices or joints between the crystals of the modified surface layer, and also upon the tendency of adsorbed drawing compositions to be squeezed out of the surface layer and out from between the work and the die during the drawing operation.

Though the explanation is still somewhat obscure, it has been generally recognized for quite some time that the suitability of a drawing composition for use on untreated steels does not insure that the same composition will be at all suitable for use on the various types of surface treated steels, or that a normally inferior drawing composition will still deserve its inferior classification when used on the surface treated steels. As a result, various modifying agents, such as wetting agents and the like, have been added to different types of drawing compositions in an attempt to improve their action under the peculiar conditions brought about by the surface treatments referred to above. While the modification of prior art drawing compositions to render them more suitable for use on surface treated steels has produced some improvements, the art is still faced with a need for more satisfactory drawing compositions for use on these metals.

One of the principal difficulties with prior art drawing compositions, when applied to surface treated steels, is that the results obtained with a given composition have frequently been incon-

sistent. This has commonly been attributed to improper preliminary surface treatment of the steels rather than to inconsistencies in the behavior of the drawing compositions.

We have found that these inconsistencies need not occur if a properly formulated drawing composition is employed. Successful operation is dependent upon the ability of the lubricant to wet the treated surface and to creep or penetrate into the pores thereof; and also upon the ability of the lubricant to desorb from the treated surface under pressure and maintain a more or less continuous lubricant film between the die and the work. In addition, of course, the drawing composition must have the required characteristic of stability, ease of application, and ease of removal.

Adequate wetting of the treated surface of the steel and penetration of the surface pores is readily obtainable with a large number of drawing compositions and may be enhanced by the use of wetting agents when the natural wetting properties of the drawing composition are not sufficient. The principal difficulty with the prior art drawing compositions when used on surface treated steels, however, appears to have been due to inability of the compositions to desorb from the surfaces under pressure.

The present invention is based upon the discovery that the water-soluble fatty acid soaps having the desired lubricating and wetting characteristics for many types of drawing operations, when mixed with a hydraphobic alcohol, and dispersed in water, produce drawing compositions having to a high degree the characteristics of satisfactory wetting and penetration together with satisfactory desorption from the treated surfaces under pressure. The alcohol provides in the composition a hydrophobic component which controls the desorption property and also plasticizes the soap.

The property of desorption of the lubricant is closely associated with the kind and amount of alcohol in the composition. While the amount is not highly critical in the sense of having any precise limits, it is essential that the alcohol constitute from about 25% to about 50% of the composition by weight. Similarly, as regards the kind of alcohol, there is no exact boundary or limitation on the classes of alcohols that are suitable. The aliphatic monohydric alcohols having 10 or more carbon atoms are quite insoluble in water and have the required hydrophobic character, and these alcohols as a class are highly satisfactory. However, cyclic and cycloaliphatic alcohols, such as cholesterol, abietyl alcohol, and hydroabietyl alcohol, are also satisfactory. Apparently the only critical limitation is that the alcohol component be equivalent in its hydrophobic character to an aliphatic monohydric alcohol having at least 10 carbon atoms. In this connection it may be noted that a six carbon ring is equivalent in hydrophobicity to about 3½ aliphatic carbon atoms, so that the suitable cyclic and cycloaliphatic alcohols must all have more than 10 carbon atoms. Where the alcohols are polyhydric, a greater number of carbon atoms in the molecule is required to give the required degree of hydrophobicity. For purposes of definition, therefore, the suitable alcohols may be defined as those having a degree of hydrophobicity at least as great as that of a monohydric aliphatic alcohol having 10 carbon atoms.

