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(54) **Carrier-free metalworking lubricant and method of making and using same.**

(57) In one aspect the invention is a carrier-free pulverulent metalworking lubricant containing a resin having a highly polar functional group, and in another aspect is a method of the invention of forging a workpiece in a die which includes the step of applying to at least one of the die and the workpiece a coating of an effective amount of the carrier-free pulverulent lubricant composition. The use of the composition and method of the invention significantly reduces smoke and oily waste generation in hot forging operations by eliminating the use of oils or volatile organic compounds as carriers, while providing acceptable performance, cleanability, and sprayability.

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This invention relates to the field of metalworking lubricants in general and, in one particular respect, to forging lubricants. More particularly, it relates in one aspect to a new forging lubricant composition and a method of using that composition in the hot forging of metal workpieces. Metal parts of a multitude of sizes and shapes are manufactured by various types of forging operations, and these parts are formed from stock composed of a great many metals and metal alloys. A great many parts are forged from such metals and metal alloys as, for example, steel, aluminum, titanium, and high nickel alloys, to name but a few.

The conditions under which metal parts are forged, of course, are widely variable, depending upon not only the nature of the metal, but upon the size and complexity of configuration of the desired part. Small, thin, simply shaped parts may obviously be forged from a relatively flowable metal such as aluminum under much less rigorous conditions than are required to forge large more complex shaped parts from a metal such as steel.

Each set of forging conditions requires a specialized lubricant, and there is therefore a multitude of aqueous-based, oil-based and organic solvent-based lubricants currently in use in various forging operations. Many such lubricant systems, particularly those used under the most demanding forging conditions, by their nature require the user to make compromises in order to achieve the desired functional characteristics while avoiding as much as possible any safety, occupational health or environmental hazards involved in their use. Moreover, in some instances, more restrictive health and environmental guidelines are now in force which may make the use of certain lubricant systems either extremely expensive or simply unworkable. It is to these and related concerns which the present invention is directed.

In a typical high performance forging operation, such as one which might be devoted to the manufacture of large, complex parts from aluminum alloy stock, an effective lubricant is one which ordinarily contains a variety of lubricity agents in a carrier comprising mineral oil and/or volatile organic solvents. The dies used in such forging operations are maintained at high temperatures, in the range of 350°F to 825°F, in order to permit proper metal flow during the forging operation.

The forging lubricant is typically applied to the die and the workpiece by spraying, and, on account of the temperatures involved, the mineral oil and volatile organic compounds immediately flash off, leaving only a relatively small amount of residue which actually functions as the lubricant. As anyone who has observed such a forge operation well knows, the flashing off of the mineral oil and volatile organic compounds creates a significant amount of open flames, and the spray wand by which the lubricant is applied takes on the appearance of a flame thrower. Moreover, a large amount of smoke is typically generated when the mineral oil and volatile organic compounds flash off, since, at the same time, a rather significant portion of the lubricity agents may burn off as well. In this context, it is well known that any improvements in the performance of the forge lubricant which are achieved by reformulation frequently come at the cost of significantly higher smoke generation.

Similar difficulties are inherent when oil-based paste type lubricants are utilized. While the paste lubricants contain little or no volatile organic compounds, their oil carriers partially or completely burn at typical forging temperatures, resulting in significant heavy smoke generation.

The hazards, expense and environmental problems associated with such forging operations are of great proportion and are quickly becoming even more so.

In a state such as California, where environmental protection statues and regulations impose rigid standards on industrial operations, and in other states which have similar environmental protection schemes, the smoke generated by a large forge operation creates tremendous difficulties.

Since environmental agencies frequently monitor smoke emissions by aerial surveillance, there is close attention paid to reducing the smoke generated in the forging operation. Unfortunately, this often limits the efforts made to vent the smoke from the buildings in which the forge operation is housed. The result of this is a significant degradation of the air quality within the buildings.

An important economic consideration is that in California, for example, a tax may be levied upon each gallon of volatile organic compounds emitted into the air. More importantly, as air quality standards are progressively raised, there will soon come a time when a forge operation will simply be prohibited from emitting large amounts of smoke. The choice then will be to find an alternative lubricant which produces significantly reduced amounts of smoke or to cease operations entirely.

Similar problems exist with respect to the use of oil or solvent-based lubricants in smaller scale forge and other metalworking operations, since waste lubricant materials of this type are considered an environmental hazard. Disposal is therefore tightly controlled and increasingly expensive.

Other related concerns create a strong demand for alternative metalworking lubricants.

As described above, open flame is generated when conventional mineral oil and volatile organic compound-based lubricants are applied to a heated die. One must therefore have available fire prevention and fire control equipment, such as fire extinguishers and sprinkler systems, in the immediate area of the forge operation. Indeed, fire extinguishers see regular use in many forge operations, and the cost of their maintenance is significant. In general, fire prevention, fire control and fire detection systems of all types are regular and significant capital and maintenance cost items for hot forge operations.

A related problem associated with the use of conventional volatile organic compound-based lubricants is the need for special storage facilities on account of their high flammability. This too imposes a significant cost associated with the use of conventional lubricants.

Transportation of these flammable lubricants in special containers and special vehicles is yet another source of additional cost, hazard, and inconvenience associated with their use.

A still further disadvantage of conventional lubricant systems which results from the flashing off of oil and solvent carriers is that the smoke generated forms tar-like deposits on machinery, finished parts, floors, windows, and nearly everything else housed in the same building with the forge operation. Quite apart from the aesthetic undesirability of such deposits, there are economic and health concerns as well. Many large forge operations maintain permanent steam-cleaning facilities at a significant cost.

