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LUBRICANTS FOR COATING AND WORKING LIGHT METALS

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This invention relates to lubricants for use in working and protectively coating aluminum and magnesium, and alloys containing greater than 70 percent by weight of one of these metals. More particularly, the present invention concerns an improved method of working aluminum and magnesium, and said alloys of these metals, by using certain lubricants as hereinafter described.

Many problems are encountered when a piece of light metal such as aluminum or magnesium is worked, i. e. deformed, cut, or otherwise altered in shape, size, or section by the action of a tool as during extrusion, drawing, forging, pressing, broaching, machining, cutting, rolling, sawing, drilling, and the like. One of the most troublesome of these problems relates to contamination of the tool whereby some of the light metal being worked is picked up or seized by the tool and becomes attached thereto. This tool contamination, or "pick up" as it is called, often materially affects the quality of the article being worked in that the surface of the article is frequently rough or marred by imperfections. As is well known, tool pick up increases in severity with rise in temperature of the surface of the tool as well as the light metal being worked and is more severe in working magnesium than aluminum. In commercial metal-working practice, much effort has been concentrated towards reducing the amount of tool contamination by interposing lubricants such as mineral, vegetable, and animal oils between the surface of the tool and the light metal workpiece. Unfortunately, however, such lubricants are often not very effective in this capacity.

The use of conventional lubricants in woking light metals also introduces other problems which are frequently as serious as the problem of tool contamination itself. For example, some of these metal working lubricants form carbonaceous deposits on the light metal during hot working or during annealing following working. Some lubricants require special organic solvents to remove them from the work-piece or are otherwise difficultly removable therefrom.

It is therefore an object of this invention to provide lubricants for lubricating light metal bars, rods, sheets, and the like, especially during the pressure forming thereof, said lubricants being substantially non-corrosive to, easily removable from, and non-carbonizing on the light metal surfaces. It is also an object of this invention to provide an improved method of working aluminum and magnesium, and alloys containing greater than 70 percent by weight of one of these metals, whereby tool contamination and the effects thereof at the interface of the tool and light metal are prevented or substantially decreased. A particular object is to provide an improved method of forming light metals in pressure dies, such as extruding, drawing, pressing, and the like. Another object is to provide light metal surfaces which have good physical properties and are protectively coated against corrosion and abrasion. Other objects and advantages will be apparent from the following description which describes but does not limit the invention.

These objects are accomplished in accord with the present invention as hereinafter explained. It has now been found that tool contamination during the working of light metals, and the effects thereof at the interface of the tool and light metal, can be prevented or substantially decreased by maintaining at said interface a metal working lubricant comprising an alkali or alkaline earth metal alkyl phosphate having from 1 to 20 carbon atoms in the alkyl group. Of these compounds, the monoalkyl metal phosphate compounds having from 8 to 20 carbon atoms in the alkyl group are particularly good metal working lubricants, e. g. the octyl, decyl, dodecyl, tridecyl, cetyl, and octadecyl metal phosphates. In the metal alkyl phosphates of the invention, the metal may be any of the alkali metals or alkaline earth metals. Thus, the metal alkyl phosphates include disodium octyl phosphate, dipotassium decyl phosphate, dilithium tridecyl phosphate, and calcium cetyl phosphate. Of these compounds, the monoalkyl phosphates of sodium and potassium are preferred, especially the disodium monoalkyl phosphates such as disodium octyl phosphate and disodium cetyl phosphate.

In working aluminum or magnesium metals with the aid of an alkali or alkaline earth metal alkyl phosphate, the metal alkyl phosphate may be dissolved in a suitable liquid organic solvent which is essentially non-corrosive both to the tool and the light metal, e. g. an aliphatic alcohol or glycol. Such a solution of the metal alkyl phosphate may then be applied to the tool or the light metal, or both, as by wiping, pouring, or spraying the solution thereupon. Depending upon the temperature of the metal working operation, almost any of the liquid monohydric aliphatic alcohols are suitable organic solvents, viz., ethyl, propyl, butyl, amyl, hexyl, octyl, decyl, dodecyl, and tridecyl alcohols. Both branched and straight chain alcohols may advantageously be used, especially a primary or secondary alcohol having a hydroxyl group located at or near the end of the carbon atom chain. The alkyl phosphates may also be dissolved in glycols such as liquid mono-, di-, and poly-glycols, especially polypropylene glycols having average molecular weights of from about 300 to about 1000. A minor proportion, i. e. less than 50 percent, and ordinarily from 1 to 10 percent of the metal alkyl phosphate is dissolved in a major proportion of the alcohol or glycol, i. e. greater than 50 percent by weight thereof. Since liquid monohydric aliphatic alcohols having from 8 to 13 carbon atoms in the molecule are good metal-working lubricants in their own right, metal alkyl phosphates dissolved in such alcohols form unique metal-working compositions which are particularly effective for use in rolling aluminum and magnesium.

