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3,301,780

**PREPARATION OF COMPACTED BODY OF
IMPROVED LUBRICATING AGENTS**

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This invention relates to laminated metallic lubricating agents and more particularly to a method for substantially improving the wear rates of laminated metallic lubricating agents and the manufacture of a compacted body of such improved lubricating agents.

Certain inorganic compounds, i.e., the sulphides, disulphides, selenides and tellurides of such metals as molybdenum, tungsten, titanium and uranium are characterized by a laminated or plate-like crystal structure in which the metallic atoms are attached to each other in a single common plane while the non-metallic atoms are attached to the metallic atoms to form layers on both sides of this plane. The non-metallic atoms in each of the several crystals thus formed have very little attraction for each other so that these crystals will slip readily with response to each other under the action of low shearing forces. Moreover, the non-metallic atoms have an affinity for adjacent metal surfaces so that the crystals attached to such surfaces will exhibit very strong resistance to the action of forces normal to the direction of shear. Consequently, these compounds have excellent lubricating properties and also have excellent anti-seizing properties.

Most promising of these solid lubricants or "lamellar metal compound lubricants" as they will hereinafter be referred to are, molybdenum disulfide (MoS_2) and tungsten disulfide (WS_2). It has been found, however, that each of these aforementioned lamellar metal compound lubricants unfortunately have a high rate of wear and are consumed quite rapidly as they provide their lubricant function thereby necessitating continual replacement. An additional disadvantage is also contingent upon this high wear rate in that large amounts of this material as it is worn away becomes accumulated between the moving surfaces being lubricated contributing to possible jamming of the lubricated moving parts.

It is therefore an object of this invention to provide a method for greatly improving the wear characteristics of lamellar metal compound lubricants (particularly molybdenum disulfide and tungsten disulfide) while not adversely affecting the lubrication characteristics thereof.

This object may be achieved by exposing the lamellar metal compound lubricant to a mixed gaseous atmosphere of hydrogen sulfide and an inert gas, as for example nitrogen, in about equal volumes.

Although it is known that the wear rate of certain lamellar metal compound lubricants may be improved by the addition of particular additives thereto, it has been experimentally determined that by the practice of the method described herein even lower wear rates can be produced than would be produced by the addition of such additives. These same tests establish that molybdenum (or tungsten) disulfide treated in accordance with this invention exhibits coefficients of friction comparable to MoS_2 (or WS_2) pellets containing additives such as are described in U.S. Patent No. 3,127,346.

In accordance with this invention it has been found that by brining hydrogen sulfide gas diluted with an inert gas into contact with crystals or particles of molybdenum disulfide or tungsten disulfide the particular quality of enhanced resistance against wear inures to these crystals or particles after this treatment. A theory proposed to explain this behavior holds that the hydrogen sulfide gas

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becomes adsorbed on the surface of these crystals or particles. When these treated crystals or particles are then compacted into a pellet the adhered hydrogen sulfide promotes cohesion of the crystals or particles. This increased cohesiveness will in itself diminish the rate at which minute portions of such pellets are torn from the surface. However, in addition to the aforementioned behavior, the chemical and physical activity which takes place when a treated molybdenum (or tungsten) disulfide pellet is rubbed over a metal surface under pressure is believed strongly contributory to diminished wear. During this rubbing contact the adsorbed hydrogen sulfide is released to function as a sulfiding agent to attack the metal surface forming a thin layer of the sulfide of the metal along the path of contact. This thin layer of sulfided metal promotes the distribution thereof of a layer of molybdenum (or tungsten) disulfide, which is much thinner than would be produced in the absence of the sulfided layer. The thinner layer of molybdenum (or tungsten) disulfide is much more tenacious, less easily fractured and therefore less prone to need replacement. As the result thereof the life of the pellet is greatly increased.

This development is of particular value in connection with multi-point bearing support of rotating shafts as is described in U.S. 3,051,535, assigned to the assignee of this invention. One construction would employ a cylindrical treated pellet of laminar metal compound lubricant as one of these projections mounted in a common plane spaced circumferentially 120° apart and projecting radially inward from a bearing housing to support a rotating shaft out of contact with the bearing housing. Pellets prepared and tested in completing this invention were produced by compacting pure microsize molybdenum (or tungsten) disulfide into a $\frac{5}{16}$ inch diameter cylindrical unit under a compacting pressure of about 10 to about 15 tons per square inch. The tests were conducted by applying a load of about 2 kilograms to the test pellet while the pellet was oriented to be rubbed against a series of metal surfaces at a speed of about 130 feet per minute. The term "microsize" is a sizing designation employed in industry to indicate that most of the particles in material so designated have a size ranging from about 1 to about 2 microns. The tests were carried on at room temperature and relative humidity (about $23-25^\circ\text{C}$. and about 55 percent, respectively) in room air.