Various types of dry lubricants and methods for applying them to metal surfaces have been proposed for use in diverse environments, but none has been widely adopted on account of certain inherent disadvantages in either the lubricant itself or the method of its application.

For example, in titanium forging operations, it has been proposed to utilize a powdered lubricant composed of glass and ceramic components, with the optional use of steel shot, in a process in which the lubricant is imbedded in the forge tool surface by a high pressure spray. This process is described in terms of sandblasting the lubricant onto the tool surface, and is intended to effect a cold working and smoothing of the tool surface. Of course, such a high pressure spray process involves the use of rather expensive spray equipment, and it also presents the risk of worker injury due to misdirected spray.

Others have proposed to spray dry reactant materials onto hot metal surfaces in order to form a reaction product lubricant in situ. Still others have proposed various combinations of dry lubricant components for use in a wide range of applications. Many of these lubricant compositions, however, have drawbacks, as well.

After forging, whether with a conventional or dry lubricant, aluminum parts are subjected to a caustic etch for the purpose of removing lubricant residues. In a preferred procedure which is well known in the art, the caustic etch may be used in combination with an acid wash. In many aluminum forge operations, the acid wash advantageously precedes the caustic etch.

As is well known in the art, the conditions of these wash and etch procedures are quite harsh. Typically, the caustic etch bath is 5% to 15% by weight alkali metal hydroxide in water. Typical acid baths are similarly strong, often containing a high concentration of nitric acid. In forge operations using conventional solvent or oil based lubricants, the wash and etch procedure works quite well to remove essentially all lubricant residues from the forged parts.

Notwithstanding the harsh conditions of the wash and etch, however, it has been found that residues of powdered lubricants may still adhere to the parts with such tenacity that even subjecting the parts to physical removal procedures, such as brushing and scraping, after the etch will not adequately clean them.

It has also been found, in working with multi-component powdered lubricants, that obtaining a consistent spray pattern using conventional powder coating equipment is extremely difficult. Overspray, underspray, puffing, and sputtering have been found to be serious drawbacks, both from the standpoint of obtaining a functional lubricant coating on the workpiece and from the standpoint of efficient use of powder lubricant material. Overall, the spray process has heretofore been found too erratic to be acceptable commercially. Moreover, it has been unexpectedly found that the spray was particularly unpredictable when utilizing powder coating equipment which, as is quite common, utilizes a fluidized bed as a reservoir from which the powder was sprayed. Even utilizing powder coating equipment which has a gravity-fed reservoir has typically provided only a marginal improvement in consistency.

While the particular problems encountered in an aluminum forge operation have been described in detail, many of the same and other related concerns exist in other metal working environments. These include not only other hot forge operations, such as the manufacture of forged steel and titanium parts, but also a wide variety of other metalworking and metal forming operations. Examples include extrusion, drawing, stamping, and other hot and cold forming operations, many of which employ lubricants in aqueous or solvent based carriers. Thus, many of the same technical and economic benefits could be realized in such operations by adopting an improved dry lubricant composition.

It is therefore a principal object of the present invention to provide a forge lubricant and a method of its use which significantly reduce the amount of smoke and oily waste generated during the forging operation.

A related object is to eliminate the organic carrier materials which are essential parts of conventional high performance forging lubricants.

Thus, a general object of the present invention to provide a lubricant which eliminates many health, environmental and safety drawbacks of conventional lubricants having mineral oil and volatile organic compounds as carriers.

Another more particular object is to eliminate the need for special transportation and storage facilities which are required for conventional lubricants.

A further important object of the present invention is to provide a powdered lubricant composition which may be applied to a workpiece and/or die in a substantially uniform coating by the use of conventional powder coating equipment.

A related object is to provide a method of manufacturing a powdered lubricant composition which may be more readily applied to a workpiece and/or die in a substantially uniform coating by the use of conventional powder coating equipment.

Yet another important object is to provide a high performance dry lubricant which does not form residues which resist removal by conventional cleaning procedures.

Other objects and advantages of the present invention will be apparent to those skilled in the art from the following description of the invention and the appended claims.

In its most basic form, the composition of the present invention is a carrier-free pulverulent metalworking lubricant, i.e., one which is entirely free of the oils and volatile organic compounds commonly employed as carriers for forge lubricant compositions. Similarly, in one form, the method of the invention is a method of forming a workpiece in a metal-forming apparatus which includes the steps of applying to at least one of the metal-forming apparatus and the workpiece a coating of an effective amount of a carrier-free pulverulent lubricant composition, and forming the workpiece in the apparatus.

The carrier-free pulverulent metalworking lubricant of the invention may, in general, include any material which will provide lubricating properties at the temperatures typically encountered in a forging process and which can be put into a physical form which permits it to be applied to the die and/or the workpiece by conventional powder-coating equipment.

In accordance with the present invention, the need to incorporate a mineral oil and/or a volatile organic compound-based carrier is completely eliminated, with the result that the smoke generated by conventional lubricants is significantly reduced.

In accordance with a first aspect of the invention there is provided a carrier-free pulverulent metalworking lubricant composition including at least one resin having a highly polar functional group, which may be solubilized under strong acid or basic conditions, and which is a solid at room temperature.

In a second aspect, the invention is a carrier-free metalworking lubricant composition having a substantially uniform particle size.

Yet a third aspect of the invention is a method of forming a carrier-free pulverulent metalworking lubricant composition, which includes the steps of forming a dry admixture of lubricant components, heating the admixture to a temperature sufficient to melt at least one component of the admixture, agitating the heated admixture to form a substantially homogenous melt, cooling the substantially homogenous melt to form a substantially solidified mass, and comminuting the substantially solidified mass to a desired particle size. In an alternative aspect, the invention is a method of forming a homogeneous melt of lubricant components and then spray -- drying the melt to a desired particle size.