Alternatively, the alkali or alkaline earth metal alkyl phosphate may be dissolved or dispersed in water. Or a solution of the metal alkyl phosphate in a suitable organic solvent, e. g. an alcohol or a glycol, may sometimes be dissolved or emulsified in water. Solutions, dispersions, and emulsions such as these may be employed in metal working operations. Concentrations of the metal alkyl phosphate of from 1 to 20 percent by weight of the total phosphate-water solution (dispersion or emulsion) are generally satisfactory for practicing the method of the invention, although concentrations from 0.1 to 40 percent and higher may occasionally be used to advantage.

When the metal alkyl phosphate is insoluble in water, it usually becomes necessary to disperse it or to emulsify an organic solution of it in water by means of a dispersing or emulsifying agent, such as an alkyl or aryl sulfate or sulfonate; an alkyl, aryl, or alkaryl ether of a polyethylene glycol; a rosin or fatty acid ester of a polyethylene glycol, etc. The surface active agent may be added to water or the aqueous phase prior to mixing it with a

water-insoluble metal alkyl phosphate or an organic solution of such a phosphate e. g. an alcohol or glycol solution thereof. The concentration of surface active agent required to form a suitable dispersion or emulsion is usually at least 0.1 percent by weight and frequently from 1 to 10 percent by weight based on the metal alkyl phosphate or organic solution thereof, i. e. the water immiscible or non-aqueous phase.

The metal alkyl phosphate alone or in combination with other materials such as solvents, lubricants, surface active agents, etc. may be employed as an aqueous solution, dispersion, or emulsion in the working of light metals. Alternatively, the metal alkyl phosphate solution, dispersion, or emulsion may be applied, e. g. by spraying, to one or more surfaces of the light metal, especially the heated light metal, and partially or completely dried thereupon prior to working the metal. This latter procedure produces a coating on the light metal which is preferred for most hot working operations such as extruding, drawing, pressing, and the like. Such coatings may also be formed by applying a paste of the metal alkyl phosphate to the light metal work-piece, e. g. by dipping.

These coatings of alkali and alkaline earth metal alkyl phosphates on light metals have good separating power and prevent or substantially reduce adhesion, pick up, seizure, and tearing of the light metal by the tool. Such coatings also have good lubricity and thereby reduce friction between the tool and the surface of the light metal being worked. This property of being able to reduce friction is distinct from and not necessarily related to the ability to prevent adhesion of metal surfaces. In addition, metal alkyl phosphate coatings are unusually adherent to aluminum and magnesium during metal working operations such as rolling, drawing, pressing, and the like. Apart from their metal working properties, these coatings are simply applied and may easily be removed by water dissolution, especially hot water. Furthermore, the dry coatings serve to protect aluminum and magnesium metal from mechanical abrasion and atmospheric oxidation. In fact, the light metals themselves after being worked with the aid of these metal alkyl phosphates, have low corrosion rates as determined by salt-water immersion tests.

Alkali and alkaline earth metal monoalkyl phosphates are thermally quite stable and do not decompose to leave carbonaceous deposits on the surfaces of light metals during hot working or during annealing following metal-working operations. It is therefore frequently possible to work aluminum or magnesium with the aid of such a metal alkyl phosphate, then subsequently anneal and rework the light metal without any further addition of the phosphate or any other metal-working lubricant. For example, aluminum or magnesium sheet may be deep drawn with the help of a metal-working composition containing a large proportion of the alkali or alkaline earth metal monoalkyl phosphate and thereafter subjected directly to another deep draw without any interim treatment other than heating, e. g. annealing.