The fatty acid soaps which are suitable for use as the other principal constituent of the drawing compositions comprise the water-soluble sodium and potassium soaps of the saturated and unsaturated fatty acids having from 10 to 20 carbon atoms in the molecule. While it is permissible, and sometimes desirable, to include small amounts of soaps of other acids for modifying the consistency of the drawing compositions, soaps of the aliphatic acids must preferably constitute at least the major portion of the soap component, and the soap component should comprise from 50% to 75% of the total disperse phase by weight. Suitable modifying soaps for combination with the fatty acid soaps are the water-soluble metallic soaps of naphthenic acids, rosin acids, tall oil acids, and the like.

If desired, the composition may also contain a minor proportion of a natural or synthetic fatty oil. In general, the polyhydric alcohol esters of fatty acids having from 10 to 20 carbon atoms in the molecule are suitable for use as the fatty oil component of the compositions. Among the fatty oils that may be satisfactorily used are: the naturally occurring vegetable oils, such as linseed, soya bean, castor, cotton seed, rape-seed and the like, modifications thereof such as blown oils of the unsaturated types, chemical modifications of these oils such as result from the substitution of glycol, ethylene glycol, pentaerythritol, mannitol, sorbital, and the like for the glycerol component of natural oils, including the partial esters of such alcohols.

The preparation of the compositions merely involves melting the fatty acids, the alcohol, and the fatty oil, if any; adding sufficient alkali dissolved in water to react with the fatty acid; heating the mixture to from about 180° to about 190° F.; neutralizing any unreacted alkali with a suitable acid such as oleic acid; and adding the balance of the water required to form an emulsion of the desired consistency. Because of the readily emulsifiable character of the components of the compositions, the use of additional emulsifying agents and stabilizers is generally unnecessary.

To further illustrate the nature and scope of the invention, reference is made to the following specific examples in which representative compositions embodying the invention are described for illustrative purposes, all proportions being by weight.

EXAMPLE 1

50 parts potassium oleate
25 parts cetyl alcohol
25 parts tallow (fatty acid glycerides)

A mixture of these components in the proportions set forth were dispersed in sufficient water to form a stable water in oil emulsion as a concentrate for dilution with additional water before use to a concentration of 15% to 25% disperse phase. In this example, the cetyl alcohol is a normally solid, water-insoluble alcohol. Stearyl alcohol may be substituted with little or no change in the properties of the composition.

EXAMPLE 2

75 parts potassium oleate
25 parts oleyl alcohol

A mixture of these components was dispersed in water as in Example 1. In this example the alcohol is normally a liquid and the fat (tallow) is omitted.

EXAMPLE 3

50 parts sodium oleate
50 parts oleyl alcohol

A mixture of these components was dispersed in water as in Example 1. The higher proportion of alcohol to soap in this example, as compared with Example 2, is preferable because the use of a sodium soap rather than a potassium soap requires a greater amount of plasticizing agent, and the additional alcohol serves this purpose.

EXAMPLE 4

To form the soap in situ, the following reagents and formulation procedure may be employed:

26.0 parts tallow
19.6 parts potassium hydroxide (24% aqueous)
25.0 parts oleyl alcohol

The tallow and the alkali are first reacted in a conventional manner to form a soap and the alcohol is then added. The resulting mixture is dispersed in water as in Example 1.

EXAMPLE 5

An alternative procedure is exemplified by the use of wool fat as the source of both the fatty acid and the alcohol components. Wool fat is a complex mixture comprising about 40–50% saturated and unsaturated fatty alcohols of various carbon chain lengths up to about C_{27}, about 40–50% saturated and unsaturated fatty acid esters of higher fatty alcohols, minor amounts of free fatty acids, and minor amounts of unsaponifiable and ether-insoluble constituents and other compounds that vary with the source and recovery methods used. In this case, 30 parts of wool fat and 12 parts of 24% aqueous potassium hydroxide are agitated together at 180° F. for 12 hours, after which time any unreacted alkali is neutralized with oleic acid. The resulting alcohol-soap mixture is dispersed in water as in Example 1.