A still further aspect of the invention is a method of forging a workpiece in a die which includes the steps of applying to at least one of said die and said workpiece a coating of an effective amount of a carrier-free pulverulent lubricant composition having at least one resin having a highly polar functional group, which may be solubilized under strong acid or basic conditions, and which is a solid at room temperature, and forging the workpiece in the die.

The advantages inherent in the composition and method of the invention are numerous.

The elimination of much of the smoke previously generated by the flashing off of a mineral oil and volatile organic compound carrier permits a forging operation to continue in business in full compliance with environmental statutes and regulations. Moreover, the business may continue without the economic burden of tax payments based on the emission of volatile organic compounds. In many instances, the use of the composition and method of the present invention will permit a forge operation to continue in existence under a stringently regulated environmental scheme which would otherwise cause it to be shut down entirely.

Other economic advantages of the composition and method of the invention are of equally great importance.

The reduction in weight and volume which occurs when the carriers of conventional lubricants are eliminated leads to savings in the cost of shipment and storage. Even further savings are realized in transportation and storage costs because the carrier-free composition of the invention is neither flammable nor hazardous, and it can be shipped and stored in the same manner as any other nonhazardous arterial. Moreover, packaging costs are significantly reduced, since a five-gallon plastic pail of the carrier-free pulverulent metalworking lubricant of the present invention will be the functional replacement for a fifty-five gallon steel drum of a conventional lubricant.

In the forge operation itself, the composition and method of the invention result in significant reductions in the cost of installing and maintaining fire prevention and fire control systems, and in general permit the maintenance of a much safer environment for personnel at a much lower cost.

Still further savings resulting from the use of the composition and method of the invention may be realized in reduced premiums for fire, workmen's compensation, and liability insurance.

The elimination of the carrier material significantly reduces raw material cost, since, on a weight and volume basis, the carrier in conventional lubricants accounts for well over 90% of the composition.

The need to maintain expensive and space-consuming cleaning facilities for plant and finished parts is also reduced by the use of the composition and method of the invention, since significantly less combustion residues will be produced in the absence of the flashing off of mineral oil and volatile organic compound carriers.

Additional functional advantages are also achieved by the present invention.

In connection with the first aspect of the invention the incorporation of a resin which is solubilized in an alkali and/or acid bath provides the advantage of a cleanable forged part, even with the use of a dry powder lubricant.

Further, in the second aspect of the invention maintaining the particle size of the lubricant powder within a narrow range permits a uniform coating of lubricant powder to be applied with conventional powder coating equipment, even when utilizing equipment which employs a fluidized bed as a powder reservoir. And, controlling the particle size of the lubricant powder by its novel method of manufacture not only provides spray consistency, but improves lubricant properties and cleanability as well.

As stated above, the composition of the present invention, in its most basic form, is a carrier-free pulverulent metalworking lubricant. It may include any material which will provide lubricating properties at the temperatures typically encountered in a metal-forming process and which can be put into a physical form which permits it to be applied to the die and/or the workpiece by conventional powder-coating equipment.

Many materials which will perform the function of lubricating the die and maintaining a physical separation between the die and the workpiece are well known, and, of these materials, many are in the physical form necessary to the practice of the present invention; namely, a solid at room temperature. It is not necessary that the materials employed in the composition of the invention remain either solid or pulverulent at the temperatures typically encountered during a hot forging operation, e.g., about 600°F up to 1000°F for aluminum, and about 1500°F up to 2500°F for steel or titanium. It is enough that they may be made to exist in a particulate form at ambient temperatures. In that form, they can be applied by conventional powder-coating equipment, even though they may partially or completely melt or burn when in contact with the heated die or workpiece. Indeed, it is preferred that at least one component of the carrier-free pulverulent metalworking lubricant becomes sticky upon being heated so as to assist in adhering the dry metalworking lubricant composition to the workpiece and die surfaces.

Typical materials which are capable of maintaining a physical barrier between the die and the workpiece and which function as solid lubricants are contemplated for use in the composition of the first and second aspects of the invention. They include, by way of example only, metal soaps, fatty acids, graphite, ceramics, high melting polymer resins, natural and synthetic waxes, gilsonite, glasses, and mixtures of these materials.

Useful metal soaps are those which are solids at room temperature, including many sulfonates, naphthenates, and carboxylates. Of these, fatty acid soaps such as zinc stearate and sodium stearate are preferred on account of their known properties, their ready availability and low cost. However, other metal soaps known for their lubricant properties, including, by way of example only, tin, copper, titanium, lithium, calcium, and other alkali and alkaline earth metal soaps of fatty acids, may be advantageously included.

Fatty acids themselves which are solids at room temperature may also be included, and their relatively low cost, ready availability, and their contribution to the overall lubricity of the composition makes them attractive for such use. One example is stearic acid, which is advantageously used since it has good lubricating properties, is nontoxic, inexpensive, and readily available.

Materials such as graphite and certain ceramic materials such as boron nitride are useful for maintaining a physical separation between the die and the workpiece. While the precise mechanism of the physical separation is not known, this characteristic is believed to be attributable to the relatively planar crystalline structure of these materials.