It has now been found that the thermostability of these alkali and alkaline earth metal monoalkyl phosphates, though good per se, may be improved for hot working light metals by the addition of an alkali metal hydroxide, viz. potassium, sodium, or lithium hydroxide. The heat stabilizing effectiveness of the alkali hydroxides increases in the order in which they are listed above, i. e. lithium hydroxide is more effective than sodium hydroxide, etc. Furthermore, a light metal coated with a lubricant composition consisting of a metal monoalkyl phosphate and at least 5 percent by weight of an alkali metal hydroxide may be effectively worked at a higher temperature than when coated with the metal monoalkyl phosphate alone. Good results may be obtained in working light metals coated with a lubricating composition consisting of disodium octyl phosphate stabilized with sodium hydroxide, disodium cetyl phosphate stabilized with sodium hydroxide, dilithium cetyl phosphate stabilized with lithium hydroxide, calcium cetyl phosphate stabilized with calcium hydroxide, etc. An adherent coating of such a lubricant composition, viz. the metal monoalkyl phosphate-alkali metal hydroxide composition, may easily be applied to the unheated light metal work piece and steel tool, e. g. die, by spraying it thereupon as an aqueous solution and subsequently heating the parts so coated to dryness before starting the metal working operation. Aqueous metal monoalkyl phosphate solutions, however, containing a minor proportion of the alkali metal hydroxide do not form adherent coatings when sprayed directly into a hot steel die, e. g. at 500° F. To cause aqueous metal monoalkyl phosphate-alkali metal hydroxide coatings to adhere to a steel die under such conditions, a major proportion of the alkali metal hydroxide is generally required, e. g. an admixture with the metal monoalkyl phosphate of the hydroxide containing up to 90 percent by weight thereof. Such compositions, as hereinafter shown, are satisfactory for coating and working the light metal work piece.

Other compounds in minor proportions may also be employed to improve the heat stability of these metal monoalkyl phosphates either with or without the use of alkali metal hydroxides. For example, improved results have been obtained when using greater than 5 percent by weight of an alkali or alkaline earth metal salt of a monohydroxy aliphatic alcohol having from 8 to 18 carbon atoms in the molecule, e. g. sodium or calcium cetylate. In such use, these heat stabilizing substances sometimes allow effective hot working of aluminum and magnesium at temperatures as much as 100° F. higher than is feasible in the absence of these stabilizers.

Trisodium phosphate is another compound which may be admixed with metal monoalkyl phosphates to improve their heat stability. Furthermore, when such compositions containing a minor though effective proportion of trisodium phosphate are applied to the surfaces of light metals in accord with the teachings of the invention, the resultant coatings are more stable to abrasion than coatings of metal monoalkyl phosphate compounds per se.

The following examples illustrate but do not limit the invention.

Example 1

The effectiveness with which alkali and alkaline earth metal monoalkyl phosphates may be employed in metal-working operations is well demonstrated in drawing blanks of a magnesium-base alloy sheet into cylindrical cups as hereinafter described. The nominal composition of the magnesium alloy was approximately 3 percent by weight of aluminum, 1 percent zinc, 0.3 percent manganese, and balance magnesium. The blanks were all .064 inch thick but of two different diameters, viz. 4 and 5 inches. These were drawn at reductions of 62.5 and 70 percent respectively into cups about 1.5 inches inside diameter with conventional drawing apparatus (an example of which appears in the U. S. Patent 2,396,218) having a die radius of 0.385 inch (6T) and a punch radius of 0.5 inch (8T). In drawing the cup, the work was lubricated with various alkali and alkaline earth metal monoalkyl phosphates and compositions thereof with trisodium phosphate or an alkali metal hydroxide. The lubricant was applied to both sides of the blank and to the die by spraying it thereupon as an aqueous solution or dispersion. In this manner, a heavy uniform coating of the lubricant was placed upon the surfaces subjected to rubbing or frictional forces during the draw. In each draw, the blank was first coated with the lubricant and then heated to the temperature of the draw, at which temper-

ature the lubricated blank was immediately drawn into the cup. In each instance, the monoalkyl phosphate was stable to decomposition at the temperature of the draw, the thermal stability being increased appreciably by the presence of trisodium phosphate and also by an alkali metal hydroxide. The blanks were easily drawn at speeds ranging from 5 to 15 inches per minute without any significant amount of scoring. The die was not coated with contaminating light metal after the draw. The drawn cups were rendered free from lubricant residue by an aqueous alkali cleaner followed by a water rinse. Corrosion tests of similarly lubricated and drawn cups, in which the cups were subjected to the corrosive action of a 3 weight percent solution of salt in water by immersion therein for 16 hours before cleaning, revealed no corrosion resulting from lubricant residue.

