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[54] **HIGH TEMPERATURE LUBRICANT**

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[58] **Field of Search** .... 148/6.15 R; 117/127; 252/27, 79.2, 29, 30, 313, 12

[56] **References Cited**

UNITED STATES PATENTS

2,470,136 5/1949 Bramberry .... 148/6.15 R
2,939,809 6/1960 Jensen .... 148/6.15 R X

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[57] **ABSTRACT**

Adherent inorganic high temperature lubricant films on metal surfaces are formed by applying an inorganic lubricant-etchant composition to a metal surface, permitting the etchant portion of the composition to react with the metal surface and heating the metal to drive off volatile materials including the etchant leaving an adherent, dry, lubricant film. Specifically disclosed is the composition containing an inorganic acid such as phosphoric acid as the etchant material and molybdenum disulfide, lead oxide and graphite as the lubricant substances.

3 Claims, No Drawings

HIGH TEMPERATURE LUBRICANT

FIELD OF THE INVENTION

This invention relates to a method of forming an adherent lubricating film of inorganic materials on metals for high temperature operations.

This invention further relates to a method of forming a lubricant film and a composition of matter suitable for forming such a film which includes a substance which will etch the metal surface upon which the lubricant is being applied and suitable inorganic lubricants which will form an adherent film on the etched metal surface.

PRIOR ART

In the lubrication of moving parts at elevated temperature in high temperature environments encountered in jet engine operation or the like, it is essential that an adherent film of lubricating material be formed on the sliding surfaces of engaging metal parts. Inorganic lubricants such as molybdenum disulfide, lead oxides and the like, frequently are used as lubricants for sliding metal contact in high temperature applications. Adhesion of the dry lubricants to the metal surface has been less than satisfactory in prior art lubricant systems. A related copending application, Ser. No. 256,907, on an invention by Sullivan and Lipp which is owned by the assignee herein, and is entitled "High Temperature Lubricant" teaches a composition and method of usage of a high temperature lubricant which may be utilized to form an adherent lubricant film on certain types of metals, especially certain of the nickel based alloys widely used in the aerospace industry. Other metals, such as certain of the stainless steels, some nickel based alloys, titanium, zirconium, hafnium, columbium, tantalum, platinum and other metals used in high temperature and other specialty applications, as well as their alloys heretofor have been less than totally satisfactory in sliding contact applications due to the tendency of such metals to galling and seizure. The passive nature of the surface of many metals and alloys prevents adhesion thereto by prior art lubricants.

OBJECTS OF THE INVENTION

It is therefore an object of this invention to provide a composition of matter and a method of usage thereof which, when applied to a metallic surface and heated will form an adherent lubricative film suitable for use at elevated temperatures. The composition of matter acts both as a surface preparative agent and a precursor for formation of the adherent lubricant film.

It is a further object of this invention to provide a method for forming an adherent lubricative film on metals utilizing the composition of matter set forth herein and to provide a resultant novel adherent lubricative film on metal surfaces.

SUMMARY OF THE INVENTION

An adherent and relatively stable film of lubricating material may be formed on a metal surface by mixing together with suitable materials to form the film a suitable etching substance with one or more lubricant materials such as molybdenum disulfide, graphite, lead oxide, polymeric lubricants and the like. The mixture when applied to the metal surface cleans and etches the surface and forms a film of lubricant materials on the surface of the metal. When the surface has been suitably prepared for adhesion of the lubricant by the action of the etchant the metal part is heated and any volatile materials, preferably including the etchant, are driven off leaving behind an adherent lubricant film.

It can be theorized that the successful formation of lubricant films by use of the method and compositions of this invention are due to the unique formulation of the combined lubricant-etchant of this invention. When applied to a metal surface it solvates foreign substances on the surface, wets the surface intimately, and during curing and baking the surface activators become concentrated immediately prior to evaporation. The lubricating powder is then exposed to a receptive surface and an adherent film is formed.