Useful high melting polymer resins include, by way of example, poly(tetrafluoroethylene) (PTFE), high density polyethylene (HDPE), poly(vinylchloride) (PVC), polyesters, polyethylene glycols, polyacrylates, polymethacrylates and polyamides. Indeed, almost any thermoplastic material may be used. In the first aspect of the invention resins selected from the group polyethylene glycol resins, polyester resins, polyacrylate resins, polymethacrylate resins, polyamide resins and mixtures thereof are suitable as resins having a highly polar functional group whereby said resin may be solubilized under strong acid or basic conditions, said resin being a solid at room temperature.

Of the natural and synthetic waxes which may be advantageously employed, polyethylene waxes of relatively high molecular weights are in general preferred on account of the lubricity which they impart.

Glass materials useful in the present invention are preferably the low melting glasses, including alumina, alumina/silica, silica and borax. Optionally, these glass materials may be used in chopped fiber form.

In one basic form of the method of the invention, a coating of an effective amount of a carrier-free pulverulent lubricant composition is applied to at least one of the die and the workpiece, and the workpiece is then formed into the desired finished part. In general, the application of the lubricant in accordance with the invention may be accomplished by any conventional powder-coating equipment.

In one alternative method falling within the scope of the present invention, the carrier-free pulverulent metalworking lubricant is applied by means of an electrostatic spray apparatus, inasmuch as there is little loss of material on account of the electrostatic attraction of the particles to the die and/or workpiece, and, since electrostatic spray is known to produce a uniform coating on even complex-shaped parts.

In high temperature environments, such as aluminum, steel, and titanium forging operations, maintaining sufficient charge on the lubricant particles is quite difficult when the powder spray is directed to the die or workpiece in the vicinity of the press, and the electrostatic powder coating apparatus provides little advantage over non-electrostatic equipment. However, an electrostatic apparatus provides a significant benefit for pre-coating aluminum, steel, or titanium workpieces at ambient temperature, after which the workpiece is heated in an oven prior to insertion into the press. Similarly, in cold forming operations, such as stamping and the like, which are carried out at much lower temperatures, the advantages of electrostatic spray are maintained.

The lubricant of the invention may be applied to a heated or heating die in a manner analogous to the application of conventional lubricants. Alternatively, the lubricant composition may be sprayed onto a cold unforged workpiece, after which the workpiece is heated to achieve a partial melt of the composition and subsequently placed into a heated die for forging. In cold-forming operations, the workpiece may be spray-coated, and the conventional step of heating the workpiece to flash off or evaporate an aqueous, solvent or oil carrier may be eliminated.

It has been found that on account of their very powdery, even dust-like, nature, such materials as graphite and amorphous boron nitride are, unless they have an electrostatic charge, less easily retained on the surfaces of the die and workpiece than are some of the other materials enumerated above. Drafts or currents of air may therefore undesirably remove the pulverulent forging lubricant from the die and/or the workpiece prior to the forging operation. Thus, when including one or more of these materials in a lubricant of the second aspect of the invention formed as a dry admixture which is to be delivered by a non-electrostatic powder coating apparatus, it is preferred to also include at least one component having adhesive properties at typical forging temperatures, such as a glass, gilsonite, or high melting polymer resin for the purpose of retaining the lubricant on the die and the workpiece.

Some examples of the lubricant composition and metalworking method of the invention are set forth below.

Examples 1 and 2

The following compositions were used to forge a box channel with high walls, approximately 0.125 inches thick, in a wrap die from aluminum alloy stock. The press was of the hydraulic type, with the workpiece temperature being 700°F and the die temperature 375°F:

Example 1

Component	Weight%
gilsonite	5
zinc stearate	34
sodium stearate	10
graphite	17
polyethylene	34
	100

Example 2

Component	Weight%
gilsonite	5
zinc stearate	34
sodium stearate	10
graphite	17
amide wax	34
	100

Only seven parts were forged; thus, optimization of spray techniques could not be achieved. However, examination of the forged parts showed excellent metal movement, with a complete die fill of the walls of the channel. There was excellent downsize of the critical part dimension, and the parts released easily from the die, with no sticking. The dies had some tendency to stick together; however, this is normally experienced with this configuration of parts. Smoke levels were noticeably lower than those produced when a conventional solvent, oil and graphite lubricant was used. Based on this rather limited trial, the composition of Example 1 outperformed the composition of Example 2 in each of the observed respects, though both were effective as forging lubricants.

Example 3

In a comparative trial, the composition of Example 1 was evaluated using a conventional solvent-based zinc stearate forging lubricant as a standard. The press was of the mechanical type, with the workpiece temperature being 700°F and the die temperature 400°F.

Forty parts were forged from each composition. Examination of the forged parts showed excellent metal movement with no drag. There was excellent downsize of the critical part dimension. The parts released easily from the die, with no sticking, and there was no buildup of lubricant residue on the parts. Smoke levels when using the composition of Example 1 were significantly lower than those produced during the trials reported in Examples 1 and 2.

Examples 4 and 5

Each of the following compositions was evaluated under the same conditions as those of Example 3, and each was found to perform satisfactorily with significantly lower smoke generation than conventional solvent-based lubricants.

Example 4

Component	Weight%
graphite	33.0
zinc stearate	34.5
gilsonite	10.9
polyethylene wax	21.1
	99.5

Example 5

Component	Weight%
graphite	23.8
sodium stearate	33.4
gilsonite	23.8
polyethylene wax	9.5
zinc stearate	9.5
	100.0

The composition of Example 5 was also evaluated in the high-temperature environments of steel and titanium forging, and it was found to perform satisfactorily in the forging of both metals.

