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PROCESS OF LUBRICATING METAL SURFACE
AND ARTICLE RESULTING THEREFROM

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This invention relates to a process of improving the lubricant properties of metal surfaces, and, more particularly, to a process of treating metal surfaces with inorganic disulphides, diselenides and ditellurides and to the surfaces of metal thereby produced.

Molybdenum disulphide is a mined product refined by oil flotation and marketed as a finely divided black powder. It contains various impurities, including polysulphides, oxides, acid soluble salts, free sulphur and flotation oil. After refining, the powder exhibits remarkable properties in reducing the friction of moving iron and steel parts, particularly at high temperatures where ordinary lubricants, such as oils and greases, rapidly oxidize and fail to provide lubrication, and also where heavy pressure is brought to bear on small areas, such as in the lubrication of center stocks in machining operations. It has also been found effective for ball bearings.

Heretofore, the steel surface has been impregnated or coated by rubbing the dry powder on the steel, or by painting the steel with a suspension of the powder in an organic solvent. Surfaces lubricated in this manner are noisy because of the absence of a cushion of oil or grease to deaden the sound; moreover, the lubricant impregnation is not permanent. To reduce the sound, molybdenum disulphide can be used in combination with mineral oils or grease. In such combinations, the material with which it is mixed may coat the metallic surface or may contribute to lubrication by its inherent oiliness. However, because of the presence of oil or grease such a surface does not stand up under high temperatures and pressures.

In an effort to obtain a more permanent surface under such conditions, molybdenum disulphide has been mixed into a smooth paste with an organic binder. This paste is painted on the steel, which has previously been heated to an elevated temperature, and after the steel has cooled it is finished by abrasion, followed by washing in ethyl alcohol. This gives a more enduring surface, but again the surface is not as permanent as could be desired, particularly at high temperatures and pressures.

It has now been determined that if molybdenum disulphide is subjected to pressure, preferably in excess of approximately 1000 pounds per square inch, at an elevated temperature, preferably in excess of 300° F. or higher, while in contact with the surface of the metal to be treated, the metal acquires a lubricant surface which has a very low coefficient of friction and excellent

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stability and endurance at high temperatures and pressures.

Furthermore, in addition to molybdenum disulphide, under these conditions lubricant surfaces may be imparted to steel, iron, copper and bronze by use of molybdenum diselenide, molybdenum ditelluride, or indeed any disulfide, diselenide or ditelluride of the form MX_2 , where M is a metal selected from the group consisting of elements of Groups IV(b), VI(b), VII(b) and VIII of the periodic classification of elements, and X is selected from the group consisting of sulfur, selenium and tellurium. This group includes iron, in the form of pyrites, (FeS_2) , manganese, in the form of hauerite, (MnS_2) , cobalt, molybdenum, tungsten, ruthenium, titanium, zirconium, uranium, palladium, platinum, osmium and iridium in the form of their corresponding disulfides, diselenides and ditellurides. It is also possible to employ compounds which decompose under the process conditions employed to give the desired disulfide, diselenide or ditelluride; for example, MoS_3 and WS_3 give, respectively, MoS_2 and WS_2 when heated, and therefore are equivalent of the corresponding disulfides. Most polysulphides do likewise.

The remainder of the description describes use of disulfides, but it will be understood that the diselenides and ditellurides are equivalent thereto and may be substituted therefor.

It is desirable that the surface of the metal article to be coated first be cleaned thoroughly. This may be done by a vapor blast (mechanical cleaning process involving high pressure abrasion of the surface by an aqueous dispersion of powdered abrasive) or by washing it with ethyl alcohol, followed by immersion in dilute aqueous acid (such as sulfuric, nitric or hydrochloric acids) solution for a brief period, say a few seconds or minutes, rinsing with water, and rewashing with alcohol. This facilitates the combination of the disulphide with the metal surface.