Sperm oil may be substituted for wool fat in this example without otherwise changing the procedure. Sperm oil is a complex mixture of esters of fatty acids (C_{14} to C_{20}) and fatty alcohols (C_{16} to C_{20}) which are preponderantly unsaturated in the acid radical, in the alcohol radical, or in both. Saponification produces the soaps of the acids and the free alcohols.

EXAMPLE 6

Other suitable combinations of starting materials are the following:

A

192 parts lauric acid
125 parts oleyl alcohol
125 parts monoglycerol stearate
58 parts potassium hydroxide (anhydrous)
500 parts water

B

222 parts Neo-fat No. 19 (drying oil acid)
100 parts stearyl alcohol
25 parts abietyl alcohol
125 parts ethylene glycol laurate
28 parts sodium hydroxide (anhydrous)
500 parts water

C

175 parts palmitic acid
300 parts sperm oil
25 parts sodium hydroxide (anhydrous)
500 parts water

D

200 parts stearic acid
50 parts naphthenic acid (200 av. mol. wt.)
200 parts cetyl alcohol
50 parts potassium hydroxide (anhydrous)
500 parts water

E

250 parts stearic acid
60 parts ricinoleic acid
150 parts cetyl alcohol
40 parts sodium hydroxide (anhydrous)
500 parts water

F

263 parts tall oil (25% rosin content)
200 parts oleyl alcohol
37 parts sodium hydroxide (anhydrous)
500 parts water

Preparation

Melt the fatty acid, alcohol, and polyhydric alcohol ester (where employed) at about 150° F., add the alkali dissolved in about 5 parts of water, heat to about 190° F., and add the balance of the water.

Usage

Dilute the concentrate with from 1 to 2 parts of water for use as a dip for the surface treated steel to be drawn. Formulae A, B, and F are particularly formulated for application to steels having heavily treated surfaces (coating weights of more than 50 mg. per sq. ft. in the case of the phosphated surfaces), and Formulae C, D, and E are preferred for more lightly treated steel surfaces. The residues formed on the steel surfaces by Formulae C, D, and E are more stiffly plastic than those of Formulae A, B, and F.

As explained above, the drawing compositions of this invention have been found to be highly successful when applied to what have been termed "surface treated steels," for which the prior art drawing compositions have generally not been satisfactory. The above described advantages have been achieved to a particularly high degree when the compositions of the invention are applied to steels that have been previously treated with phosphoric acid or metallic phosphate salts to form various kinds of phosphate surfaces on the steels. Variations of these surface treating processes are disclosed, for example, in the following United States Letters Patent:

Patent No.	Inventor	Date
1,750,270	Elmer M. Jones	Mar. 11, 1930.
1,755,391	Mathew Green et al	Apr. 22, 1930.
1,761,186	Marlin C. Baker et al	June 3, 1930.
1,791,715	Van M. Darsey	Feb. 10, 1931.
1,842,085	Mathew Green et al	Jan. 19, 1932.
1,860,505	Elmer M. Jones	May 31, 1932.
1,869,121	John S. Thompson et al	July 26, 1932.
1,873,363	Robert R. Tanner	Aug. 23, 1932.
1,888,189	do	Nov. 15, 1932.
2,293,716	Van M. Darsey	Aug. 25, 1942.
2,293,779	Robert R. Tanner	Do.
2,295,545	Wilfred J. Clifford et al	Sept. 15, 1942.
2,298,280	do	Oct. 13, 1942.
2,301,209	Robert C. Gibson	Nov. 10, 1942.
2,302,510	Robert R. Tanner	Nov. 17, 1942.
2,303,242	do	Nov. 24, 1942.
2,314,565	John S. Thompson	Mar. 23, 1943.
2,314,887	Herman J. Lodeeson et al	Mar. 30, 1943.
2,335,868	Herman J. Lodeeson	Dec. 7, 1943.
2,336,071	Wilfred J. Clifford et al	Do.
2,336,072	do	Do.