Examples 6-8

The following carrier-free pulverulent lubricant compositions have also been found useful for the forging of aluminum and aluminum alloy workpieces:

Example 6

Component	Weight %
graphite	23.8
gilsonite	23.0
sodium stearate	33.4
polyamide	9.5
zinc stearate	9.5
	100.0

Example 7

Component	Weight %
graphite	23.8
gilsonite	23.8
sodium stearate	33.4
polyacrylate	9.5
dibutyl tin carboxylate	9.5
	100.0

Example 8

Component	Weight %
graphite	75
gilsonite	25
	100

Example 9

Component	Weight%
graphite	50
gilsonite	25
zinc stearate	15
poly(tetrafluoroethylene)	10
	100

Examples 10-15

Other carrier-free pulverulent lubricant compositions have been found useful for high temperature forging of titanium and steel, and they include the following:

Example 10

Component	Weight%
graphite	20.0
gilsonite	20.0
sodium stearate	30.0
stearic acid	20.0
polyethylene wax	10.0
	100.0

Example 11

Component	Weight%
graphite	15.0
gilsonite	20.0
sodium stearate	30.0
stearic acid	20.0
polyethylene wax	10.0
boron nitride	5.0
	100.0

Example 12

Component	Weight%
graphite	40.0
gilsonite	20.0
sodium stearate	20.0
stearic acid	20.0
	100.0

Example 13

Component	Weight%
alumina/silica glass	40
graphite	60
	100

Example 14

Component	Weight%
boron nitride	25
borax	75
	100

Example 15

Component	Weight%
graphite	35
borax	65
	100

It is possible to achieve a limited improvement in cleanability of aluminum and aluminum alloy parts by reducing or eliminating gilsonite from the composition, since it tends to contribute to the formation of tar-like residues on the forged parts. But eliminating this component improves cleanability only marginally, and at the price of reduced performance, since the gilsonite provides good lubricity, while at the same time its tacky character at forging temperatures tends to help a lubricant formed as a dry admixture to adhere to the workpiece and the die.

What has been discovered to be extremely effective, however, is to replace the gilsonite with a component which unexpectedly provides the combination of the same desirable performance attributes contributed by gilsonite and other similar tacky substances, together with a level of cleanability which is the equal of a conventional solvent and/or oil based forging lubricant.

Specifically, the use of a resin component having certain physical and chemical attributes can provide the combination of good performance and far superior cleanability required for successful industrial use.

In general, any resin which has good lubricity properties at forging temperatures, is a solid at ambient temperatures, and contains a highly polar functional group which enables the resin to be solubilized in the caustic etch and/or acid bath will provide this combination of properties. In general, halogenated resins are preferably avoided in hot forging operations on account of their tendency to form hazardous combustion products.

Particular resins which have been found useful in the practice of the invention include the polyethylene glycol resins, polyester resins having terminal hydroxyl or carboxyl functional groups, polyacrylate, polymethacrylate, and polyamide resins and mixtures of these resins. Presently preferred are the polyester and polyethylene glycol resins on account of their good lubricity properties, superior cleanability, and lack of objectionable burn characteristics. Some examples of such resins are the polyethyleneglycol resins sold under the tradename Pluracol by BASF, such as E4000 and E8000, the hydroxyl functional polyester resins sold by Cargill, such as 30-3016, and the carboxyl functional polyester resins sold by Cargill, such as 30-3065. These materials are generally dry solids at room or ambient temperature, so that they are readily applied to the workpiece and die by conventional powder coating equipment.

These resins provide the desired combination of lubricity and cleanability characteristics when utilized in the composition of the invention in amounts of from about 5% to about 50% by weight of the composition, with a preferred range of from about 10% to about 30% by weight of the composition. Most preferably, the amount of resin is maintained as low as possible while still providing the desired performance characteristics, since these resins tend to be more expensive on a weight unit basis than many of the other components of the composition. While, in general, an observable improvement in cleanability is achieved when at least about 5% by weight of the composition is a high-melting resin having a highly polar functional group, the upper concentration limit is more an economic than a functional one.

It is important to note in this regard (and with respect to the determination of the optimum concentration of any of the other components of the composition) that small variations in the amount of resin used do not manifest themselves in readily observable variations in performance or cleanability. Indeed, the evaluation of performance and cleanability is highly subjective and not susceptible to quantification to any meaningful degree. Thus, the weight percentage of resin or any other component in the lubricant composition is not narrowly critical to the practice of the present invention and may vary considerably without an adverse effect on performance.

Example 16

A lubricant powder composition was formulated in accordance with the invention as follows:

Component	Weight%
graphite	15
stearic acid	15
zinc stearate	30
sodium stearate	10
polyethylene glycol	20
carboxyl functional polyester	10
	100

The lubricant so formulated was successfully utilized in a high performance aluminum forge operation for the purpose of forging a number of aircraft parts. The lubricant of Example 16 was further found to perform successfully in typical steel (engine valves) and titanium (turbine blades) forging operations.

Example 17

The forging of a first group of aluminum parts using the composition of Example 16 was carried out together with the forging of a second group of aluminum parts using the composition of Example 5, and a series of three comparative cleaning tests was conducted. The cleaning procedures and the results obtained are summarized below:

Cleaning Tests Detail

Test A - Process (Standard Etch)

Step 1 -	Caustic soda, 8 oz/gal, 175-180° F, 120 sec
Step 2 -	Rinse, cold
Step 3 -	Rinse, cold
Step 4 -	Desmut, nitric acid 25%, 60 sec
Step 5 -	Rinse, cold
Step 6 -	Rinse, cold
Step 7 -	Rinse, hot

Results:

Removing Example 5 lubricant: poor cleaning
Removing Example 16 lubricant: marginally acceptable cleaning

Test B - Process:

Step 1 -	24% sulfuric acid, 6% nitric acid, 180 F, 10 min.
Step 2 -	Rinse, cold
Step 3 -	Rinse, cold
Step 4 -	Caustic soda, 8 oz/gal, 175-180 F, 120 sec
Step 5 -	Rinse, cold
Step 6 -	Rinse, cold
Step 7 -	Desmut, nitric acid 25%, 60 sec
Step 8 -	Rinse, cold
Step 9 -	Rinse, cold
Step 10 -	Rinse, hot

Results:

Removing Example 16 lubricant: essentially clean; equivalent to cleaning liquid lubricant with standard etch process.