In the following table are listed the approximate compositions of the lubricants as well as the diameter of the blanks in inches, the temperature of the draw in degrees Fahrenheit, and the percent reduction obtained during the draw. These draws are comparable to the maximum draws obtainable with graphite at the same temperatures and using the same magnesium alloy and equipment. However, drawn cups with their graphite coatings are considerably more corrodible than those drawn in accord with the invention as shown by salt-water immersion tests.

Lubricant Composition	Diameter of Blank, inches	Temperature of Draw, ° F.	Percent Reduction
disodium octyl phosphate	5	450	70
3 parts disodium octyl phosphate and 1 part monobasic sodium phosphate	5	450	70
3 parts disodium octyl phosphate and 1 part trisodium phosphate	5	500	70
calcium cetyl phosphate	4	500	62.5
1 part disodium octyl phosphate and 6 parts sodium hydroxide	5	550	70
1 part dilithium cetyl phosphate and 6 parts lithium hydroxide	5	600	70

Example 2

This example illustrates die-forming of battery cans by the impact extrusion of slugs of a magnesium-base alloy similar in composition to that employed in the preceding example. The slugs were approximately one inch in diameter by one inch long. These were extruded into battery cans 1 inch inside diameter by 3 inches high, and having a wall thickness of approximately .05 inch. Prior to extrusion, the slug and die were sprayed with an aqueous disodium octyl phosphate lubricant composition. The slug was then heated in the die to the temperature of the extrusion and immediately extruded into the shape of a can, viz. a battery can. Both lubricants were stable at the temperature of extrusion and exhibited good separating power as evidenced by the ease of removal of the cans from the die. Corrosion tests on the extruded cans as taken from the die revealed no corrosion resulting from lubricant residue. Upon carrying out these tests for 7 days by alternately immersing the battery cans in salt water and then drying in air, the corrosion rates were found to average about 1.2 milligrams per square centimeter of surface per day. For battery cans extruded in a like manner and in the same equipment but using graphite as a lubricant the corrosion rate was considerably higher, i. e. 9.3 milligrams per square centimeter per day.

In the following table are listed the approximate compositions of the lubricants and the temperature of the impact extrusion in ° F.

Lubricant Composition	Temperature of Extrusion, ° F.
3 parts disodium octyl phosphate and 1 part monobasic sodium phosphate	450
1 part disodium octyl phosphate and 6 parts sodium hydroxide	500

That which is claimed is:

1. In a method of hot working the light metals, aluminum, magnesium, and alloys containing at least 70 percent by weight of one of such materials by the action of a tool, said method being carried out at a hot working temperature above about 450° F., the improvement which consists of controlling tool contamination and the effects thereof by applying to the surface of the light metal to be worked a substance selected from the class consisting of alkali and alkaline earth metal monoalkyl phosphates having from 8 to 20 carbon atoms in the molecule, said substance being stabilized against thermal decomposition by incorporation therein of an effective amount of a hydroxide selected from the class consisting of alkali and alkaline earth metal hydroxides.

2. In a method of hot working the light metals, aluminum, magnesium, and alloys containing at least 70 percent by weight of one of such materials by the action of a tool, said method being carried out at a hot working temperature above about 450° F., the improvement which consists of controlling tool contamination and the effects thereof by applying to the surface of the light metal to be worked a substance selected from the class consisting of alkali and alkaline earth metal monoalkyl phosphates having from 8 to 20 carbon atoms in the molecule, said substance being stabilized against thermal decomposition by the incorporation therein of an effective amount of trisodium phosphate.

3. A method according to claim 1 wherein the substance applied is disodium octyl phosphate stabilized against thermal decomposition with sodium hydroxide.

4. A method according to claim 1 wherein the substance applied is disodium cetyl phosphate stabilized against thermal decomposition with sodium hydroxide.

5. A method according to claim 1 wherein the substance applied is dilithium cetyl phosphate stabilized against thermal decomposition with lithium hydroxide.

6. A method according to claim 1 wherein the substance applied is calcium cetyl phosphate stabilized against thermal decomposition with calcium hydroxide.

References Cited in the file of this patent

UNITED STATES PATENTS

1,932,156	Ducamp	Oct. 24, 1933
2,108,641	Blodgett	Feb. 15, 1938
2,126,128	Montgomery	Aug. 9, 1938
2,285,854	Baxter et al.	June 9, 1942
2,310,175	Farrington	Feb. 2, 1943
2,369,596	Millen	Feb. 13, 1945
2,529,188	Rocchini	Nov. 7, 1950
2,529,189	Rocchini	Nov. 7, 1950
2,613,626	Whitney	Oct. 14, 1952