The chemical compositions of the surface skins formed on ferrous metals by the processes disclosed in the above patents are rather uncertain in many cases. In all of the patents listed, however, a compound containing a phosphate radi-

cal, generally either in the form of an acid or a metallic salt, is an essential ingredient of the surface treating compositions employed. It appears that the iron or steel being treated reacts chemically with the treating composition and forms, in situ, an integral skin on the treated metal, and that this skin comprises as an essential constituent a complex, including both iron and phosphate radicals. As stated above, this skin is of a crystalline nature and is relatively porous compared to ordinary iron and steel surfaces. The need for the use of the special drawing lubricants of the present invention is believed to result both from the capillary cracks or crevices in the treated metal surfaces and from the chemical and/or physical characteristics of the skin resulting from its chemical composition. For the purposes of this application, ferrous metal surfaces of this broad class are generically referred to hereinafter and in the claims as "phosphated" surfaces.

Similarly, the compositions of the invention are of especial value for use on ferrous metals having various complex ferrous sulfide surfaces formed thereon. Representative examples of processes for forming such sulfide surfaces are disclosed in the following United States Letters Patent:

Patent No.	Inventor	Date
2,301,983	Robert R. Tanner	Nov. 17, 1942.
2,302,643	John S. Thompson	Do.
2,313,925	Leo P. Curtin	Mar. 16, 1943.
2,318,642	John S. Thompson	May 11, 1943.
2,327,602	do	Aug. 17, 1943.

These surfaces are hereinafter generically referred to as "sulfide" surfaces.

Another type of surface for which the compositions of the invention are of especial value is one comprising an oxalate as the essential film or skin component. Representative examples of processes for forming such surfaces are disclosed in the following United States Letters Patent:

Patent No.	Inventor	Date
2,137,968	Robert R. Tanner	Nov. 22, 1938.
2,273,234	do	Feb. 17, 1942.

These surfaces are hereinafter generically referred to as "oxalate" surfaces.

Other complex ferrous surfaces comprehended by the present invention consist essentially of the products of reaction of the ferrous metal being treated with fluorides and/or chromates, as exemplified by the processes of the following United States Letters Patent:

Patent No.	Inventor	Date
2,276,353	John S. Thompson	Mar. 17, 1942.
2,315,564	do	Apr. 6, 1943.

Surfaces having a complex fluoride as an essential ingredient are hereinafter generically termed "fluoride" surfaces. The surfaces formed essentially by reaction of an acid of chromium with the ferrous metal being treated are comprehended by the term "chromate" surfaces.

For purpose of further definition, the different classes of surfaces mentioned herein (with untreated iron surfaces as a standard of comparison) are hereinafter designated "relatively porous integrally formed surfaces of a crystalline ferrous, metal complex"; and the invention may be summarized as comprehending the preparation of ferrous metals having any of the various surfaces of this character to lubricate the surfaces during subsequent drawing operations, i. e., deforming operations performed upon the metal by the application of pressure thereto.

While the drawing compositions of the present invention are particularly suited for application to surface treated metals by dipping the metals into a bath of the drawing compositions, other methods of applications well known in the art may be employed when desired. For instance, the drawing compositions may be applied to the metal surfaces by brushing or spraying, or by any other device suitable for spreading the drawing compositions uniformly over a surface.

When dealing with surfaces that have not been carefully or completely pretreated to form thereon a continuous corrosion resistant surface, it is sometimes desirable to include corrosion inhibitors in the drawing compositions of the invention. Corrosion inhibitors such as the alkali nitrites and chromates, for example, may be incorporated in the drawing compositions to minimize corrosion under such circumstances.

Having described our invention, we claim:

1. The process of preparing ferrous metals for drawing operations when said metals have been previously treated to produce thereon relatively porous integrally formed surfaces of a crystalline ferrous metal complex, said process comprising applying to said surfaces a drawing composition consisting essentially of a mixture of water-soluble alkali metal soap and water-insoluble alcohol, said soap comprising by weight from about 50% to about 75% of said mixture and at least the major proportion of said soap being soap of fatty acids having at least 10 carbon atoms, and said alcohol comprising from about 50% to about 25% of said mixture by weight and having a degree of hydrophobicity at least as great as a 10 carbon atom monohydric aliphatic alcohol, said mixture being applied in the form of an emulsion in water.