Test C - Process:

Step 1 -	Nitric acid 50%, 120 sec
Step 2 -	Rinse, cold
Step 3 -	Caustic soda, 8 oz/gal, 140 F, 30-180 sec
Step 4 -	Rinse, cold
Step 5 -	Desmut, nitric acid 50%, 120 sec
Step 6 -	Rinse, cold
Step 7 -	Rinse, hot

Results:

Removing Example 16 lubricant: essentially clean; equivalent to cleaning liquid lubricant with the same etch process.

Following a number of such comparative cleaning tests, a still further advantage of the lubricant of Example 16 over a conventional zinc-containing lubricant was discovered; namely, a 95% reduction in the amount of zinc present in the etch solutions. Reduction of the metal content of industrial wastes is, of course, a valuable environmental and economic benefit.

Example 18-21

Lubricant powder compositions also formulated in accordance with the present invention are:

Example 18

Component	Weight%
graphite	15
stearic acid	20
dibutyl tin carboxylate	20
sodium stearate	25
polyamide	10
hydroxyl functional polyester	10
	100

Example 20

Component	Weight%
graphite	40
hydroxyl functional polyester	20
zinc stearate	20
stearic acid	20
	100

Example 19

Component	Weight%
graphite	15
carboxyl functional polyester	20
sodium stearate	20
stearic acid	20
polyethylene glycol	10
boron nitride	5
	100

Example 21

Component	Weight%
alumina/silica glass	40
graphite	55
polyethylene glycol	5
	100

In another aspect of the invention, maintaining a narrow particle size range provides greatly improved spray efficiency and consistency, such that a substantially uniform coating of powder lubricant could be applied to the workpiece. The best results are obtained, of course, when the particles of lubricant powder are of essentially uniform size. For the purpose of the invention, "substantially uniform size" means that there are relatively few or no particles more than 15% larger or smaller than the average. Most preferably, there are relatively few or no particles more than 10% larger or smaller than the average.

In general, a particle size of from 10 microns to 300 microns produces acceptable results, though a size of 40 microns or greater is preferred to minimize the extent to which lubricant particles remain airborne in the form of dust. There are two objectives in minimizing dusting; namely, to provide an environmentally safer environment for the worker, and to reduce lubricant material loss by increasing the efficiency and accuracy of the powder spray.

The upper limit on particle size is essentially a function of the capability of the spray equipment and of the ability of the particles to adhere to the surface of the workpiece in a substantially uniform coating. Most preferably, a particle size of about 50 microns to about 100 microns is utilized, since commercially available powder coating equipment functions best in this range.

One manner of controlling both particle size and the range of particle sizes is to utilize as starting materials lubricant components which have been ground or sieved to a substantially uniform size. The components may then be readily admixed by conventional dry mixing techniques, such as by use of a ribbon blender, a tumbling blender, or a twin shell blender. An obvious drawback of this procedure is the time, effort, and expense involved in either purchasing or processing each of the components to the desired size and size range. Moreover, the dry blending process itself causes the particles to abrade one another, thereby creating a multitude of small particles, and therefore once again broadening the particle size range.

It has been discovered that one may overcome the shortcomings of the method of controlling particle size just described, and may also achieve other significant improvements, by forming the lubricant powder in an entirely different manner. Specifically, it has been discovered that a high performance powdered lubricant having a well-controlled particle size range may be formed by the following method: First, the lubricant components, which may be in any conveniently available comminuted form, such as powders, flakes, small pellets, and the like, essentially regardless of their particle size, are admixed in the desired proportions to form a dry lubricant premix. The dry lubricant premix is then heated with agitation to form an essentially homogenous melt. A temperature of from about 100° C to about 200° C is usually sufficient to provide a consistency which permits melt mixing. The homogenous melt is then cooled to form a solid mass. The solid mass is then ground at low temperature to the desired particle size by conventional cold-grinding techniques. Equipment capable of performing this operation is commercially available. In one such process, the homogenous lubricant melt is discharged onto a rotating metal plate which is chilled to about 40° F (10° C) to solidify the mass in sheet form. The sheets are then broken into shards which are in the range of 1 to 3 centimeters across. The shards are then, in turn, hammer-milled to the desired particle size in an air-conditioned room. Other similar processes solidify the melt into ribbon form, after which it is broken into chips and milled to the desired particle size under suitable conditions.

Typically, the milling equipment is rather massive, and is constructed of steel or other metal. It therefore becomes conditioned to the ambient room temperature of on the order of about 60°F to 70°F, and provides a highly efficient heat sink for the lubricant composition as it is milled. If necessary, the apparatus can be further chilled by, for example, circulating liquid nitrogen through a network of internal channels provided for that purpose. This temperature control permits optimization of the process in terms of controlling particle size, since many of the lubricant components would become tacky or semi-solid upon being subjected to the heat generated in conventional grinding or milling processes, but remain dry solids at lower temperatures.

Alternatively, the lubricant of the invention may be produced by forming a homogenous melt of the components as described above, and then spray-drying the melt in a conventional manner to the desired particle size.