Formation of an adherent lubricant film will increase the usability of many alloys for high and low temperature applications encountered in the aerospace and other industries.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Inorganic lubricants including lead monoxide, molybdenum disulfide and graphite when combined with an etchant phosphoric acid in an aqueous paste and permitted to air dry on a surface of metal to promote etching and then baked at elevated temperatures form adherent lubricant coatings. Organic lubricants may also be present if desired for lower temperature usage with certain types of metals. Examples could include various of the polymerized fluorocarbon compositions widely used as lubricants, the polyisobutylenes, heavy greases and other organic lubricants.

The composition ranges of the individual components of the high temperature lubricant-etchant which has been found best suited for usage on one of the cobalt-chromium-nickel alloys widely used in the aerospace industry for high temperature applications are set forth below in Table 1.

Table 1

A.	Dry Ingredients	Weight %
	Molybdenum Disulfide (MoS_2)	20 – 60
	Yellow Lead Oxide $(PbO)_2$	20 – 60
	Graphite	5 – 30
B.	Liquid Ingredients	Volume %
	Water	30 – 40
	Wetting Agent (Triton X-100)[1]	1 – 3
	Phosphoric Acid (86% H_3PO_4)	43 – 52
	Ethylene glycol monobutyl ether solvent	10 – 22

[1]A surfactant manufactured by the Rohm & Haas Co., Independence Mall West, Philadelphia, Pa., 19015. This surfactant is based upon alkylaryl polyether alcohols, sulfonates and/or sulfates, specific composition unknown.

The dry ingredients and liquid ingredients are mixed together in proportions so that the powder is completely wetted. For example, a very thick paste results when approximately one-half U.S. gallon of the liquid is mixed with eight pounds of the powder. The ratio of liquid to powder may be increased to result in a mixture having the desired viscosity for usage by brushing or spraying.

EXAMPLE 1

The dry ingredients set forth in Table 1 were combined with enough of the liquid ingredients of Table 1 to form a paste. This mixture was applied to specimen strips of an alloy containing 40% cobalt, 20% chronium, 15% nickel by weight as alloying components. These specimen strips were utilized as received, after sandblasting, and after wire brushing to compare the surface preparation effects. The paste was air dried on the alloy specimens for approximately 20 minutes to promote etching and then the specimens were baked at temperatures ranging from 500°F. to 900°F. to concentrate and finally drive off the liquid portion. Adherent dry films of the three inorganic lubricants remained on the alloy strips after one hour exposure at 500°F., at 700°F. and at 900°F. The dry films remaining on these surfaces exhibited excellent bond strength when burnished by wire brushing.

EXAMPLE 2

Alloy cable flat wraps were coated with a thick paste of the phosphoric acid-powdered lubricant mixture. One flat wrap was sanded and another sandblasted prior to lubricant application. An adherent film remained on both after baking at 700°F. and the resultant dry film lubricant which was formed during baking was tested and found satisfactory.

EXAMPLE 3

Several short lengths of the alloy material used in Example 1 were coated with the lubricant-etchant paste as in Example 1. These short sections of cable were passed through a cable wrap rotary swaging apparatus to evaluate coating flexibility. In both cases the coating remained firmly attached to the substrate after eight to ten passes through the swaging apparatus.

EXAMPLE 4

A thin paste of etchant-lubricant material was formed by blending 1 U.S. gallon of the liquid ingredients set forth below with 8 pounds of the powder ingredients set forth below:

Liquid Ingredients	Volume %
Distilled Water	34.0 ± 2
Wetting agent (Triton X-100)	2.0 ± 2
Phosphoric acid	48.0 ± 2
Ethylene glycol monobutyl ether	16.0 ± 2
	100.0

Powder Ingredients	Weight %
Molybdenum disulfide (MoS_2)	40.0 ± 2
Yellow lead oxide $(PbO)_2$	40.0 ± 2
Graphite	20.0 ± 2
	100.0

The thin paste was applied by brushing to a titanium metal sample. The paste was air dried for approximately 20 minutes to promote etching of the metal surface and then baked at 500°F for one hour. An adherent dry film of molybdenum disulfide, lead oxide and graphite remained on the sample. Vigorous wire brushing of the surface did not remove the lubricant film.