The metal article may then be packed in disulphide powder in a pressure chamber of standard type, equipped with heating elements. Pressure may then be exerted upon the packed metal article while the temperature of the chamber is raised to the desired point, and held there for the requisite time. Thereafter, the assembly is cooled to room temperature and the surfaced metal article removed. Excess disulphide may then be brushed or dusted off; no further processing is necessary.

It has been found that pressures from about 4000 to about 7000 pounds per square inch, at a

temperature from about 500 to about 900° F. and a reaction period of about 3 to about 7 hours, give the best surfaces. At low pressures, say 1000 to 4000 pounds per square inch, it is desirable to use either a lengthy reaction time or high temperature, or both. Likewise, if low temperatures are used, high pressures and/or lengthy reaction times should be used. In general, good results will be obtained if there is employed a reaction time from 1 to 9 hours, temperatures ranging from about 300° to about 1000° F. and pressures from 1000 to 10,000 pounds per square inch. If the temperature is too low, the surface will flake off, and if the pressure is too low the surface will lack durability at high temperatures and pressures.

The process of the invention is particularly applicable to iron and steel surfaces but it may also be applied to copper, bronze and other customary metal bearing surfaces with good results.

The practice of the invention is illustrated by the following example:

Example I

Several annular rings made of cold-roll steel and having as a bearing surface a raised central platform portion are washed with ethyl alcohol and then immersed in 25% aqueous nitric acid for a few seconds. They are rinsed and dried and then rewashed with alcohol. Thereafter, they are packed in molybdenum disulphide in a thick-walled brass pressure cup, whose upper portion is shaped to receive a 2-inch diameter circular pressure plunger. The plunger is forced into the pressure cup under a load of 40,000 pounds and the temperature of the cup raised to 540° F. and held at that pressure and temperature for 5 hours. The assembly is then cooled to room temperature and the molybdenum disulphide-surfaced steel rings removed and dusted off.

The lubricant properties of a surface obtained as above described were tested by rotating the bearing surface of one ring 220 revolutions per minute in contact with the bearing surface of a stationary second identical ring which was spring-mounted to produce a unit pressure of 240 pounds per square inch. The specimens endured 265,922 revolutions before seizing; the coefficient of friction was 0.21 at the start and gradually decreased to 0.07.

A second pair of rings prepared as above set forth was brushed with a soft brass wire plater's brush having 0.005 inch diameter bristles one inch long to remove loose matter. This pair endured 236,022 revolutions; the coefficient of friction was 0.17 at the start and gradually decreased to 0.06.

For purposes of comparison, the results of a previous procedure are given: A set of identical steel rings was coated with molybdenum disulphide by the following procedure: Finely divided molybdenum disulphide was mixed into a smooth paste with commercial corn syrup. This homogeneous material was painted on a steel ring which had been previously heated to 350° C. A thick heavy coating was formed on the metal. After the ring had cooled, the outer loosely adherent molybdenum disulphide was scraped off with a straight edge, and the ring was finished by abrasion with 3/0 emery paper, washed with ethyl alcohol and then dried. The rings were tested as above set forth and seized after 8,400 revolutions; the coefficient of friction was 0.26 at the start, gradually decreasing to 0.12.

In a second comparative test, one portion molybdenum disulphide was mixed with one portion benzene, and the suspension brushed on a clean steel ring of the same type and dried. The ring seized after 12,000 revolutions and had a coefficient of friction of 0.14 at the start, gradually reducing to 0.10.

How disulphides of the character set forth above function as a lubricant cannot as yet be fully explained. When the surface that has been treated with disulphide is examined, it can be seen even with the naked eye that the metal surface has darkened in colour and has apparently retained something from this union that is not readily removed by friction. All of the above-mentioned disulfides have a similar crystal structure. It has been shown that the sulphur atoms lie together in pairs at some distance from the metal atom. The metal atoms form face-centered cubic lattices and the bond between two metal atoms cuts that between two sulfur atoms in an oblique direction. Each metal atom is surrounded by six sulphur atoms, arranged at the corners of an octahedron, and belonging to six different S₂ groups. Conversely, each S₂ group is surrounded by six metal atoms arranged octahedrally.