Not only do these processes of producing the lubricant of the invention greatly facilitate controlling the particle size of the composition, thus optimizing the process of applying it to the die and workpiece, but they produce improvements in the performance of the lubricant composition. Since the lubricant particles are ground or spray-dried from an essentially homogenous mass, the lubricant components are far more evenly distributed in the composition than could be accomplished using conventional dry mixing techniques. Indeed, the process described can produce individual particles of heterogeneous composition, and having much more uniform dielectric properties than a strictly dry-mixed composition.

The result is that, when sprayed onto the die and workpiece at elevated temperatures, the particles melt and fuse to form a lubricant film which is substantially uniform. Not only are the lubricant components more evenly distributed on the die and workpiece surfaces when the particles are manufactured in this fashion, thus providing improved resistance to sticking and more uniform metal flow along surfaces, but the cleanability of the composition is improved on account of the more uniform distribution of the resins which are included for that purpose.

The process of applying the lubricant of the invention is carried out at essentially ambient pressure by the use of conventional powder coating equipment. For example, it is well known that, in a conventional electrostatic powder coating apparatus, a fluidized bed of powder feeds a spray wand having an electrode at its tip. While the apparatus injects air into the powder at rather low pressure to form the fluidized bed, by the time the powder reaches the applicator wand tip (typically a distance of about 20 feet), the air carrying the powder (and therefore the powder stream) is at quite low, essentially ambient pressure. The charge imparted to the powder by the electrode provides the acceleration necessary to carry the powder to the die (maintained at ground). Once on the die surface, the lubricant powder may be retained there by the adhesive properties of at least one component included for that purpose.

Alternatively, a conventional powder coating apparatus, whether electrostatic or non-electrostatic, may utilize a gravity-fed conical hopper as a powder source, rather than a fluidized bed. Such an apparatus has been found particularly useful when utilizing lubricant powders of widely varying particle size or relatively heavy lubricant blends, which do not readily form fluidized beds. When such a gravity-fed apparatus is utilized, it has been further found that optimal results in feeding the powder to the spray wand are obtained when the lubricant particles are either substantially spherical in shape or have substantially smooth surfaces, or, most preferably, both. These characteristics permit the lubricant particles to flow more easily, since they will have less tendency to fuse on account of impact or to wedge against one another, thereby blocking flow of material. From the standpoint of optimizing both shape and surface characteristics, the method of manufacture described above which employs spray-drying is the preferred one, since spray-drying inherently produces substantially spherical, substantially smooth particles.

In the process of the invention, a coating of the lubricant powder is applied to the workpiece and the die in a fashion much like painting. The lubricant is not worked onto or into the die or workpiece surface. Rather, the process is more akin to painting the lubricant onto the die than to hammering it into the surface.

From the foregoing description and examples, it is apparent that the objects of the present invention have been achieved. While only certain embodiments have been set forth, alternative embodiments and various modifications will be apparent to those skilled in the art. These and other alternatives and modifications are considered equivalents and within the spirit and scope of the present invention.

Claims

1. A carrier-free pulverulent metalworking lubricant composition, said composition comprising at least one resin having a highly polar functional group, whereby said resin may be solubilized under strong acid or basic conditions, said resin being a solid at room temperature.

2. A composition according to claim 1, wherein said at least one resin is selected from the group consisting of polyethylene glycol resins, polyester resins, polyacrylate resins, polymethacrylate resins, polyamide resins and mixtures thereof.

3. A composition according to claim 2, wherein said at least one resin comprises a polyester resin having a carboxyl functional group.

4. A composition according to any preceding claim wherein said at least one resin is present in an amount of from about 5% to about 50% by weight of the composition.

5. A composition according to any preceding claim, said composition being substantially free of gilsonite.

6. A composition according to any preceding claim further comprising a solid lubricant selected from the group consisting of metal soaps, graphite, ceramics, natural and synthetic waxes, glasses, fatty acids and mixtures thereof.

7. A composition according to claim 6, wherein said metal soap is selected from the group consisting of carboxylates, naphthenates, sulfonates and mixtures thereof.

8. A composition according to claim 6 wherein said metal soap comprises at least one fatty acid soap.

9. A carrier-free pulverulent metalworking lubricant composition, said composition having a substantially uniform particle size.

10. A composition according to claim 9, said composition being substantially free of particles having a particle size which varies more than 15% from the average particle size of said composition.

11. A composition according to claim 9 or claim 10 comprising at least one solid lubricant selected from the group consisting of metal soaps, graphite, ceramics, high melting polymer resins, natural and synthetic waxes, glasses, fatty acids and mixtures thereof.

12. A composition according to claim 11, wherein said metal soap is selected from the group consisting of carboxylates, naphthenates, sulfonates and mixtures thereof.

13. A composition according to claim 11, wherein said metal soap comprises at least one fatty acid soap.

14. A composition according to any one of claims 11 to 13 wherein said high melting polymer resin is poly (tetrafluoroethylene), high density polyethylene, poly(vinylchloride), polyethylene glycol, polyester, polyamide, polyacrylate, polymethacrylate or mixtures thereof.

15. A composition according to any one of claims 11 to 14 wherein at least one component of said composition has adhesive properties at forging temperatures.

16. A composition according to claim 15 wherein said at least one component having adhesive properties at forging temperatures is selected from the group consisting of natural and synthetic waxes, high melting polymer resins, gilsonite and glasses.

17. A carrier-free pulverulent metalworking lubricant according to claim 16 comprising:
   (a) at least one component selected from the group consisting of graphite and boron nitride; and,
   (b) a glass.