EXAMPLE 5

A thin, sprayable lubricant-etchant mixture was formed by blending 1 U.S. gallon of the liquid ingredients listed in Example 3 with 4 pounds of the dry ingredients listed in Example 3. Samples of the metals listed below were cleaned by wire brushing the surface and coated with the lubricant-etchant mixture by spraying upon the exposed metal surface. The samples were then cured at room temperature for 30 minutes and baked at 700°F. for one hour to drive off volatiles.

Sample	Metal
A	Titanium Base Alloy 6% Aluminum 4% Vanadium
B	Stainless Steel (corrosion resistant) 21% Chromium 6% Nickel 9% Manganese
C	Cobalt Base Alloy "Haynes 25"
D	Nickel Base Alloy "Inconel 625"
E	Martensitic Stainless Steel 16–18% Chromium 1% Carbon
F	Age Hardenable Stainless Steel 12% Chromium 9% Nickel

All samples (A–F) demonstrated an adherent lubricant coating after the baking step. Vigorous wire brushing smoothed the surface, but did not remove the lubricant film.

Our etchant-lubricant composition of matter may utilize any of the known etchant substances for the particular metal upon which the adherent lubricant film is desired and which is compatible with the necessary lubricant film forming substances. For example, various of the aqueous acids may be used. For our preferred etchant-lubricant composition we use aqueous phosphoric acid with lead oxide, graphite or molybdenum disulfide or mixtures of these inorganic lubricants. For best results it is necessary to at least have the inorganic ingredients thoroughly wetted with the aqueous phosphoric acid to form a thick paste. For situations in which brushing and/or spraying of the etchant-lubricant composition is to be employed a thinner composition having a higher aqueous phosphoric acid content is used. Other solvents may be used.

While the invention herein has been described with reference to certain preferred embodiments, notably using the compositions containing aqueous phosphoric acid as the etchant substance, it is to be understood that equivalent etchant substances may be utilized within the scope of this invention. Similarly, lubricants other than the exemplory lubricants noted above may advantageously be employed in the practice of this invention. The foregoing disclosure will so fully reveal the gist of the present invention that others can, by applying knowledge readily available to those skilled in the art, modify the invention without, however, departing from the inventive concept. Such modifications are meant to be comprehended within the scope of the appended claims.

We claim:

1. A lubricant-etchant composition consisting essentially of a liquid portion containing:

Component	Volume %
Phosphoric acid (86% H_3PO_4)	43 – 52
Water	30 – 40
Wetting agent	1 – 3
Organic solvent	10 – 22

combined with a film-forming dry powder portion containing:

Component	% by Weight
Molybdenum disulfide (MoS_4)	20 – 60
Yellow lead oxide $(PbO)_2$	20 – 60
Graphite	5 – 30

said portions being combined in proportions so that said powder portion is at least completely wetted by said liquid portion.

2. The composition of claim 1 containing at least one-half U.S. gallon of said liquid portion for each 8 pounds of said powder portion.

3. A lubricant-etchant composition consisting essentially of a liquid portion consisting essentially of:

Component	% by Volume
Water	34.0 ± 2
Wetting agent	2.0 ± 2
Phosphoric acid (86% H_3PO_4)	48.0 ± 2
Solvent	16.0 ± 2

combined with a dry material consisting essentially of:

Component	% by Weight
Molybdenum disulfide	40.0 ± 2
Yellow lead oxide	40.0 ± 2
Graphite	20.0 ± 2

said liquid portion being present in the range of ½ to 2 U.S. gallons for each 8 pounds of said dry material.

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