It is thought that the disulphide may form a sheet or lamina at the surface of the metal, with two rows of sulphur atoms disposed respectively on the inside and on the outside surfaces of this sheet. The inner surface of the lamina thus is believed to adhere strongly to the iron, steel, copper or bronze surface because of formation of a strong metal-sulphur bond, while the outer lamina slips easily because of the non-existence of such a bond and because the outer row of sulphur atoms slips more readily over surfaces it contacts than the original metal surface.

Experiments indicate that this layer of disulphide provides a cushion which aids in reducing the friction on moving parts much in the same manner as an oil film, but apparently not to the same degree. However, it does not thin out with heat and does not become readily oxidized at even relatively high temperatures and pressure and is therefore highly stable under those conditions, whereas oils or other lubricants are not stable.

The treatment may be used to improve the lubricant properties of steel and iron chains on conveyors operating in ovens and elsewhere at temperatures up to several hundred degrees Fahrenheit. The non-carbonizing properties and stability of the surface enable a considerable reduction in labor costs, due to a decrease in lubrication schedules, and are therefore a definite economic advantage. Further, the surface does not at high temperatures build up a non-lubricating film or coating which must be removed before adequate lubrication may be obtained. After the surface has worn out, resurfacing with more disulphide in accordance with the process of the invention is all that is necessary for continued uninterrupted service.

It is also possible to draw hardenable steels to the desired hardness while simultaneously applying the lubricant surface of the invention by carrying out the process at a temperature and pressure and for a time sufficient to draw the steel to the desired degree of hardness. For example, the lubricant surface may be applied simultaneously to spinning rings while hardening them. Also, in the tool and die industry, the lubricant surface of the invention may be ap-

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plied while hardening punches and dies. This is illustrated by the following example:

Example II

A "Stentor" undrawn steel punch is heat-treated in the usual way. Preparatory to drawing the temper, the punch is packed in molybdenum disulphide, and then subjected to a pressure of 3,000 pounds per square inch and a temperature of 375° F. for 4 hours. The punch is then brushed off as in Example I. The tool has a very durable lubricant surface, increasing its useful life.

The following is claimed:

1. A process of treating the surface of a metal of the type employed for bearing surfaces to improve its lubricant properties, which comprises depositing on the surface of said metal a compound of the general formula MX_2 , where M represents an element selected from the group consisting of iron, manganese, cobalt, molybdenum, tungsten, ruthenium, titanium, zirconium, uranium, palladium, platinum, osmium and iridium, and X represents an element selected from the group consisting of sulfur, selenium and tellurium, and then subjecting the compound to the action of mechanical pressure of at least 1000 pounds per square inch at a temperature of at least 300° F. for at least one hour.

2. A process in accordance with claim 1, in which the compound is molybdenum disulphide.

3. A process in accordance with claim 1, which comprises preparing the surface of said metal for treatment with said compound by washing it with an alcohol and then subjecting it to the action of dilute aqueous acid solution and thereafter washing the surface to remove the acid solution.

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4. A process in accordance with claim 1, in which the metal being treated is steel.

5. The process in accordance with claim 4 in which the steel is undrawn and is drawn to temper concurrently with the application of the lubricant surface.

6. A process of treating the surface of a bearing metal to improve its lubricant properties which comprises depositing on the surface of said metal a compound of the general formula MX_2 , where M represents an element selected from the group consisting of iron, manganese, cobalt, molybdenum, tungsten, ruthenium, titanium, zirconium, uranium, palladium, platinum, osmium and iridium, and X represents an element selected from the group consisting of sulfur, selenium and tellurium, and then subjecting the compound to the action of mechanical pressure substantially from 4000 to 7000 pounds per square inch at a temperature substantially from 500° F. to 900° F. for a period of substantially three to seven hours.

7. A process, in accordance with claim 6, in which the compound is molybdenum disulfide.

8. The product produced in accordance with the process of claim 1.

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