18. A method of forming a carrier-free pulverulent metalworking lubricant composition from lubricant components, comprising the steps of
   (a) forming a dry admixture of lubricant components,
   (b) heating said admixture to a temperature sufficient to melt at least one component of said admixture,
   (c) agitating said heated admixture to form a substantially homogenous melt,
   (d) cooling said substantially homogenous melt to form a substantially solidified mass, and
   (e) comminuting said substantially solidified mass to a desired particle size or spray drying said melt to a desired particle size.

19. A method according to claim 18, wherein said step of comminuting said substantially solidified mass to a

desired particle size is carried out under conditions of lower than room temperature.

20. A method of forging a workpiece in a die comprising the steps of
(a) applying to at least one of said die and said workpiece a coating of an effective amount of a carrier-free pulverulent lubricant composition, said composition comprising at least one resin having a highly polar functional group, whereby said resin may be solubilized under strong acid or basic conditions, said resin being a solid at room temperature, and,
(b) forging said workpiece in said die.

21. A method according to claim 20 wherein the workpiece is aluminium.

22. A method according to claim 20 or 21 wherein said carrier-free pulverulent lubricant composition is applied by spraying.

23. A method according to claim 22 wherein said carrier-free pulverulent lubricant composition is applied by electrostatic spraying.

24. A method according to any one of claims 20 to 23 comprising the steps of
(a) applying to said workpiece a coating of an effective amount of said carrier-free pulverulent lubricant composition;
(b) heating said workpiece to a pre-selected temperature;
(c) inserting said workpiece into said die; and,
(d) forging said workpiece in said die.

25. A method of forging a workpiece in a die comprising the steps of
(a) applying to at least one of said die and said workpiece a coating of an effective amount of a carrier-free pulverulent lubricant composition, said composition having a substantially uniform particle size, and,
(b) forging said workpiece in said die.

26. A method according to any one of claims 20 to 24 wherein said lubricant composition further comprises graphite, a fatty acid which is a solid at room temperature and a metal soap.

27. A method according to claim 25 wherein said lubricant composition comprises, by weight, from 5% to about 50% of said at least one resin, from about 15% to about 60% graphite, from about 10% to about 30% of a fatty acid which is a solid at room temperature and from about 20% to about 50% metal soap.

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PARTIAL EUROPEAN SEARCH REPORT

under Rule 46, paragraph 1 of the European Patent Convention

Application Number

EP 92 30 1823

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X Y	GB-A-2 227 022 (HANANO COMMERCIAL) * Page 5, line 2; page 4, line 7 - page 5, line 14; page 6, last paragraph - page 7, line 4 * ---	1,2,4,5-8 20-24	C 10 M 111/04 // (C 10 M 111/04 C 10 M 103:00 C 10 M 103:02 C 10 M 105:22 C 10 M 105:72 C 10 M 107:04 C 10 M 107:28 C 10 M 107:32 C 10 M 107:34 C 10 M 107:38 C 10 M 107:44) C 10 N 40:24 C 10 N 50:08 C 10 N 70:00
Y	EP-A-0 403 306 (CASTROL INDUSTRIAL) * Page 3, lines 25-43; claims 12-14 * ---	20-24	
P,X	EP-A-0 468 278 (DAIDO MACHINERY) * Page 2, lines 44-46,54-55; page 3, lines 13-19; page 4, lines 41-48; page 5, lines 4-9 * ---	1,2,4-6,20,22-24	
X	PATENT ABSTRACTS OF JAPAN, vol. 6, no. 143 (C-117), 3rd August 1982; & JP-A-57 065 795 (NIPPON STEEL CORP.) 21-04-1982 * Abstract * --- -/-	1-3,6	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			C 10 M

LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

See Sheet B

The present partial European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims.

Place of search	Date of completion of the search	Examiner
THE HAGUE	17-06-1992	HILGENGA K.J.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

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European Patent Office

PARTIAL EUROPEAN SEARCH REPORT

Application Number
EP 92 30 1823

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	EP-A-0 116 882 (LONZA AG) * Page 3, lines 13-15; page 4, lines 2-5,14-17; page 5, lines 30-32 * ---	1,2,4-8	
X	PATENT ABSTRACTS OF JAPAN, vol. 012, no. 015 (C-469), 16th January 1988; & JP-A-62 169 899 (NIPPON STEEL CHEM. CO.) 27-07-1987 * Abstract * ---	1,2,6,20	
A	FR-A-1 183 959 (J.J. CAUBET) * Claims 1,2 * ---	26,27	
A	EP-A-0 295 074 (VAN STRAATEN CORP.) * Column 3, line 64 - column 4, line 5 * ---	6,7,8,26,27	TECHNICAL FIELDS SEARCHED (Int. Cl.5)
A	FR-A-2 339 671 (TRW INC.) * Page 4, line 28 - page 5, line 17; page 13, lines 9-18 * ---	1,2,5,6,20,22,23,24	
A	US-A-4 752 405 (G.H. KYLE) * Column 1, line 61 - column 3, line 10 * -----	1,2,6-8	

European Patent Office

EP 92 30 1823 - B -

LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirement of unity of invention and relates to several inventions or groups of inventions, namely:

1. Claims 1-8: Carrier-free pulverulent metalworking lubricant composition, said composition comprising at least one resin having a highly polar functional group.
2. Claims 9-17: Carrier-free pulverulent metalworking lubricant composition, said composition having a substantially uniform particle size.
3. Claims 18,19: Method of forming a carrier-free pulverulent metalworking lubricant composition.
4. Claims 20-24, 26, 27: Method of forging a workpiece by applying lubricant composition of claim 1.
5. Claim 25: Method of forging a workpiece by applying lubricant composition of claim 9.