2. The process of claim 1 in which the alkali metal of said soap is selected from the class consisting of sodium and potassium.

3. The process of claim 1 in which said mixture also contains a fatty oil in an amount not exceeding 25% of the mixture by weight.

4. The process of preparing ferrous metals for drawing operations when said metals have been previously treated to produce thereon relatively porous integrally formed surfaces of a crystalline ferrous metal complex, said process comprising applying to said surfaces a drawing composition consisting essentially of a mixture of water-insoluble alcohol and water-soluble soap selected from the class consisting of alkali metal soaps having from 10 to 20 carbon atoms, said alcohol comprising by weight from about 25% to about 50% of said mixture and having a degree of hydrophobicity at least as great as a 10 carbon atom monohydric aliphatic alcohol, and said soap comprising from about 75% to about 50% of said mixture by weight, at least the major proportion of said soap being soap of fatty acids, said mixture being applied in the form of an emulsion in water.

5. The process of claim 4 in which the alkali metal of said soap is selected from the class consisting of sodium and potassium.

6. The process of claim 4 in which said mix-

ture also contains a fatty oil in an amount not exceeding 25% of the mixture by weight.

7. The process of preparing ferrous metals for drawing operations when said metals have been previously treated to produce thereon relatively porous integrally formed surfaces of a crystalline ferrous metal phosphate complex, said process comprising applying to said surfaces a drawing composition consisting essentially of a mixture of water-soluble alkali metal soap and water-insoluble alcohol, said soap comprising by weight from about 50% to about 75% of said mixture and at least the major proportion of said soap being soap of fatty acids having at least 10 carbon atoms, and said alcohol comprising from about 50% to about 35% of said mixture by weight and having a degree of hydrophobicity at least as great as a 10 carbon atom monohydric aliphatic alcohol, said mixture being applied in the form of an emulsion in water.

8. The process of claim 7 in which the alkali metal of the soap component is selected from the class consisting of sodium and potassium.

9. The process of claim 7 in which the major proportion of the alcohol component is selected from the class consisting of monohydric aliphatic alcohols having at least 10 carbon atoms.

10. The process of claim 7 in which said dispersed phase also contains a fatty oil in an amount not exceeding 25% of the dispersed phase by weight.

11. The process of claim 7 in which the alkali metal of the soap component is selected from the class consisting of sodium and potassium, the major proportion of the alcohol component consists of normal monohydric aliphatic alcohols having at least 10 carbon atoms, and the dispersed phase also contains a fatty oil in an amount not exceeding 25% of the dispersed phase by weight.

12. The process of preparing ferrous metals for drawing operations when said metals have been previously treated to produce thereon relatively porous integrally formed surfaces of a crystalline ferrous metal phosphate complex, said process comprising applying to said surfaces a drawing composition consisting essentially of a mixture of water-insoluble alcohol and water-soluble soap selected from the class consisting of alkali metal soaps having from 10 to 20 carbon atoms, said alcohol comprising from about 25% to about 50% of said mixture by weight and having a degree of hydrophobicity at least as great as a 10 carbon atom monohydric aliphatic alcohol, and said soap comprising from about 50% to about 75% of said mixture by weight, at least the major proportion of said soap being soap of fatty acids, said mixture being applied in the form of an emulsion in water.

13. The process of claim 12 in which said mixture is applied in the form of a water emulsion and is then allowed to dry.

14. The process of claim 12 in which the alkali metal of said soap is selected from the class consisting of sodium and potassium.

15. The proces of claim 12 in which said mixture also contains a fatty oil in an amount not exceeding 25% of the mixture by weight.

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