In the conduct of this method the molybdenum (or tungsten) disulfide material being treated, either as a pellet or as microsize particles later to be consolidated into pellet form, is exposed at atmospheric pressure and about room temperature to a mixture of about equal quantities of H_2S and N_2 for a period of about 2 hours. In place of nitrogen, which functions primarily as a diluent or carrying agent for the hydrogen sulfide, other inert gases, as for example carbon dioxide or helium, could be employed. If, however, the inert gas is completely eliminated, the wear rate has been found, quite unexpectedly, to increase in value to at least about the value obtained for the untreated lubricant pellet.

Although the use of atmospheric pressure is exemplary, this treatment can be accomplished at subatmospheric or superatmospheric pressures, if desired. Likewise, temperature is not critical.

Since moisture has a pronounced effect upon the behavior of molybdenum (and tungsten) disulfide, both the hydrogen disulfide and the inert diluent should be as dry as possible. The rate of flow is not critical and, if a mixture is used, it is preferred that the mixture comprise about equal quantities of hydrogen sulfide and diluent gas. If desired, pellets may be prepared of combined treated particles of both molybdenum disulfide and tungsten disulfide.

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Table I presents examples of the wear rate for treated molybdenum disulfide pellets. Thus, Examples 2, 3, 4, 6 and 8 reflect the performance of pellets treated with H_2S and N_2 , or H_2S alone, as described hereinabove in the form of microsize particles, which particles were later consolidated into pellets. Examples 1, 5 and 7 are included to provide a clear basis for comparison. The tests varied in length from about 15 to about 54 hours.

Table I

Test system:	Wear rate, mgm. per hour
(1) Untreated pellet on bright copper ----	0.028
(2) Treated (H_2S/N_2) pellet on bright copper -----	0.0006
(3) Treated (H_2S) pellet on bright copper -----	0.0303
(4) Treated (H_2S) pellet on bright copper -----	0.0437
(5) Untreated pellet on smooth chromium -----	About 0.030 or less
(6) Treated (H_2S/N_2) pellet on smooth chromium -----	0.00048
(7) Untreated pellet on chromium-plated mild steel -----	About 0.035
(8) Treated (H_2S/N_2) pellet on chromium-plated mild steel -----	0.0091

Microsize particles of tungsten disulfide were treated for about 2 hours with H_2S and N_2 as described hereinabove, compacted into pellets and tested over a period of about 20 hours on bright copper. Although the results obtained on test were not as spectacular as in the case of molybdenum disulfide, a reduction in the wear rate of more than 40% was obtained.

Applicant has, accordingly, discovered a novel method for quickly and effectively treating lamellar metal compound lubricants, particularly MoS_2 and WS_2 , to reduce the wear rate of the untreated material and for preparing useful compacted bodies of these solid lubricants so treated.

While modifications or variations of this invention may be effected in the particular mode of bringing the lamellar metal compound lubricant in contact with an adherent sulfiding agent the particular method disclosed herein is

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given by way of example and this invention is intended to include all such equivalent methods as may be recognized in the art including such methods as may employ obvious modifications of time, flow rate and environmental conditions.

What I claim as new and desire to secure by Letters Patent of the United States is:

1. A method for preparing a compacted body of laminated metallic lubricating material having a greatly reduced wear rate comprising the steps of:

(a) subjecting microsize particles of at least one crystalline material selected from the group consisting of molybdenum disulfide and tungsten disulfide to the flow of a substantially dry mixture of hydrogen sulfide gas and an inert gas for a period of at least about two hours,

said inert gas being present in an amount ranging from a maximum of about 50 percent by volume to a volume effective to substantially reduce the wear rate of a pellet produced from said particles after treatment with said mixture as compared to a similar pellet produced from said particles without said treatment, and

(b) compacting said particles into a pellet.

2. The method substantially as set forth in claim 1 wherein the mixture consists of about equal volumes of hydrogen sulfide gas and inert gas.

3. The method substantially as set forth in claim 2 wherein the inert gas is nitrogen.

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